DOE/ER-0589

ENERGY MATERIALS COORDINATING COMMITTEE (EMaCC)

Fiscal Year 1992

May 1993

Annual Technical Report

U.S. Department of Energy
Office of Energy Research
Office of Basic Energy Sciences
Division of Materials Sciences

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Washington, D.C. 20585

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INTRODUCTION

The DOE Energy Materials Coordinating Committee (EMaCC) serves primarily to enhance coordination among the Department's materials programs and to further effective use of materials expertise within the Department. These functions are accomplished through the exchange of budgetary and planning information among program managers and through technical meetings/workshops on selected topics involving both DOE and major contractors. In addition, EMaCC assists in obtaining materials-related inputs for both intra- and interagency compilations.

Six topical subcommittees have been established to focus on materials areas of particular importance to the Department; the subcommittees and their respective chairmen are:

Electrochemical Technologies - Richard Kelly, ER-132, (301) 903-6051
Metals and Intermetallics - Matthew McMonigle, CE-231, (202) 586-2082
Radioactive Waste Containers - Alan Berusch, RW-22, (202) 586-9362
Semiconductors - Jerry Smith, ER-132, (301) 903-4269
Structural Ceramics - Charles Sorrell, CE-232, (202) 586-1514
Superconductivity - James Daley, CE-142, (202) 586-1165

Membership in the EMaCC is open to any Department organizational unit; participants are appointed by Division or Office Directors. The current active membership is listed on the following four pages.

The EMaCC reports to the Director of the Office of Energy Research in his capacity as overseer of the technical programs of the Department. This annual technical report is mandated by the EMaCC terms of reference. This report summarizes EMaCC activities for FY 1992 and describes the materials research programs of various offices and divisions within the Department.

The Chairman of EMaCC for FY 1992 was Stanley M. Wolf; the Executive Secretary was Alan Dragoo. The compilation of this report was assisted by Technology Assessment and Transfer, Inc.

Dr. Alan Dragoo
Office of Basic Energy Sciences
Office of Energy Research
Chairman of EMaCC, FY 1993

MEMBERSHIP LIST
DEPARTMENT OF ENERGY
ENERGY MATERIALS COORDINATING COMMITTEE

Organization	Representative	Phone No.
ENERGY EFFICIENCY AND RENEWABLE ENERGY		
Building Technologies		
Building Systems and Materials	Peter Scofield, CE-421	202/586-9193
Industrial Technologies		
Industrial Energy Efficiency	Scott Richlen, CE-221	202/586-2078
Waste Materials Management	Donald Walter, CE-222	202/586-6750
Improved Energy Productivity	Matthew McMonigle, CE-231	202/586-2082
Advanced Industrial Materials	Charles Sorrell, CE-232	202/586-1514
Transportation Technologies		
Advanced Transportation Materials	Sidney Diamond, CE-34	202/586-0832
Utility Technologies		
Wind/Hydro/Ocean Technologies	William Richards, CE-121	202/586-5410
Geothermal Technology	Raymond LaSala, CE-122	202/586-4198
Photovoltaic Technology	Morton B. Prince, CE-131	202/586-1725
Advanced Utility Concepts	James Daley, III, CE-142	202/586-1165

MEMBERSHIP LIST (Continued)

Organization	Representative	Phone No.
ENERGY RESEARCH		
Basic Energy Sciences		
Materials Sciences	Iran L. Thomas, ER-13	301/903-3426
Metallurgy and Ceramics	Robert J. Gottschall, ER-131	301/903-3428
Solid State Physics and Materials Chemistry	W. Oosterhuis, ER-132	301/903-3426
Engineering and Geosciences	Oscar P. Manley, ER-15	301/903-5822
Advanced Energy Projects	Cynthia Carter, ER-16	301/903-5995
Program Analysis	Timothy Fitzsimmons, ER-32	301/903-9830
Health and Environmental Research		
Physical and Technology Research	Gerald Goldstein, ER-74	301/903-5348
Fusion Energy		
Fusion Technologies	F. W. (Bill) Wiffen, ER-533	301/903-4963
NUCLEAR ENERGY		
Civilian Reactor Development		
Advanced Reactor Programs	Andrew Van Echo, NE-45	301/903-3930
Space and Defense Power Systems		
Defense Energy Projects	C. Chester Bigelow, NE-52	301/903-4299
Special Applications	William Barnett, NE-53	301/903-3097

MEMBERSHIP LIST (Continued)

Organization	Representative	Phone No.
NUCLEAR ENERGY (continued)		
Naval Reactors	Robert H. Steele, NE-60	703/603-5565
Nuclear Safety Self-Assessment		
Nuclear Quality Assurance	John Dowicki, NE-84	301/903-7729
DEFENSE PROGRAMS		
Research and Advanced Technology		
Research and Technology Development	Gregory J. D'Alessio, DP-242	301/903-6688
Inertial Confinement Fusion	Carl B. Hilland, DP-28	301/903-3687
ENVIRONMENTAL RESTORATION AND WASTE MANAGEMENT		
Waste Operations		
Waste Management Projects	Mark Frei, EM-34	301/903-7201
Technology Development		
Transportation Management	Chester E. Miller, EM-50.1	301/903-7967
Research and Development	Stanley M. Wolf, EM-54	301/903-7962

MEMBERSHIP LIST (Continued)

Organization	Representative	Phone No.
FOSSIL ENERGY		
Management, Fundamental Research, and Cooperative Development		
Technical Coordination	James P. Carr, FE-14	301/903-6519
CIVILIAN RADIOACTIVE WASTE MANAGEMENT		
Analysis and Verification	Alan Berusch, RW-2	202/586-9362

FY 1992 BUDGET SUMMARY TABLE FOR DOE MATERIALS ACTIVITIES

(These numbers represent materials-related activities only. They do not include that portion of program budgets which are not materials related.)

	FY 1992
Office of Building Technologies	$ 750,000
Office of Building Energy Research	750,000
Buildings Systems and Materials Division	750,000
Office of Industrial Technologies	$24,107,000
Office of Waste Reduction Technologies	16,822,000
Industrial Energy Efficiency Division	11,040,000
Waste Material Management Division	5,782,000
Office of Industrial Processes	7,285,000
Improved Energy Productivity Division	*
Advanced Industrial Concepts Division	7,285,000
Office of Transportation Technologies	$33,777,000
Office of Transportation Materials	14,897,000
Office of Propulsion Systems	18,460,000
Advanced Propulsion Division	14,055,000
Electric Hybrid Propulsion Division	4,405,000
Office of Alternative Fuels	420,000

*No input was received from this division.

FY 1992 BUDGET SUMMARY TABLE FOR DOE MATERIALS ACTIVITIES

	FY 1992
Office of Utility Technologies	$ 40,426,000
Office of Solar Energy Conversion	23,400,000
Photovoltaic Energy Technology Division	23,400,000
Office of Renewable Energy Conversion	627,000
Geothermal Division	627,000
Office of Energy Management	16,399,000
Advanced Utility Concepts Division	16,399,000
Office of Energy Research	$298,503,852
Office of Basic Energy Sciences	273,741,651
Division of Materials Sciences	261,500,000
Division of Engineering and Geosciences	7,957,651
Engineering Research	5,109,651
Geosciences Research	2,848,000
Division of Advanced Energy Projects	4,284,000
Office of Fusion Energy	*
Small Business Innovation Research Program	24,762,201

*No input was received from this office.

FY 1992 BUDGET SUMMARY TABLE FOR DOE MATERIALS ACTIVITIES (Continued)

	FY 1992
Office of Environmental Restoration and Waste Management	$ 20,015,000
Office of Management Projects	13,951,000
Office of Technology Development	6,154,000
Office of Nuclear Energy	$156,262,000
Office of Uranium Enrichment	20,362,000
Office of Civilian Reactor Development	28,945,000
Office of Advanced Reactor Programs	3,345,000
Division of High Temperature Gas-Cooled Reactors	3,345,000
Office of Technology Support Programs (LMRs)	25,600,000
Office of Isotope Power Systems	1,955,000
Office of Special Applications	1,955,000
Office of Naval Reactors	105,000,000*
Office of Civilian Radioactive Waste Management	$ 700,000

*Approximate

FY 1992 BUDGET SUMMARY TABLE FOR DOE MATERIALS ACTIVITIES (Continued)

	FY 1992
Office of Defense Programs	$ 78,597,000
Office of Research and Advanced Technology	78,597,000
Research and Technology Development Division	78,597,000
Sandia National Laboratory	45,696,000
Lawrence Livermore National Laboratory	21,784,000
Los Alamos National Laboratory	11,117,000
Office of Fossil Energy	$ 6,614,000
Office of Advanced Research	6,614,000
TOTAL	**$659,751,852**

OFFICE OF ENERGY EFFICIENCY AND RENEWABLE ENERGY

The Office of Energy Efficiency and Renewable Energy seeks to develop the technology needed for the Nation to use its existing energy supplies more efficiently, and for it to adopt, on a large scale, renewable energy sources. Toward this end, the Office conducts long-term, high-risk, high-payoff R&D that will lay the groundwork for private sector action.

A number of materials R&D projects are being conducted within the Energy Efficiency and Renewable Energy program. The breadth of this work is considerable, with projects focusing on coatings and films, ceramics, solid electrolytes, elastomers and polymers, corrosion, materials characterization, transformation, superconductivity and other research areas. The level of funding indicated refers only to the component of actual materials research.

The Office of Energy Efficiency and Renewable Energy conducts materials research in the following offices and divisions:

	FY 1992
1. <u>Office of Building Technologies</u>	$ 750,000
a. Office of Building Energy Research	750,000
(1) Buildings Systems and Materials Division	750,000
2. <u>Office of Industrial Technologies</u>	$24,107,000
a. Office of Waste Reduction Technologies	16,822,000
(1) Industrial Energy Efficiency Division	11,040,000
(2) Waste Material Management Division	5,782,000
b. Office of Industrial Processes	7,285,000
(1) Advanced Industrial Concepts Division	7,285,000
3. <u>Office of Transportation Technologies</u>	$33,777,000
a. Office of Transportation Materials	14,897,000
b. Office of Propulsion Systems	18,460,000
(1) Advanced Propulsion Division	14,055,000
(2) Electric Hybrid Propulsion Division	4,405,000
c. Office of Alternative Fuels	420,000

4.	Office of Utility Technologies	$24,537,000
a.	Office of Solar Energy Conversion	23,400,000
	(1) Photovoltaic Energy Technology Division	23,400,000
b.	Office of Renewable Energy Conversion	627,000
	(1) Geothermal Division	627,000
c.	Office of Energy Management	510,000
	(1) Advanced Utility Concepts Division	510,000

OFFICE OF BUILDING TECHNOLOGIES

	FY 1992
Office of Building Technologies - Grand Total	$750,000
Office of Building Energy Research	$750,000
Building Systems and Materials Division	$750,000
Materials Properties, Behavior, Characterization or Testing	$750,000
Development of Non-CFC Foam Insulations	150,000
Compact Vacuum Insulation	150,000
Evacuated Powder Panel Insulation	225,000
Gas-Filled Reflective Insulation Panel	100,000
High-R Test Procedure Development	125,000

OFFICE OF BUILDING TECHNOLOGIES

Office of Building Energy Research

The Office of Building Energy Research works to increase the energy efficiency of the buildings sector through performance of R&D on building systems and building equipment. In addition, it conducts research to support the establishment of appliance standards and labeling and building energy performance standards. Specific objectives include providing the technology to:

- reduce energy consumption in existing buildings, and in new buildings;
- increase the energy efficiency of oil and gas combustion heating systems and of oil- and gas-fired heat pump systems; and
- improve the energy efficiency of advanced electric heat pump and refrigeration systems, and of lighting systems.

Building Systems and Materials Division

The goal of this Division is to provide a scientific and technical basis (including model standards) for reducing the use of energy in residential and commercial buildings by 35 percent by the year 2000 from that used in 1975, while maintaining existing levels of human comfort, health and safety. The Division's primary objectives are to support research that advances the scientific and technical options for increased energy efficiency in buildings, to promote the substitution of abundant fuels for scarce fuels in buildings, and to promulgate standards for increased efficiency of energy use. To accomplish a portion of this, the Building Materials Program seeks to develop new and improve existing insulating materials; to develop and verify analytical models that are useful to building designers and researchers for predicting the thermal performance characteristics of materials; to develop methods for measuring the thermal performance characteristics; and to provide technical assistance and advice to industry and the public. The DOE contact is Peter Scofield, (202) 586-9193.

Materials Properties, Behavior, Characterization or Testing

1. Development of Non-CFC Foam Insulations

FY 1992
$150,000

DOE Contact: Peter Scofield, (202) 586-9193
ORNL Contact: Tom Kollie, (615) 574-7463

This is the final year of a three-year joint project with the rigid foam industry and EPA for the development of alternative blowing agents to be used as drop in replacements for the CFC blowing agents currently being used in the manufacture of foam insulation products. Prototype rigid foam boards with five different blends of HCFC-123 and 141b will be manufactured by industry and sent to ORNL for testing and evaluation both in the laboratory and in outdoor test facilities. Tests will be conducted to determine mechanical and thermal properties and aging characteristics.

Keywords: CFC, Foam Insulation, Insulation Sheathing, Roofs

2. Compact Vacuum Insulation

FY 1992
$150,000

DOE Contact: Peter Scofield, (202) 586-9193
NREL Contact: Robert Parish, (303) 231-1756

This advanced technology insulation project is for the development of a vacuum insulation concept. Two thin stainless sheets are welded together at the edges and a hard vacuum is drawn in the cavity between the sheets separated by a matrix of small glass spheres. Panels with center R-values of five have been made that are 0.1 inches thick. The project goal is to improve center R-values to fifteen.

Keywords: Insulation, Vacuum, Heat Transfer, Radiation

3. Evacuated Powder Panel Insulation

FY 1992
$225,000

DOE Contact: Peter Scofield, (202) 586-9193
ORNL Contact: Tom Kollie, (615) 574-7463

This project is for the development of an advanced technology super insulation concept. A layer of powder is sandwiched between two films and a soft vacuum is drawn on the powder filler. Current technology produces a R-30 per inch panel. Improved powders and longer life encasing films are being developed.

Keywords: Insulation, Vacuum, Heat Transfer

4. <u>Gas-Filled Reflective Insulation Panel</u>

FY 1992
$100,000

DOE Contact: Peter Scofield, (202) 586-9193
LBL Contact: Dariush Aresteh, (415) 486-6844

This project is for the development of a super insulation concept that utilizes layers of reflective films enclosed in a flexible film panel which is filled with low conductivity gases. Mechanisms to provide greater structural rigidity are being investigated as are low permeability films and environmentally benign low conductivity gases.

Keywords: Insulation, Reflective Films, Low Conductivity Gases

5. <u>High-R Test Procedure Development</u>

FY 1992
$125,000

DOE Contact: Peter Scofield, (202) 586-9193
ORNL Contact: Tom Kollie, (615) 574-7463

This project is for the development of an ASTM standard test procedure for measuring the thermal resistance of anisotropic materials with R-values in excess of 20 per inch. The procedure requires the development of a specialized measurement configuration and the modelling of the test specimen within the test configuration.

Keywords: Thermal Resistance, Test Procedures

OFFICE OF INDUSTRIAL TECHNOLOGIES

	FY 1992
Office of Industrial Technologies - Grand Total	$24,107,000
Office of Waste Reduction Technologies	$16,822,000
Industrial Energy Efficiency Division	$11,040,000
Materials Properties, Behavior, Characterization or Testing	$ 2,649,000
Advanced Heat Exchanger Material Technology Development	699,000
Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components	0
Ceramic Fiber Residue Measurement	0
CFCC Supporting Technologies	1,950,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 4,155,000
CFCC Program - Industry Tasks	$ 4,155,000
Device or Component Fabrication, Behavior or Testing	$ 4,236,000
Ceramic Composite Heat Exchanger for the Chemical Industry	576,000
HiPHES System Design Study for Energy Production from Hazardous Wastes	560,000
HiPHES System Design Study for an Advanced Reformer	426,000
Ceramic Components for Stationary Gas Turbines in Cogeneration Service	2,100,000
Long-Term Testing of Ceramic Components for Stationary Gas Turbines	574,000

OFFICE OF INDUSTRIAL TECHNOLOGIES (Continued)

	FY 1992
Office of Waste Reduction Technologies (Continued)	
Waste Material Management Division	$ 5,782,000
Waste Utilization and Conversion	$ 4,598,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 4,598,000
Wood Wastes to Adhesives	1,289,000
Waste Rubber-Polymer Composite	700,000
Zinc-Contaminated Steel Conversion	1,400,000
Silicon Oxide Recovery-Conversion	459,000
Waste Food Carbohydrates to Lactide Copolymer Plastics	750,000
Solar Materials Research	$ 1,184,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 1,184,000
Titanium Dioxide for Photocatalysts (WBS No. CEWS121)	450,000
Solar Materials Processing (WBS No. CEWS121)	734,000
Office of Industrial Processes	$ 7,285,000
Advanced Industrial Concepts Division	$ 7,285,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 3,595,000
Thin-Wall Hollow Ceramic Spheres from Slurries	180,000
Synthesis and Design of Intermetallic Materials	680,000
Polymers with Improved Surface Properties	180,000
Development of Weldable, Corrosion-Resistant Iron-Aluminide Alloys	300,000
Composites Through Reactive Metal Infiltration	350,000
Magnetic Field Processing of Inorganic Polymers	150,000
Development of Improved Aerogel Superinsulation	320,000

OFFICE OF INDUSTRIAL TECHNOLOGIES (Continued)

	FY 1992
Office of Industrial Processes (continued)	
Advanced Industrial Concepts Division (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Microwave-Driven Spray Drying of Ceramic Powders	260,000
Conducting Polymers: Synthesis and Industrial Applications	300,000
Microwave Assisted Chemical Vapor Infiltration	260,000
High Deposition Rate Chemical Vapor Deposition	335,000
Development of Chemical Vapor Composite Materials	280,000
Materials Properties, Behavior, Characterization or Testing	$ 180,000
Three Dimensional X-Ray Tomography of Composites	180,000
Materials Structure and Composition	$2,260,000
The Development of Advanced Ceramic Membrane Technology	150,000
Metallic and Intermetallic Bonded Ceramic Composites	400,000
Advanced Ordered Intermetallic Alloy Development	400,000
Superior Metallic Alloys Through Rapid Solidification Processing by Design	100,000
Polymerization and Processing of Organic Polymers in a Magnetic Field	130,000
Microwave Joining of SiC	80,000
Microwave Sintering of Composites	300,000
Chemical Vapor Infiltration of Ceramic Composites	300,000
Chemical Recycling of Plastics	650,000
Biomimetic Thin Film Synthesis	300,000
Recoverable Thermosets	100,000

OFFICE OF INDUSTRIAL TECHNOLOGIES (Continued)

	FY 1992
Office of Industrial Processes (continued)	
Advanced Industrial Concepts Division (continued)	
Device or Component Fabrication, Behavior or Testing	$1,250,000
Advanced Engineering Coatings Development	100,000
Ni_3Al Technology Transfer: Castability and Weldability of Ni_3Al	500,000
Advanced Microwave Processing Concepts	200,000
Chemically Specific Coatings	450,000

OFFICE OF INDUSTRIAL TECHNOLOGIES

The Office of Industrial Technologies conducts research and development to conserve energy in industry. The goal of these activities is to save energy, achieve higher efficiency, provide for fuel flexibility, and increase productivity in industrial unit operations and processes. To accomplish these objectives, the Office has adopted the basic strategy of identifying, in cooperation with private industry, the technological needs of energy conservation in the industrial sector; identifying what private industry is currently doing or will not do alone; selecting the highest priority targets not being pursued by the private sector; and negotiating cost-shared contracts with private industry or contracts with national laboratories or universities to carry out the necessary research.

Office of Waste Reduction Technologies

The mission of the Office of Waste Reduction is to develop and maintain a balanced program of research and development on generic technologies which contribute to enhanced industrial energy use efficiency and which have wide application throughout industry and agriculture. The program includes activities in waste heat recovery, improved thermal energy management, combustion systems, waste products utilization including municipal solid wastes, and waste stream detoxification.

Industrial Energy Efficiency Division

Materials Properties, Behavior, Characterization or Testing

6. Advanced Heat Exchanger Material Technology Development — FY 1992 $699,000

DOE Contact: S. Richlen, (202) 586-2078
ORNL Contact: M. Karnitz, (615) 574-5150

This project conducts research to develop improved ceramic materials and fabrication processes and to expand the materials data base for advanced heat exchangers. Currently the project is studying the effects of corrosive waste stream constituents on candidate ceramic and ceramic composite materials through coupon tests and exposure to high pressure exhaust gas environments and developing advanced wet forming techniques for monolithic ceramic components.

Keywords: Structural Ceramics, Corrosion-Gaseous, Industrial Waste Heat Recovery

7. Assessment of Strength Limiting Flaws in Ceramic Heat Exchanger Components

FY 1992
$0

DOE Contact: S. Richlen, (202) 586-2078
Babcock & Wilcox Contact: J. Bower, (804) 522-5742

This project studies the flaw populations and the effect of operating environments on flaw populations of ceramic heat exchanger components. Currently the project is correlating acoustic response to flaw growth. The goal of the project is to develop lifetime prediction correlations for ceramic components. Research is conducted cooperatively with the Idaho National Engineering Laboratory.

Keywords: Structural Ceramics, Structure, NDE, Industrial Waste Heat Recovery

8. Ceramic Fiber Residue Measurement

FY 1992
$0

DOE Contact: S. Richlen, (202) 586-2078
ORNL Contact: M. Karnitz, (615) 574-5150

This project determines whether whisker-like particles can be generated during the handling, processing, or machining of continuous ceramic fiber ceramic matrix composites. A test protocol has been written and is under review.

Keywords: Ceramic Composites, Whiskers

9. CFCC Supporting Technologies

FY 1992
$1,950,000

DOE Contact: S. Richlen, (202) 586-2078
ORNL Contact: M. Karnitz, (615) 574-5150

This project provides basic or generic support to the industry teams conducting CFCC research. Tasks include: composite design, materials characterization, test methods development, data base generation, and life prediction.

Keywords: Ceramic Composites, Fiber Architecture

Materials Preparation, Synthesis, Deposition, Growth or Forming

10. CFCC Program - Industry Tasks

FY 1992
$4,155,000

DOE Contact: S. Richlen, (202) 586-2078

The goal of the CFCC Program is to develop in U.S. industry, the primary processing methods for the reliable and cost-effective fabrication of continuous fiber ceramic composite components for use in industrial applications. The first phase which establishes performance requirements of applications and assesses feasibility of potential processing systems is underway.

Keywords: Composites

Device or Component Fabrication, Behavior or Testing

11. Ceramic Composite Heat Exchanger for the Chemical Industry

FY 1992
$576,000

DOE Contact: S. Richlen, (202) 586-2078
Babcock & Wilcox Contact: D. Hindman, (804) 522-5825

The third phase of this project has been initiated to design and build a prototype module heat exchanger using ceramic composite tubes to determine their performance under industrial conditions. Currently, ceramic composite tubes are being proof-tested in a high-temperature furnace to determine their viability for actual use.

Keywords: Ceramic Composites, Structure

12. HiPHES System Design Study for Energy Production from Hazardous Wastes

FY 1992
$560,000

DOE Contact: S. Richlen, (202) 586-2078
Solar Turbines Contact: B. Harkins, (619) 544-5398

This project is in the second phase of a three-phase effort to develop high pressure heat exchange systems for recovery of energy from hazardous wastes. A preliminary design of an advanced heat exchange process based on the use of ceramic composites has been developed. Research on critical material and design needs continues.

Keywords: Ceramic Composites, Heat Exchangers

13. HiPHES System Design Study for an Advanced Reformer FY 1992
$426,000

DOE Contact: S. Richlen, (202) 586-2078
Stone & Webster Engineering Corp. Contact: J. Williams, (617) 589-7147

This project is in the second phase of a three-phase effort to develop high pressure heat exchange systems for an advanced convective reformer. A preliminary design of an advanced heat exchange process based on the use of ceramics has been developed. Research on critical material and design needs continues.

Keywords: Composites, Heat Exchangers

14. Ceramic Components for Stationary Gas Turbines in Cogeneration Service
FY 1992
$2,100,000

DOE Contact: W. Parks, (202) 586-2093
Solar Contact: M. van Roode, (619) 544-5549

This project will design and test three major ceramic components in a stationary 3.5MW gas turbine for cogeneration service. The three components are the combustor, first stage rotor, and first stage nozzle. The project will culminate in a 4000 hour field demonstration of the engine.

Keywords: Structural Ceramics, Cogeneration, Gas Turbines

15. Long-Term Testing of Ceramic Components for Stationary Gas Turbines FY 1992
$574,000

DOE Contact: W. Parks, (202) 586-2093
ORNL Contact: M. Ferber, (615) 576-0818

This project will test monolithic ceramics in static and cyclic fatigue for up to 10,000 hours at gas turbine utilization temperatures.

Keywords: Structural Ceramics, Cogeneration, Gas Turbines

Waste Material Management Division

Waste Utilization and Conversion

Industrial waste solid, liquid, and gaseous materials are waste because they have insufficient economic potential, thus they are landfilled or discharged to the environment. Economically useful wastes are termed by-products and constitute the objective of the Waste Utilization and Conversion program. Materials research can provide technologies to upgrade wastes or create new commodity materials so that wastes can have economic, i.e., added, value to become by-product materials of value to industry or commerce. The DOE contact is Bruce Cranford, (202) 586-9496.

Materials Preparation, Synthesis, Deposition, Growth or Forming

16. Wood Wastes to Adhesives — FY 1992 $1,289,000

DOE Contact: Alan Schroeder, (202) 586-1641
NREL Contact: Helena Chum, (303) 231-7249

Wood wastes are pyrolyzed via a vortex reactor yielding pyrolysis oils. Oils are separated to give a phenols-neutrals fraction which is used to replace phenol in various phenolic resin applications. Because petroleum-based phenol is replaced by wood-based phenol, and because the overall process is cheaper, substantial energy savings of over 200 trillion BTU/year are projected for 2010.

Keywords: Wood, Wastes, Adhesives, Pyrolysis

17. Waste Rubber-Polymer Composite — FY 1992 $700,000

DOE Contact: Stuart Natof, (202) 586-2370
Air Products & Chemicals, Inc., Contact: Dr. Bernard Bauman, (215) 481-2449

A new process is being developed to activate the surface of finely ground waste tire rubber using chlorine. The surface-treated ground waste tire rubber can be used by molders to make new composites with cost savings and/or improved properties. This use of waste tires can result in a net savings of 80,000 BTUs per pound of tire rubber, as a result of displacing relatively energy intensive virgin materials.

Keywords: Tires, Composites, Surface Activation

18. Zinc-Contaminated Steel Conversion FY 1992
$1,400,000

DOE Contact: Bill O. Benchain, (202) 586-3090
Argonne National Laboratory Contact: Edward J. Daniels, (708) 972- 5279

A new process is being developed to economically remove the zinc from galvanized steel scrap, resulting in clean, specification grade, scrap steel and recyclable zinc. The process works on loose shredded scrap or baled scrap. Hot sodium hydroxide with anodic stripping and simultaneous electrowinning is employed. A pilot plant was build in FY92 for operation in FY93.

Keywords: Dezincing, Steel, Galvanized, Zinc, Scrap Metal, Metals Recycling

19. Silicon Oxide Recovery-Conversion FY 1992
$459,000

DOE Contact: Bruce Cranford, (202) 586-9496
Dow Corning Contact: James May, (517) 496-6047

A new process is being developed to economically capture waste SiO emitted from conventional silicon production furnaces and return the SiO to the furnace to increase the conversion of SiO_2 to Si metal. The CO emitted is also utilized for methanol production to improve the energy efficiency of the process. The process has been demonstrated at the pilot scale.

Keywords: Silicon Oxide, Waste Recovery, Waste Conversion

20. Waste Food Carbohydrates to Lactide Copolymer Plastics FY 1992
$750,000

DOE Contact: Alan Schroeder, (202) 586-1641
Argonne National Laboratory Contact: Jim Frank, (708) 972-3268

A process is being developed with TVA, States of Illinois and Minnesota, to produce a biodegradable mulch film/coating with time release fertilizer and pesticide properties that does not have to be taken up at the end of the growing season. An energy analysis indicates savings of 55,000 Btu/lb of plastic used, with a large potential market. It also provides a higher value product outlet for such carbohydrate wastes as cheese whey, potato processing, and corn processing.

Keywords: Biodegradable, Starch, Lactic Acid, Lactide Plastic, Fertilizer, Mulch, Irrigation

Solar Materials Research

The objective of solar materials research is to identify and develop viable materials processes that take advantage of the attributes of highly concentrated solar fluxes. Concentrated sunlight from solar furnaces can generate temperatures well over 2000°C. Very thin layers of the illuminated surfaces can be driven to remarkably high temperatures in fractions of a second. Concentrated solar energy can be delivered over very large areas, allowing for rapid processing. The result is more efficient use of bulk materials and energy, potentially lower processing cost, and reduced need for strategic materials, all with a technology that does not damage the environment.

Materials Preparation, Synthesis, Deposition, Growth or Forming

21. Titanium Dioxide for Photocatalysts (WBS No. CEWS121) — FY 1992 $450,000

DOE Contact: Frank Wilkins, (202) 586-1684
NREL Contact: Daniel M. Blake, (303) 231-1202

The objectives of this work are to determine the characteristics of titanium dioxide that influence its photocatalytic activity and to make modifications of the material which will increase the activity. Titanium dioxide is finding application as a photocatalyst for removal of low concentrations of organic compounds from contaminated water or air. Materials are being characterized and tested and methods of modifying the photocatalytic activity are being investigated.

Keywords: Photocatalyst, Titanium Dioxide, Oxidation

22. Solar Materials Processing (WBS No. CEWS121) — FY 1992 $734,000

DOE Contact: Frank Wilkins, (202) 586-1684
NREL Contact: Allan Lewandowski, (303) 231-1972

The objective of this project is to develop an alternative method of processing various advanced materials using concentrated sunlight as an energy source. A number of processes have been explored including metalorganic deposition of thin films on ceramics, production of ceramic powders, solar assisted chemical vapor deposition of a variety of films on various substrates, rapid thermal heat treating and other surface modification techniques. Two

CRADAs have been initiated to promote transfer of this technology to industry. One is for the metallization and joining of beryllia ceramics used in electronic packages and the other is for the production of small diameter, sinterable silicon carbide powders. Both of these CRADAs are in the initial phases to determine technical feasibility of the processes.

Keywords: Solar, Ceramics, Joining, Metallization

Office of Industrial Processes

Advanced Industrial Concepts Division

The mission of AICD is to support generic, long-term, high-risk applied R&D in those processes and technologies that underpin industrial unit operations. The AICD output provides a technology base to improve energy use efficiency and advance industrial capability to use alternative energy resources. Materials-related research in AICD is conducted in the Advanced Industrial Materials (AIM) Program. The AIM Program develops generic materials technologies brought to a stage for private industry or other government programs to advance further towards technology and engineering demonstration. The Program emphasizes materials as an enabling technology for industrial energy conservation and has research efforts in eight areas: Ordered Intermetallic Alloys, Crack Resistant Composites, Thermally Insulating Materials, Innovative Materials, Microwave Processing, Lightweight and Biobased Materials, Surface Modification, and Innovative Processing. The Program Manager is Charles A. Sorrell, (202) 586-1514.

Materials Preparation, Synthesis, Deposition, Growth or Forming

23. Thin-Wall Hollow Ceramic Spheres from Slurries FY 1992 $180,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Georgia Institute of Technology Contact: J. K. Cochran, (404) 894-6104

The goal of this project is to develop processes for economically fabricating hollow thin-wall spheres from conventional ceramic powders using dispersions and to assess their potential use. The feasibility of producing monosize hollow spheres of many ceramic compositions on a production basis has been successfully demonstrated. The properties of the spheres, i.e., mechanical strength and thermal conductivity, have been documented and mathematical modeling of the sphere forming process has been successful. Current research has three areas of emphasis: (1) reduce thermal conductivity at high temperatures using infrared opacifiers in the sphere walls, (2) convert the liquid slurries used to form spheres from the present organic-based to an aqueous-based system, and (3) disseminate information about sphere technology and properties to U.S. industry.

Keywords: Structural Ceramics, High Temperature Service, Insulation

24. Synthesis and Design of Intermetallic Materials — FY 1992 $680,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Los Alamos National Laboratory Contacts: J. J. Petrovic, (505) 667-0125 and A. D. Rollett, (505) 667-6133

The purpose of this project is to develop and design intermetallic materials using the composite approach to obtain fracture resistance in brittle matrices. Efforts are focused on a high temperature structural material, $MoSi_2$, because of its good oxidation resistance and docility at elevated temperatures. Micro-mechanical modeling of the complex microstructure of these composites has been successful in showing which toughening mechanisms are significant. The modeling has also demonstrated that the dispersion of the toughening additions in the silicide matrix is critical to maximizing the toughening effect.

Keywords: Composites, Intermetallics, Toughening, Micro-mechanical Modeling

25. Polymers with Improved Surface Properties — FY 1992 $180,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: E. H. Lee, (615) 574-5058

The purpose of this project is to enhance and improve the durability, dielectric/electric/magnetic/infrared properties of polymers using surface modification treatments, and to correlate changes in microstructure and microcomposition with surface property changes. Research will emphasize (1) modification of surface-sensitive properties of polymers by multiple-ion beam treatments; (2) characterization of induced changes in microstructure, microcomposition, and properties by means of analytical electron microscopy, ion scattering/nuclear reaction techniques and mechanical testing; and (3) relate ion beam processing parameters to microstructure and properties in order to develop principles for improved materials. Results indicate that near surface hardness values can be significantly improved for shallow implants compared to unimplanted polymers and that shallow implants can be used to improve surface mechanical properties of polymers.

Keywords: Polymers, Surface Modification, Ion Beam Processing

26. Development of Weldable, Corrosion-Resistant Iron-Aluminide Alloys FY 1992
$300,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: G. M. Goodwin, (615) 574-4809 and P. J. Maziasz, (615) 574-5082

The objectives of the project are (1) develop weld-overlay FeAl and other intermetallic alloys as coatings and claddings on intermetallic and conventional structural materials, (2) develop weldable, strong and corrosion resistant FeAl alloys; and (3) determine thermophysical properties of intermetallic alloys. Efforts on FeAl development have shown that environmental effects (hydrogen embrittlement) generally are the mechanisms limiting room-temperature ductility, and that proper control of alloy composition, microstructure and surface condition (oxide layer) significantly improves ductility. Current efforts are focused on the development of FeAl materials for coatings, including weld-overlay coatings and claddings. Studies have shown that such coatings are feasible, but that hydrogen-related cold-cracking of the weld-deposited FeAl and the dilution zone that forms between overlay deposit and the base material are issues that still need to be addressed.

Keywords: Iron Aluminides, Intermetallics, Coatings, Claddings, Thermophysical Properties

27. Composites Through Reactive Metal Infiltration FY 1992
$350,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Sandia National Laboratories Contact: R. E. Loehman, (505) 844-2222

Metal matrix ceramic composites have been fabricated using a novel reactive metal infiltration processing technique. The process involves reacting a molten metal with a ceramic preform to produce a fine scale composite microstructure of ceramic particles within a continuous metal matrix. Experimental variables include the reactive metal and ceramic compositions, and the processing temperature and atmosphere. Advantages of reactive metal infiltrated composites include lower temperature and near net shape processing of ceramic composites and improved toughness compared to traditional ceramics. Current efforts are focused on characterizing the aluminum/mullite system.

Keywords: Metal Matrix Composites, Reactive Metal Infiltration, Ceramics

28. Magnetic Field Processing of Inorganic Polymers FY 1992
$150,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Idaho National Engineering Laboratory Contact: D. C. Kunerth, (208) 526-0103

The application of magnetic fields during processing has been shown to modify the physical and chemical properties of inorganic polymers. Development of this technology will significantly improve current materials as well as lead to new materials for other applications. The objective of this project is to investigate and demonstrate the use of magnetic field processing, to modify the properties of inorganic-based polymers, and to develop the basic technical knowledge required for industrial implementation. Current results have shown that (1) the physical and chemical properties of polyphosphazene polymers has been modified using magnetic field processing, (2) the membrane morphologies and transport properties can be changed with the application of magnetic fields, and (3) the polymer can be textured on the molecular level.

Keywords: Polymers, Magnetic Field Processing

29. <u>Development of Improved Aerogel Superinsulation</u> <u>FY 1992</u>
$320,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Lawrence Berkeley Laboratory Contact: A. Hunt, (415) 486-5370

The purpose of this project is to develop methods of producing new aerogel-based materials using sol-gel processing and supercritical solvent extraction. Aerogel is a porous, low density, nanostructured solid with many unique properties including transparency, very high thermal resistance, and low sound velocity. The primary challenge is to develop new processing methods to prepare aerogel with improved properties for a cost-effective, high performance thermal insulation. For example, aerogel can be substantially improved by adding nanostructured phases to increase the thermal resistance by reducing radiative heat transfer inside the material. Aerogel processing techniques may also be used to make other nanocomposite materials including new magnetic materials. Applications for aerogel-based materials include multicomponent ceramics, catalyst substrates, transparent porous solids, and high-performance thermal insulators—which may replace existing CFC-containing foams currently used in refrigerators, water heaters and industrial plants.

Keywords: Thermal Insulation, Sol-Gel, Processing

30. <u>Microwave-Driven Spray Drying of Ceramic Powders</u> <u>FY 1992</u>
$260,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Los Alamos National Laboratory Contact: F. D. Gac, (505) 667-5126

The purpose of this project is to develop generic microwave processes by the direct coupling of microwave power for the spray-dry preparation of homogeneous oxide powders from aqueous and organic aerosols and for the drying and calcining of green filaments to produce ceramic fibers. Microwave-driven spray drying and filament processing are energy-efficient alternatives to the conventional thermal processing of aerosols and filaments. In thermal processing, thermal energy is transported from a hot gas of hot solid surface through the exterior surface toward the center of the aerosols and filaments. In microwave-driven processing, microwave energy is directly coupled to the entire body of aerosol particles and filaments by volumetric absorption of the electric field. The advantages of microwave processing are the direct rapid heating by the volumetric coupling of clean energy without chemical contaminants from fuel gas heating and from hot reactor surfaces, short processing time and the potential of novel microstructures due to rapid heat-up as the filament is drawn through the microwave cavity.

Keywords: Microwave Processing, Spray Drying, Powder Synthesis

31. Conducting Polymers: Synthesis and Industrial Applications — FY 1992 $300,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Los Alamos National Laboratory Contact: S. Gottesfeld, (505) 667-0853

This project is pursuing new methods for the synthesis of electronically conducting polymers, and the development of new industrial applications for these materials which will result in significant reductions in energy usage or industrial waste. Applications addressed include: (1) improved industrial metallization methods that could replace current costly and environmentally damaging technologies; (2) supercapacitors that could save large amounts of energy when introduced in power trains of electric vehicles, (3) corrosion resistant conductive coatings; and (4) electrochromic conducting polymer films for variable opacity windows.

Keywords: Electrically Conducting Polymers, Metallization, Capacitors, Coatings and Films

32. Microwave Assisted Chemical Vapor Infiltration — FY 1992 $260,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Los Alamos National Laboratory Contact: R. P. Currier, (505) 665-3601

The purpose of this investigation is to develop a viable microwave assisted process for the rapid production of ceramic matrix composites of arbitrary geometry by chemical vapor infiltration (CVI). The potential advantage in using microwaves is to heat the substrate internally, giving rise to "inverted" thermal gradients. With the internal region of the substrate hot, cool reactant gases penetrate inward prior to the onset of reaction. Consequently, deposition occurs from the inside-out. This could offer several advantages over conventional technologies including (1) removing constraints on substrate geometry, (2) obtaining more spatially uniform, high density composites, (3) shortening processing times, and (4) machining to reopen closed pores should not be necessary since densification would occur from inside-out.

Keywords: Microwave Processing, Chemical Vapor Infiltration, Ceramics, Composites

33. High Deposition Rate Chemical Vapor Deposition

FY 1992
$335,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Sandia National Laboratories Contact: M. D. Allendorf, (415) 294-2895

Comprehensive models, including detailed gas-phase and surface chemistry coupled with reactor fluid mechanics, are required to optimize and scale-up chemical vapor deposition (CVD) processes. Several investigators have proposed models that simulate aspects of silicon carbide (SIC) CVD process; however, these models predict only deposition rates and not deposit composition. Therefore, the purpose of this investigation is to develop a quantitative understanding of the chemical and physical mechanisms that result in high deposition rates and fully dense materials from innovative high-temperature ceramic synthesis processes. Current results indicate that gas-phase chemistry is an important element in determining both the deposition rate and the elemental composition. Research will continue with attempts to model the gas-phase chemistry occurring during SiC CVD.

Keywords: Chemical Vapor Deposition, Gas-Phase Chemistry, Modeling

34. Development of Chemical Vapor Composite Materials

FY 1992
$280,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Thermotrex Corporation Contact: P. Reagan, (617) 622-1347

The purpose of this project is to develop a reliable, flexible and economic process to fabricate strong, fracture tough ceramic composites to net shape in one step without machining. The chemical vapor composites (CVC) process has the potential to reduce the high costs and the multiple fabrication steps presently required to produce ceramic composites. Composites are fabricated by simply mixing powders and/or fibers with vapor reactants which are deposited on a hot machined substrate. Current efforts are focused on the development of the CVC process with various powder and fiber combinations.

Keywords: Chemical Vapor Deposition, Ceramics, Composites

Materials Properties, Behavior, Characterization or Testing

35. Three Dimensional X-Ray Tomography of Composites FY 1992
$180,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
LLNL Contact: J. H. Kinney, (415) 243-6669

X-ray tomographic microscopy (XTM) is a new technique for noninvasively imaging materials microstructures in three dimensions. The microporosity between individual filaments in the fiber bundles, the channel porosity between individual cloth layers, and the connectivity of the large through-ply holes that remain after processing can all be examined without destroying the sample. Current XTM research is focused on monitoring the chemical vapor infiltration (CVI) processing of silicon carbide matrix-micalon fiber composites.

Keywords: X-Ray, Tomography, Composites

Materials Structure and Composition $466,000

36. The Development of Advanced Ceramic Membrane Technology FY 1992
$150,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: Peter Angelini, (615) 574-4565
General Magnaplate Corporation Contact: Michael L. Pearce, (908) 862-6200

The purpose of this work is to develop a new coating technology which consists of applying hard ceramic phases in metal or polymer matrices which would be potentially capable of on-site application. The new flame spraying method may significantly increase the number of materials which can be applied as coatings, increase the energy efficiency of processes, improve the durability of coatings, and decrease manufacturing costs compared to conventionally applied coatings. Flame spray coating material combinations and processes will be developed. The structure of the composite coatings will be related to the war and corrosion properties.

Keywords: Ceramics, Composites, Coatings

37. Metallic and Intermetallic Bonded Ceramic Composites — FY 1992 $400,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contacts: T. N. Tiegs and K. B. Alexander, (615) 574-0631
University of California, Berkeley Contact: R. O. Ritchie, (415) 642-0417

To improve the reliability of ceramic components, new approaches to increasing the fracture toughness of ceramics are required. The objective of this project is to establish a framework for the development and fabrication of metallic and intermetallic-reinforced ceramic matrix composites with improved fracture toughness and fatigue resistance. The incorporation of metallic phases into a ceramic matrix allows for local plastic deformation. This deformation acts to dissipate the strain energy introduced by an applied stress, thus increasing the fracture toughness of the composite. The research will focus on the development of alumina-based as well as nonoxide-based (AlN, TiN, TiC, and WC) ceramic composites reinforced with ductile nickel aluminide alloys. This work will also identify relationships between material properties, structure and processing. Reinforced ceramic matrix composites have potential use in advanced industrial applications.

Keywords: Ceramics, Composites, Aluminides

38. Advanced Ordered Intermetallic Alloy Development — FY 1992 $400,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: C. T. Liu, (615) 574-4459

The objective of this project is to develop low-density, high-strength intermetallic alloys for structural use in the advanced heat engines and energy conversion systems. Many ordered intermetallics have attractive high-temperature properties such as excellent oxidation and corrosion-resistance, high-elevated-temperature strength, low material density, and excellent shape memory effects. However, brittle fracture and poor ductility limit their use as engineering materials. The general approach is to improve the ductility and fabricability of ordered intermetallics by controlling the crystal structure. Current efforts are focused on the development of new generation of materials including (1) NiAl alloys with high-temperature capability, (2) TiAl and TiAl, alloys with high specific strength, (3) shape-memory alloys based on NiAl/Ni_3Al, and (4) one Cooperative Research and Development agreement with industry.

Keywords: Intermetallics, Ordered Alloys, Ductility

39. Superior Metallic Alloys Through Rapid Solidification Processing by Design FY 1992
$100,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Idaho National Engineering Laboratory Contact: J. E. Flinn, (208) 526-8127

The industrial sector requires metallic alloys whose properties, performance, and reliability extend beyond those obtained from current processing practices. These needs can be fulfilled by metallic alloys that have fine and stable (to high temperature) microstructure. Rapid solidification processing (RSP) during powder atomization and control of the mirror elements, such as oxygen and strong oxide furnaces, can produce metallic alloys with superior properties and performance compared to conventionally processed alloys. Efforts will continue to collaborate with U.S. industries in applying rapid solidification processing in combination with composition adjustments to produce superior metallic alloys.

Keywords: Rapid Solidification, Alloys, High Temperature Microstructure

40. Polymerization and Processing of Organic Polymers in a Magnetic Field FY 1992
$130,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Los Alamos National Laboratory Contacts: N. E. Elliott, (505) 667-1587, R. K. Jahn, (505) 665-1751 and R. Liepins, (505) 667-2656

The purpose of this project is to evaluate and model the effects of magnetic fields on polymers during polymerization, solidification, and processing in order to improve, modify, and control the mechanical, physical, optical, and electrical properties. The orientation of polymers in a magnetic field and subsequent polymerization can improve the various properties of the material. Current research has shown that magnetic field processing of epoxy resins had a significant positive effect on tensile strength, tensile modulus, reduced water absorption, increased hardness, and improved adhesion properties. The work at Los Alamos National Laboratory emphasized the polymerization and processing of liquid crystalline polymers, non-liquid-crystalline polymers and organic composites (epoxy prepregs). Quantitative correlations have been made between the properties and structure of the material prepared with and without the presence of a magnetic field. Research will continue to focus on dynamic "in-process" magnetic field processing of a liquid-crystalline polymer film.

Keywords: Polymers, Magnetic Processing, Mechanical Properties

41. Microwave Joining of SiC

FY 1992
$80,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
LANL Contact: Joel D. Katz, (505) 665-1424
Technology Assessment and Transfer, Inc. Contact: R. Silberglitt, (301) 261-8373
George Mason University Contact: W. Murray Black, (703) 993-1571

The purpose of this work is to develop and optimize a joining method that can be applied to large scale fabrication of components such as radiant burner tubes and high temperature, high pressure heat exchangers. Efforts were focused on optimizing the properties of SiC-SiC joints made using microwave energy and this resulted in a three micrometer thin silicon interlayer. In addition, research on reaction bonded silicon carbide, which contains residual free silicon, showed no interlayer. Current efforts are focused on forming a carbide interlayer through an *in situ* reaction, and on the design of microwave applicators that will allow joining of larger diameter and longer tubes.

Keywords: Microwave Processing, Microwave Joining, SiC

42. Microwave Sintering of Composites

FY 1992
$300,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: M. A. Janney and H. D. Kimrey, (615) 576-5183
University of Utah Contact: Dr. Magdy Iskander, (801) 581-6944

The purpose of this project is to develop ceramics for energy conversion systems with improved strength, toughness, reliability, and uniformity of properties using microwave processing to engineer and control critical component microstructures. The approach combines both experimental process development and modeling to provide an understanding of microwave processing that can be used in industrial applications. Current efforts focus on (1) developing zirconia-toughened alumina; (2) measuring the mechanical properties of zirconia-toughened alumina; (3) testing the dielectric properties; (4) characterizing the thermophysical properties of ceramics; and (5) modeling the heating of an insulated ceramic body in a single-mode microwave cavity.

Keywords: Microwave Processing, Sintering, Zirconia-Toughened Alumina, Modeling

43. Chemical Vapor Infiltration of Ceramic Composites FY 1992

$300,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: T. M. Besmann
Georgia Institute of Technology Contact: Tom Starr, (404) 853-0579

This project is designed to develop continuous fiber reinforced TiB_2 and SiC matrix materials. TiB_2 materials may be useful as Hell-Heroalt aluminum smelting cathodes having substantially improved properties. Carbon cathodes currently require significant anode-to-cathode spacing in order to prevent shorting, causing significant electrical inefficiencies. The TiB_2 composite is fabricated through chemical vapor infiltration, producing a high purity TiB_2 matrix without damaging the relatively fragile plain-weave carbon fibers. Current efforts have focused on producing uniform composite specimens at the elevated temperatures required to prevent detrimental chlorine reaction. Resulting samples exhibited chemical stability in short exposures (24-48 hours) to molten aluminum. Efforts will continue to design and evaluate potential fiber reinforcements, fabricate test specimens and test the materials in a static bath and lab-scale Hall cell.

The purpose of the modeling efforts is to enable improved processing of chemical vapor infiltrated CVI ceramic composites. The structure of specimens produced during interrupted CVI experiments is evaluated by the use of X-ray tomographic methods (LLNL) and compared to model calculations. Correlations are determined between processing variables, including temperature and pressure, and properties, including strength and density.

Keywords: Chemical Vapor Infiltration, Composites, TiB_2, SiC, Cathodes, Modeling

44. Biomimetic Thin Film Synthesis FY 1992

$300,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Pacific Northwest Laboratory Contact: B. J. Tarasevich, (509) 375-2078

The objective of this project is to develop a method of forming artificial organic interfaces onto polymer and oxide substrates, and to demonstrate biomimetic thin film deposition of various materials. Organic molecules can be attached to substrates and used to promote deposition of materials from solution. This is a similar approach used by biological organisms in the formation of structural materials such as shells, bones, and teeth. The goal of this project is to tailor the organic interface to have various surface and group sites densities, site functionalities, and site structures in order to control the properties of the inorganic deposit such as orientation, phase, and deposition location. Protective coatings for corrosion, chemical and abrasion resistant applications are currently under investigation in cooperation with the automotive industry. In addition, oriented, anisotropic, magnetic iron oxide films are being developed for use as magnetic storage devices.

Keywords: Biomimetic, Organic Interfaces, Ceramic Coatings

45. Recoverable Thermosets

FY 1992
$100,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Polytechnic University Contact: G. Tesoro, (718) 643-5244 and Y. Wu

The principal objective of this project is to develop viable approaches to the recovery of materials of value from cured unsaturated polyester resin for the purpose of providing new avenues to industry participation and potential technology transfer efforts. Current research efforts focus on investigating the chemical reactions of cured unsaturated polyester yielding well characterized products, and identifying potential applications of compounds recovered. Neutral hydrolysis of cured unsaturated polyester resins was shown to be a viable approach to the solubilization of the thermosets and to the recovery of chemical compounds in good yields. In addition, potential applications of the recovered products have been explored with promising results.

Keywords: Thermoset Resins, Plastics Recycle

Device or Component Fabrication, Behavior or Testing

46. Advanced Engineering Coatings Development

FY 1992
$100,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: Peter Angelini, (615) 574-4565
K-25 Contact: Douglas Fain, (615) 574-9932
University of Wisconsin Contact: Marc A. Anderson, (608) 262-2674

The purpose of this work is to develop a semi-automated reproducible, economically attractive method for fabricating thin coatings as membrane materials for separation processes. Sol-gel techniques are used to deposit membranes as extremely thin layers on the surface of inexpensive support materials. These inorganic membranes can provide major new opportunities in the field of separations that include liquid-solid, liquid-liquid, and gas-gas systems. The project will also seek to develop a ceramic membrane characterized by photocatalytic activity that promotes self-cleaning of the membrane. A membrane fabricated from a semiconductor ceramic material will be developed that will utilize photons from a UV light source to degrade surface foulants that have accumulated on the surface and effectively clean the membrane.

Keywords: Membranes, Separations, Sol-Gel, Photocatalysis

47. Ni$_3$Al Technology Transfer: Castability and Weldability of Ni$_3$Al — FY 1992 $500,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contacts: M. L. Santella, (615) 574-4805 and V. K. Sikka, (615) 574-5112

Ductile Ni$_3$Al and Ni$_3$Al-based alloys have been identified for a range of applications. These applications require the use of materials in a variety of product forms such as sheet, plate, bar tubing, piping, wire and castings. Although significant progress has been made in the melting, casting and near net shaping of nickel aluminides, some important technological issues related to the processing, fabrication, and mechanical behavior of these alloys remain unsolved. The objective of this project is to facilitate the transfer of intermetallic alloy technology to industry by resolving technical issues constraining the industrial application of alloy systems and by providing a forum for exchange of data by industrial suppliers and users. The activities in this project also include one Cooperative Research and Development agreement with industry. This includes castability (fluidity, hot shortness, and porosity), weld repairability of castings, hot workability of cast ingots, and fracture toughness properties.

Keywords: Nickel Aluminides, Processing, Mechanical Properties

48. Advanced Microwave Processing Concepts — FY 1992 $200,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
ORNL Contact: R. J. Lauf, (615) 574-5176

The purpose of this project is to explore new techniques and uses for microwave processing, including multilayer ceramic capacitors and polymer-matrix composites. and to develop variable-frequency microwave sources since existing microwave sources are confined to extremely narrow frequency ranges. The new variable microwave process will permit (1) more efficient use of microwave energy in processing of materials by being able to operate at optimum frequency or change the frequency as the material is heated, and (2) more effective microwave field uniformity which allows for the uniform heating of single and multiple parts in furnaces. Current efforts are focused on the sintering of multilayer capacitors. Multilayer capacitors will provide an opportunity to study the effect of internet metallic phases on the behavior of parts during microwave processing.

Keywords: Microwave Processing, Multilayer Capacitors, Polymers, Composites, Variable Frequency

49. Chemically Specific Coatings — FY 1992 — $450,000

DOE Contact: Charles A. Sorrell, (202) 586-1514
Sandia National Laboratories Contacts: D. H. Doughty, (505) 845-8105 and A. C. Frye, (505) 844-0787

Chemically selective films have potential application in membrane, chromatographic separation and chemical sensor technologies. While many techniques exist to prepare chemically specific coatings, sol-gel processing offers substantial advantages in being able to tailor physical and chemical properties of the coatings. Pore size, pore size distribution, pore volume, surface chemistry and composition can be varied to provide optimal selectivity. Moreover, surface derivitizing reagents have been used to modify surface chemistry. The goal of this project is to develop a new class of chemical sensors through sol-gel processing that can be used a process monitors to improve process energy efficiency. Films have been applied to quartz oscillators in order to study the selectivity of their interactions with aqueous solutions. In addition, inorganic polymeric films have been applied to porous ceramic supports with the goal of preparing sol-gel metal oxide films for gas separation.

Keywords: Coatings, Sol Gel Processing

OFFICE OF TRANSPORTATION TECHNOLOGIES

	FY 1992
Office of Transportation Technologies - Grand Total	$33,777,000
Office of Transportation Materials	$14,897,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$6,409,000
High Temperature SX Silicon Carbide (WBS No. 1113)	312,000
Turbomilling of SiC (WBS No. 1116)	0
Powder Characterization (WBS No. 1118)	110,000
Sintered Silicon Nitride (WBS No. 1121)	0
Microwave Sintering (WBS No. 1124)	400,000
Cost Effective Silicon Nitride Powder (WBS No. 1125)	250,000
Novel Si_3N_4 Processing (WBS No. 1126)	36,000
Cost Effective Sintering of Silicon Nitride Ceramics (WBS No. 1127)	100,000
Cost Effective Manufacture of Silicon Nitride Components (WBS No. 1128)	250,000
Advanced Processing (WBS No. 1141)	1,654,000
Improved Processing (WBS No. 1142)	650,000
Processing Science for Reliable Structural Ceramics Based on Silicon Nitride (WBS No. 1144)	0
Property Optimization of Advanced Composites (WBS No. 1225)	283,000
In situ Toughened Silicon Nitride (WBS No. 1226)	543,000
Dispersion Toughened Oxide Composites (WBS No. 1231)	350,000
Low Expansion Ceramics (WBS No. 1242)	251,000
Low Thermal Expansion Ceramics (WBS No. 1243)	100,000
Low Cost CTE Components (WBS No. 1245)	200,000
Low Cost CTE Components (WBS No. 1246)	250,000
Alternative Fuel Lubricant Technology	0
Low-Wear Coatings for Transportation	275,000
HRRS Hard Coatings	100,000
IBAD of TiN and Cr_2O_3	0
Self-Lubricating Ceramic Surfaces	0
High-Current Ion Implantation	45,000
Lubricious Coatings for Transportation	250,000

OFFICE OF TRANSPORTATION TECHNOLOGIES

	FY 1992
Office of Transportation Materials (continued)	
Materials Properties, Behavior, Characterization or Testing	$4,712,000
Microstructural Modeling of Cracks (WBS No. 2111)	0
Adherence of Coatings (WBS No. 2212)	43,000
Dynamic Interfaces (WBS No. 2221)	0
Development of Standard Test Methods for Evaluating the Wear Performance of Ceramics (WBS No. 2222)	150,000
Advanced Statistic Calculations (WBS No. 2313)	106,000
Microstructural Analysis (WBS No. 3111)	50,000
Microstructural Characterization of Silicon Carbide and Silicon Nitride Ceramics for Advanced Heat Engines (WBS No. 3114)	200,000
Project Data Base (WBS No. 3117)	240,000
Fracture Behavior of Toughened Ceramics (WBS No. 3213)	290,000
Cyclic Fatigue of Toughened Ceramics (WBS No. 3214)	220,000
Tensile Stress Rupture Development (WBS No. 3215)	300,000
Rotor Materials Data Base (WBS No. 3216)	200,000
Toughened Ceramics Life Prediction (WBS No. 3217)	217,000
Life Prediction Methodology (WBS No. 3222)	0
Life Prediction Methodology (WBS No. 3223)	544,000
Environmental Effects in Toughened Ceramics (WBS No. 3314)	326,000
High Temperature Tensile Testing (WBS No. 3412)	218,000
Standard Tensile Test Development (WBS No. 3413)	125,000
Development of a Fracture Toughness Microprobe (WBS No. 3415)	0
Non-Destructive Evaluation (WBS No. 3511)	360,000
Computed Tomography (WBS No. 3515)	108,000
Nuclear Magnetic Resonance Imaging (WBS No. 3516)	65,000
Additives for High-Temperature Liquid Lubricants	100,000
Friction & Wear Mechanisms in *In-Situ*-Reinforced Ceramics	125,000
Carbon-Based Materials for Engine Applications	125,000
Advanced Materials/Additive Interactions	150,000
Liquid Lubricants for Heat Engines	150,000
Advanced Lubricants for Engine Particulate Control	150,000
Lubricious Oxide Coatings	150,000
Alternative Fuels-Compatible Materials	0

OFFICE OF TRANSPORTATION TECHNOLOGIES

	FY 1992
Office of Transportation Materials (continued)	
Technology Transfer and Management Coordination	$1,670,000
Management and Coordination (WBS No. 111)	950,000
International Exchange Agreement (IEA) (WBS No. 4115)	400,000
Standard Reference Materials (WBS No. 4116)	150,000
Mechanical Property Standardization (WBS No. 4121)	100,000
Diamond Coatings for Tribology	70,000
Device or Component Fabrication, Behavior or Testing	$2,106,000
Advanced Coating Technology (WBS No. 1311)	175,000
Coatings to Reduce Contact Stress Damage of Ceramics (WBS No. 1313)	66,000
Wear Resistant Coatings (WBS No. 1331)	224,000
Wear Resistant Coatings (WBS No. 1332)	0
Thick Thermal Barrier Coating Systems for Low Heat Rejection Diesel Engines (WBS No. 1342)	151,000
Active Metal Brazing PSZ-Iron (WBS No. 1411)	220,000
Metal-Ceramic Joints AGT (WBS No. 1412)	0
Ceramic-Ceramic Joints AGT (WBS No. 1421)	254,000
Next-Generation Grinding Wheel (WBS No. 1531)	70,000
High Speed Grinding (WBS No. 1532)	230,000
Chemically Assisted Grinding of Ceramics (WBS No. 1533)	150,000
Grindability Test (WBS No. 1542)	70,000
Grinding Consortium (WBS No. 1543)	150,000
Fluid-Film Bearings Model	69,000
Wear Mechanism Modeling	115,000
Instantaneous Friction Torque	25,000
Scale Effects in Friction Simulations	62,000
Thermomechanical Wear Model for Ceramics	0
Cylinder Kit Model Development	75,000

OFFICE OF TRANSPORTATION TECHNOLOGIES

	FY 1992
Office of Propulsion Systems	$18,460,000
Advanced Propulsion Division	$14,055,000
Materials Properties, Behavior, Characterization or Testing	$195,000
Ceramic Durability Evaluation AGT	195,000
Device or Component Fabrication, Behavior or Testing	$13,860,000
Thick Thermal Barrier Coatings	25,000
Thick Thermal Barrier Coatings	25,000
Advanced Diesel Engine Component Development Project	260,000
Advanced Piston and Cylinder Component Development	1,200,000
Advanced Piston and Cylinder Component Development	1,000,000
High Temperature Solid Lubricant Coatings	50,000
Advanced Turbine Technology Applications Project (ATTAP, AGT-5)	6,300,000
Advanced Turbine Technology Applications Project (ATTAP, AGT-101)	5,000,000
Electric and Hybrid Propulsion Division	$4,405,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$237,000
Corrosion-Resistant Coatings for High-Temperature High-Sulfur Activity Applications	132,000
Improved Container Electrode Coating for Na/S Battery Systems	105,000
Materials Structure and Composition	$466,000
New Battery Materials	78,000
Spectroscopic Studies of Passive Films on Alkali and Alkaline Earth Metals In Nonaqueous Solvents	55,000
In situ Spectroscopic Applications to the Study of Rechargeable Lithium Batteries	86,000

OFFICE OF TRANSPORTATION TECHNOLOGIES

	FY 1992
Electric and Hybrid Propulsion Division (continued)	
Materials Structure and Composition (continued)	
Raman Spectroscopy of Electrode Surface in Ambient-Temperature Lithium Secondary Battery	47,000
Battery Materials: Structure and Characterization	100,000
Zinc/Air Battery Development for Electric Vehicles	50,000
Polymeric Electrolytes for Ambient-Temperature Lithium Batteries	50,000
Materials Properties, Behavior, Characterization or Testing	$405,000
Corrosion, Passivity, and Breakdown of Alloys Used in High-Efficiency-Density Batteries	30,000
Advanced Chemistry and Materials for Fuel Cells	100,000
Electrocatalysts for Oxygen Electrodes	150,000
Novel Concepts for Oxygen Electrodes in Secondary Metal/Air Battery	125,000
Device or Component Fabrication, Behavior or Testing	$3,297,000
Proton-Exchange-Membrane Fuel Cells for Vehicles	1,400,000
Solid Polymer Electrolytes for Rechargeable Batteries	97,000
Electrochemical Energy Storage	1,800,000
Office of Alternative Fuels	$420,000
Materials Properties, Behavior, Characterization or Testing	$420,000
Cold-Start Assist Materials	120,000
CNG Adsorbents Demonstration	300,000

OFFICE OF TRANSPORTATION TECHNOLOGIES

The Office of Transportation Technologies seeks to develop, in cooperation with industry, technologies that are more energy-efficient and will enable the transportation sector to shift from near total dependence on petroleum to alternative fuels and electricity. Additional program goals are to increase the supply and availability of nonpetroleum fuels, and minimize the environmental impacts of transportation energy use. The Office of Transportation Technologies consists of the Office of Propulsion Systems, Office of Alternative Fuels, and Office of Transportation Materials, each having responsibility for specific technologies and program areas.

Office of Transportation Materials

The Office of Transportation Materials conducts research to develop an industrial technology base in transportation-related materials and materials processing in support of the Office of Transportation Technologies mission. R&D addresses materials needs for propulsion systems (heat engines, fuel cells, batteries, etc.); vehicle systems (chassis, body components) and fuel systems (fuel containment, emissions control, etc.). Materials development activities consist of four main programmatic elements: Ceramic Technology, Materials Technology (other than structural ceramics), Tribology, and the High Temperature Materials Laboratory.

The objective of the Ceramic Technology Program is to establish an industrial technology base for reliable structural ceramics and cost-effective manufacturing of ceramic components for high performance heat engines for transportation propulsion. A balanced program is conducted in the areas of materials processing, design methodology, and data base and life prediction. A majority of the research is conducted by industry. The Ceramic Technology Project is managed by the Oak Ridge National Laboratory (ORNL). The DOE contact is Robert Schulz, (202) 586-8051.

The Materials Technology Program seeks to develop improved materials and the associated processing to make such materials cost-effective for various transportation applications. The program focuses on: materials for batteries and fuel cells that will make electric and hybrid vehicles cost-competitive with current production vehicles; high-strength, lightweight structural materials for more fuel-efficient, lightweight vehicles that do not compromise the safety and comfort of passengers; cost-effective materials compatible with alternative fuels (alcohol, natural gas) for fuel systems, engine components, and exhaust systems as well as new emission control system materials; and transportation infrastructure materials ranging from low cost corrosion resistant materials to reduce the capital cost of large-scale biofuels (alcohol) plants, to maglev guideways, to more durable, crack resistant road and bridge construction materials. The DOE contacts are: Sidney Diamond, (202) 586-8032, for lightweight transportation materials, and Joseph M. Perez, (202) 586-8060, for alternative fuels-compatible materials.

The Tribology Program seeks to develop high-temperature lubricants and lubricant delivery systems as well as surface engineering and coating techniques to improve friction and wear properties of engine parts, and to develop techniques to mitigate friction and wear of lubricated and unlubricated ceramics needed to commercialize advanced heat engine concepts. The Tribology Program is managed by Argonne National Laboratory (ANL). The DOE contact is Joseph M. Perez, (202) 586-8060.

The High Temperature Materials Laboratory (HTML) at Oak Ridge, Tennessee, is a state-of-the-art research and user facility which supports Government and industry efforts in high temperature materials research and serves as a unique technology transfer vehicle through its user program. The HTML comprises six user centers: materials analysis, high temperature mechanical properties, high temperature x-ray diffraction, physical properties, ceramic specimen preparation, and residual stress measurements. The DOE contact is Sidney Diamond, (202) 586-8032.

Materials Preparation, Synthesis, Deposition, Growth or Forming

50. High Temperature SX Silicon Carbide (WBS No. 1113) — FY 1992 $312,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: E. L. Long, Jr., (615) 574-5172
Carborundum Contact: Roger S. Storm, (716) 278-2544

The major objectives for this program can be listed as follows: (1) to establish a mechanical property database for the current Hexoloy SX composition and (2) to further improve the mechanical properties via optimization of the composition, powder selection, dispersion/mixing conditions, and densification conditions.

Keywords: High Temperature Properties, Materials Processing, Silicon Carbide

51. Turbomilling of SiC (WBS No. 1116) — FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: T. N. Tiegs, (615) 574-5173
Southern Illinois University Contact: Dale E. Wittmer, (618) 536-2396

First, a small feasibility study was conducted to investigate the use of a unique turbomilling process in the preparation of SiC whisker-toughened ceramic composites. Due to the early success of the feasibility study, the scope of this project was expanded to evaluate the effect of turbomilling variables and the beneficiation of SiC whiskers, examine the dispersion/homogenization of SiC whisker/alumina composites and SiC whisker/silicon nitride, and investigate loadings for reducing aspect ratios in the absence of coarse grinding particulate.

Keywords: Alumina, Silicon Carbide, Silicon Nitride, Turbomilling, Whiskers

52. Powder Characterization (WBS No. 1118) — FY 1992 $110,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
National Institute of Standards and Technology (NIST) Contact: S. Malghan, (301) 975-2000

This effort is directed toward developing a fundamental understanding of surface chemical changes which take place when silicon nitride powder is attrition milled in an aqueous environment.

This project also will demonstrate the use of and establish operating conditions for high energy attrition milling of silicon nitride powder. These tasks will be accomplished by developing measurement techniques and data on the effect of milling variables on the resulting powder. It is expected that information gained from this study will serve in the identification and development of appropriate characterization procedures, process control techniques, and in certification of new Standard Reference Materials.

Keywords: Powder Characterization, Powder Processing, Reference Material, Silicon Nitride

53. Sintered Silicon Nitride (WBS No. 1121) — FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
AMTL Contact: G. E. Gazza, (617) 923-5408

The overall objective of this effort is to develop scale up processing conditions for a silicon nitride having the general composition 85.8 mol % Si_3N_4-4.73 mol % Y_2O_3-9.47 mol % SiO_2-1.0 mol % Mo_2C and characterizing the properties of this composite with the goal of producing complex components for testing in related heat-engine programs. The first task of the program has concentrated on processing studies and database generation, while the second task will focus on producing and characterizing engine components. Ceradyne, Inc., is conducting this work, with support from AMTL.

Keywords: Sintering, Silicon Nitride, Testing

54. Microwave Sintering (WBS No. 1124) FY 1992
$400,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: T. N. Tiegs, (615) 574-5173

The objective of this effort is to identify those aspects of microwave processing of silicon nitride that might (1) accelerate densification, (2) permit sintering to high density using much lower levels of sintering aids, (3) lower the sintering temperature, or (4) produce unique microstructures.

The investigation of microstructure development is being done on dense silicon nitride materials annealed in the microwave furnace. The sintering of silicon nitride involves two approaches. In the first approach, silicon nitride and sialon powder compositions are heated in the 2.45- or 28-GHz units. The second approach deals with using reaction-bonded silicon nitride as the starting material and is done entirely in the 2.45-GHz microwave furnace.

Keywords: Microwave Sintering, Silicon Nitride

55. Cost Effective Silicon Nitride Powder (WBS No. 1125) FY 1992
$250,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: S. G. Winslow, (615) 574-0965
Dow Contact: G. A. Eisman, (517) 638-7864

The objective of this new effort is to develop a commercial, domestic source of high-quality, low-cost ($10/lb sale price) silicon nitride powder with suitable properties for forming into components for heat engine applications. There are five technical tasks: (1) reference process flow sheet and cost estimate, (2) process development and scale up, (3) powder and sintered part characterization, (4) final process flow sheet and cost estimate, and (5) process demonstration.

Keywords: Cost Effective Ceramics, Silicon Nitride, Powder Synthesis, Powder Characterization

56. Novel Si_3N_4 Process (WBS No. 1126) FY 1992
$36,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: E. L. Long, Jr., (615) 574-5172
Sullivan Mining Corporation Contact: Thomas M. Sullivan, (317) 889-3855

The objective of this effort is to demonstrate the scalability of the Sullivan™ Process for making silicon nitride, to develop unique low- and high-temperature versions of the silicon nitride, to determine the net-shape capability of the process, and to characterize the microstructural, mechanical, tribological, and physical properties of the Sullivan™ Process silicon nitride. The intent of this project is to demonstrate that the Sullivan™ Process can be operated on a commercial scale to make significantly less costly net-shape ceramics with superior properties.

Keywords: Materials Processing, Silicon Nitride

57. Cost Effective Sintering of Silicon Nitride Ceramics (WBS No. 1127) FY 1992
$100,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: T. N. Tiegs, (615) 574-5173
Southern Illinois University Contact: D. E. Wittmer, (618) 453-7006/7924

The objective of this effort is to investigate the potential of cost effective sintering of Si_3N_4 through the development of continuous sintering techniques and the use of lower cost Si_3N_4 powders and sintering aids. The effect of heating rate on the densification, microstructure, and properties of silicon nitride will be investigated. The effects of using alternate materials for the furnace belt, and modifications in the furnace design to improve both furnace load and rate of throughput will also be determined.

Keywords: Cost Effective Ceramics, Silicon Nitride, Sintering

58. Cost Effective Manufacture of Silicon Nitride Components (WBS No. 1128) FY 1992
$250,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: R. L. Beatty, (615) 574-4536
Coors Contact: Jack Sibold, (303) 271-7164

The objective of this effort is to develop a low-cost process for manufacture of high quality ceramic engine components based on sintered reaction bonded silicon nitride (SRBSN) technology. There are three technical tasks which address the areas of low-cost materials and processes and achievement of properties required for reliable performance. The material property goals for Phase I of this effort are a mean RT four-point flexure strength of 525 MPa and a Weibull modulus of 15.

Keywords: Cost Effective Ceramics, Silicon Nitride, Sintered Reaction Bonded Silicon Nitride

59. Advanced Processing (WBS No. 1141) FY 1992
$1,654,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: R. L. Beatty, (615) 574-4536
Norton Contact: D. M. Tracey, (508) 393-5811

The purpose of this task is to develop and demonstrate significant improvements in processing methods, process controls, and nondestructive examination (NDE) which can be commercially implemented to produce high-reliability silicon nitride components for advanced heat engine applications at temperatures to 1370°C.

A silicon nitride-4% yttria composition consolidated by glass encapsulated HIPing will be used. Baseline data shall be generated from an initial process route involving injection molding. Tensile test bars will be fabricated using colloidal techniques—injection molding and colloidal consolidation. Critical flaw populations will be identified using NDE and fractographic analysis. Measured tensile strength will be correlated with flaw populations and process parameters. Flaws will be minimized through innovative improvements in process methods and controls.

Keywords: Nondestructive Evaluation, Silicon Nitride, Processing, Processing Controls

60. Improved Processing (WBS No. 1142) FY 1992
$650,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: S. D. Nunn, (615) 576-1668

The purpose of this work is to determine and develop the reliability of selected advanced ceramic processing methods. This program is being conducted on a scale that will permit the potential for manufacturing use of candidate processes to be evaluated. An effort is being made to develop processes that can be scaled most readily to high production rates. Simplicity of processing and high predictability of product quality are dominant issues. The studies are intended to generate processing schedules and procedures as well as protocols for characterization of raw and in-process materials. The principal material of interest in this work is silicon nitride. Gel-casting, a method developed at ORNL, was the process chosen for initial consideration.

Production of tensile and other specimens of Al_2O_3 has demonstrated the potential for process predictability and dimensional control. A collaborative agreement with Garrett Ceramic Components Division to develop the process for silicon nitride was initiated in FY 1990.

Keywords: Alumina, Powder Processing, Silicon Nitride

61. Processing Science for Reliable Structural Ceramics Based on Silicon Nitride (WBS No. 1144) — FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: R. L. Beatty, (615) 574-4536
University of California, Santa Barbara, Contact: Fred F. Lange, (805) 893-8248

The current objective of this effort is to try to increase the understanding of the role of interparticle forces in the processing of ceramics. In particular, short-range repulsive forces are being created. Utilizing these forces, the rheological properties of dispersions, the kinetics of pressure filtration, and the mechanical properties and microstructure of the resulting bodies can be modified. Both aqueous and non-aqueous systems are being studied. For non-aqueous systems, the reaction of alcohol with the surface of silicon nitride is being explored. In aqueous systems, attempts are being made to make the surface of silicon nitride appear more alumina-like by coating it with an alumina layer.

Keywords: Alumina, Powder Processing, Silicon Nitride

62. Property Optimization of Advanced Composites (WBS No. 1225) — FY 1992 $283,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
University of Michigan Contact: T. Y. Tien, (313) 764-9449

The purpose of this effort is to optimize the properties of silicon nitride by microstructural design, specifically by developing fiber-like β-Si_3N_4 grains and control of the grain-boundary phase. Optimization of the silicon carbide ceramics will be accomplished by formation of composites containing AlN polytypoids.

Keywords: Composites, Physical/Mechanical Properties, Silicon Nitride, Silicon Carbide

63. *In situ* Toughened Silicon Nitride (WBS No. 1226) FY 1992
$543,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: T. N. Tiegs, (615) 574-5173
Garrett Contact: H. C. Yeh, (213) 618-7449

The purpose of this effort is to develop compositions and processes to obtain high fracture toughness and strength for silicon nitride (Si_3N_4)-based ceramic materials through microstructure control. The work is divided into two stages. The first is a refinement stage focusing on the effects and interactions of the chemical composition and thermal processing variables on microstructure, mechanical behavior, and oxidation resistance. The second stage will be an optimization stage focusing on the development of in situ reinforced Si_3N_4 with optimized microstructure and properties which meet or exceed the property goals and on the establishment of composition-processing-property correlations. A simulated engine component will be fabricated in order to demonstrate process feasibility.

Keywords: Physical/Mechanical Properties, Silicon Nitride, Toughened Ceramics

64. Dispersion Toughened Oxide Composites (WBS No. 1231) FY 1992
$350,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: T. N. Tiegs, (615) 574-5173

This work involves development and characterization of SiC whisker-reinforced oxide composites for improved mechanical performance. To date most of the efforts involving SiC whisker-reinforced alumina, mullite, silicon nitride, and sialon have been completed. In addition, studies of whisker-growth processes were conducted to improve the mechanical properties of SiC whiskers by reducing their flaw sizes and, thereby, improving the mechanical properties of the composites. Currently, in situ acicular grain growth is being investigated to improve fracture toughness of silicon nitride materials.

Keywords: Composites, Alumina, Silicon Carbide, SiAlON, Toughened Ceramics

65. Low Expansion Ceramics (WBS No. 1242) FY 1992
$251,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: Victor J. Tennery, (615) 574-5123
Virginia Polytechnic Institute and State University Contact: J. J. Brown, (703) 961-6640

A major objective of this research is to investigate selected oxide systems for the development of a low expansion, high thermal shock resistant ceramic. Specifically, it is the goal of this study to develop an isotropic, ultra-low thermal expansion ceramic which can be used above 1200°C and which is relatively inexpensive, and to determine conditions necessary for synthesis, densification, and characterization of these systems.

Two low thermal expansion compositions based on the zircon (NZP) and the β-eucryptite-$AlPO_4$ systems, respectively, have been selected for optimization. The major objective is to demonstrate fabricability and to promote commercialization of these ceramics.

Keywords: Structural Ceramics, Aluminum Phosphate, Zirconia, Ultra-low Expansion, Beta-eucryptite, Physical/Mechanical Properties

66. Low Thermal Expansion Ceramics (WBS No. 1243) FY 1992 $100,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. R. Johnson, (615) 576-6832
ORNL Contact: D. P. Stinton, (615) 574-4556

The objective of this effort is to develop a work plan and coordinate efforts regarding the application of low-expansion ceramics in advanced heat engines.

Keywords: Alumina, Beta-eucryptite, Phosphate, Physical/Mechanical Properties, Structural Ceramics, Ultra-low Expansion

67. Low Cost CTE Components (WBS No. 1245) FY 1992 $200,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. P. Stinton, (615) 574-4556
LoTEC, Inc., Contact: Santosh Limaye, (801) 277-6940

The purpose of this new effort is to scale up the work done at universities and develop materials that can be used as exhaust port liners in diesel engines and regenerators in automotive turbine engines. LoTEC will develop and scale up production of sodium-zirconium-phosphate (NZP) materials developed at Penn State University.

Keywords: Structural Ceramics, Ultra-low Expansion, Zirconia

68. Low Cost CTE Components (WBS No. 1246) FY 1992 $250,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. P. Stinton, (615) 574-4556
Coors Contact: R. N. Kleiner, (303) 277-4739

The purpose of this new effort is to develop materials technology and fabrication technology for diesel engine exhaust port liners. Coors will scale up production of a unique NZP material developed at Virginia Polytechnic Institute and State University.

Keywords: Structural Ceramics, Ultra-low Expansion, Zirconia

69. Alternative Fuel Lubricant Technology FY 1992 $0

DOE Contact: J.M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Penn State University Contact: E.E. Klaus (814) 863-4804

Technological solutions aimed at reducing the environmental concerns of both automotive and diesel engines have resulted in the use of a variety of alternative fuels that are being researched by industry as relevant to future power plants to reduce emissions. However, major concerns exist with respect to the compatibility of the lubricants with the new fuels. The ability of the additives to remain soluble and perform their designed function is a problem with some of the leading alternative technologies. This activity will work cooperatively with industry in resolving the lubricant issues. The use of methanol and ethanol in automotive and diesel engines is a highly visible alternative to reducing atmospheric pollution in highly populated centers. The field testing of a number of diesel- and gasoline-powered vehicles is in progress in several areas of the U.S. and Canada. The lubricant problems are significant in a number of these studies and a timely solution is required.

Keywords: Coatings and Films, Chemical Vapor Deposition, Lubrication, Ceramics, Alternative Fuels

70. Low-Wear Coatings for Transportation FY 1992 $275,000

DOE Contact: J.M. Perez, (202) 586-8060
ANL Contact: Fred Nichols, (708) 252-8292

The recent reorganization of the Tribology Program from ECUT to the Office of Transportation Technologies places greater emphasis on working collaboratively with, and transferring technology and information obtained in this program to industrial organizations that are involved in the development of advanced energy-conserving engines. The objective of this activity focuses on this aspect. Meetings will be held with industrial organizations that are either directly involved in the development of low heat rejection engines (LHREs), advanced gas turbines, or associated with these projects by virtue of providing components for these engines. These meetings will focus on developing collaborative research programs to transfer the technologies developed under the ETI task to industry. Examples of these efforts include programs with Caterpillar Inc, Cummins Engine Company Inc., the Allison Gas Turbine Division of General Motors Corp., and a tentative program with Detroit Diesel Corp.

Keywords: Ion-Beam-Assisted Deposition, Coatings, Tribology, Friction, Wear, Heat Engines, Diesels, Gas Turbines

71. HRRS Hard Coatings — FY 1992 $100,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
BIRL Contact: Raymond Fessler, (708) 491-4941

The high-rate reactive-sputtering (HRRS) process developed in the hard-coatings-for-cutting-tools program showed great promise for producing well-adherent, hard, wear-resistant coatings on steel substrates. Significant increases in the cutting lifetimes of coated inserts were observed; however, the wear mechanisms and in particular the effect of frictional heating on the coating failure are not fully understood. In the upcoming year, rolling-contact fatigue (RCF) samples will be coated with two reactively sputtered materials, TiN and TiC. These coated samples will be run in RCF and scuffing tests. The coatings will be characterized by scanning and transmission electron microscopy, Auger electron spectroscopy, x-ray diffraction, microhardness, and scratch-adhesion tests. Emphasis will be placed on understanding the failure mechanism in the coated RCF samples. Coated samples will also be supplied to ANL for high-temperature pin-on-disc tests to characterize their friction and wear performance during dry sliding.

Keywords: High-Rate Reactive Sputtering, Coatings, Friction, Wear

72. IBAD of TiN and Cr_2O_3 — FY 1992 $0

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
NRL Contact: Fred Smidt, (202) 767-4800

The objective is to determine the mechanism by which ion-beam-assisted deposition (IBAD) produces beneficial modifications of tribological coatings and to establish the necessary correlations between processing parameters, microstructural features of the coating and tribological properties such as friction, wear and adhesion. General principles for producing the improved coatings will then be defined for the application of these coatings to advanced energy-system requirements. Technical activities are virtually complete. Effort this year will concentrate on preparation of a Final Report.

Keywords: Surface Modification, Coatings, Friction, Wear, Ion Assisted Deposition

73. Self-Lubricating Ceramic Surfaces | FY 1992 $0

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Universal Energy Systems Contact: Rabi Bhattacharya, (513) 426-6900

The objective of this project at Universal Energy Systems, Inc. is to establish optimal conditions for ion implantation and ion-beam mixing of suitable additives into the surfaces of bulk ZrO_2, Al_2O_3, and hardened steel for obtaining self-lubricating, low friction-and-wear characteristics. The work to be performed in the future will focus on investigations of BaF_2/Ag coatings. Sputtering of a composite CaF_2 + Ag film will be made to obtain a coating that will be subsequently ion-mixed using MeV ions. Friction and wear of this coating will be evaluated by ANL at room temperature and high temperature. A Final Report will be prepared.

Keywords: Surface Modification, Coatings, Solid Lubricants, Friction, Wear, Ion Implantation, Ion Beam Mixing, Ceramics

74. High-Current Ion Implantation | FY 1992 $45,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
CO State Univ Contact: Prof. Paul Wilbur (303) 491-8564

Ion implantation is a technology that has great potential for industrial applications in the field of tribology. One problem that is hindering wider acceptance of this technology is the long time it takes to implant surfaces due to low current densities of most commercial implanters. The objective of this program is to develop a high-current ion source in order to achieve low-cost, rapid ion implantation of surfaces. A unique broad beam (10 cm diameter) ultrahigh current density (1500 $\mu A/cm^2$) gas-ion implanter has been developed and successfully operated at Colorado State University using ion-rocket thruster technology. The research to be performed in this program will attempt to extend this ultrahigh-current-density technology to develop a unique high-current-density metal-ion implantation system. Another objective of this research is to investigate the effects of ultrahigh-current-density implantations on the tribological properties of the treated surfaces. These studies will establish permissible time-temperature relationships (during ion implantation) that do not cause deleterious changes.

Keywords: Surface Modification, Coatings, Solid Lubricants, Friction, Wear, Ion Implantation, Ion Beam Mixing, Ceramics

75. Lubricious Coatings for Transportation FY 1992 $250,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292

This activity focuses on the development of low-friction coatings on engineering surfaces. The program involves efforts to develop processes, to characterize films by electron microscopy and mechanical testing techniques, and to evaluate their tribological performance under different conditions. During FY92, tribological testing of IBAD Ag-coated ceramic substrates will continue to characterize the effects of processing parameters on tribological performance of coatings. Effects of load (stress), sliding velocity (particularly high speed) and sliding distance will be investigated. The substrates selected will focus on advanced ceramics such as silicon nitride, silicon carbide, alumina, and zirconia. Other metals and coating processes will also be studied.

Keywords: Surface Modification, Coatings, Solid Lubricants, Friction, Lubricious Coatings

Materials Properties, Behavior, Characterization or Testing

76. Microstructural Modeling of Cracks (WBS No. 2111) FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
University of Tennessee Contact: J. A. M. Boulet, (615) 974-2171

A goal of this research is to develop mathematical procedures by which existing design methodology for brittle fracture could accurately account for the influence of non-planar crack faces on fracture of cracks with realistic geometry under arbitrary stress states. Existing models all consider the crack faces to be smooth and planar, but examination of fresh fracture surfaces by microscopy often indicates rough, irregular surfaces. In this study, the effect of protrusion interference on fracture of cracks with realistic geometry under arbitrary stress states is being examined.

Keywords: Predictive Behavior Modeling, Structural Ceramics

77. Adherence of Coatings (WBS No. 2212) FY 1992 $43,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: L. L. Horton, (615) 574-5081

Financial support is provided for a graduate research assistantship in the Department of Materials Science and Engineering at the University of Tennessee to conduct studies on the effects of ion bombardment on the structure of thin ceramic films on ceramic substrates.

Keywords: Adherence, Ion Beam, Coatings and Films, Structural Ceramics

78. Dynamic Interfaces (WBS No. 2221) FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: E. L. Long, Jr., (615) 574-5172
Battelle Contact: K. F. Dufrane, (614) 424-4618

The objective of this study is to develop an understanding of the friction and wear processes of ceramic interfaces based on experimental data. The supporting experiments are conducted at temperatures to 650°C under reciprocating sliding conditions reproducing the loads, speeds, and environment of the ring/cylinder interface of advanced internal combustion engines. The test specimens are carefully characterized before and after testing to provide detailed input into the model.

The current direction of this program is to conduct wear tests with a variety of advanced coatings on actual engine components instead of using specially prepared specimens.

Keywords: Dynamic Interfaces, Wear, Structural Ceramics, Coatings, Predictive Behavior Modeling, Monolithics, Adiabatic Diesels, Friction, Lubrication

79. Development of Standard Test Methods for Evaluating the Wear Performance of Ceramics (WBS No. 2222) FY 1992 $150,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. R. Johnson, (615) 576-6832
ORNL Contact: P. J. Blau, (615) 574-5377

The goal of this effort is to improve consistency in reporting ceramic wear test data by helping to develop one or more standard test methods for quantitatively determining the wear resistance of structural ceramics in reciprocating sliding, a type of motion which is experienced by several types of engine parts.

Keywords: Structural Ceramics, Test Procedures, Wear

80. Advanced Statistics Calculations (WBS No. 2313) FY 1992 $106,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
GE Contact: C. A. Johnson, (518) 387-6421

The design and application of reliable load-bearing structural components from ceramic materials requires a detailed understanding of the statistical nature of fracture in brittle materials. The overall objective is to advance the current understanding of fracture statistics, especially in the areas of optimum testing plans and data analysis techniques, consequences of time-dependent crack growth on the evolution of initial flaw distributions, and confidence and tolerance bounds on predictions that use the Weibull distribution and function. The studies are being carried out largely by analytical and computer simulation techniques. Actual fracture data are then used as appropriate to confirm and demonstrate the resulting data analysis techniques.

Keywords: Design Codes, Life Prediction, Statistics, Weibull, Fracture, Structural Ceramics, Instrumentation or Technique Development

81. Microstructural Analysis (WBS No. 3111) FY 1992 $50,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
National Institute of Standards and Technology (NIST) Contact: S. M. Wiederhorn, (301) 975-5772

The objective of this work is to identify the mechanisms of failure in structural ceramics subjected to mechanical loads in various test temperatures and environments. This is a companion project to a related task in which advanced ceramics are characterized in tensile creep. Of particular interest is the damage that accumulates in structural ceramics as a consequence of high temperature exposure to environments and stresses normally present in heat engines. Materials to be studied include sialons, silicon nitride, and sintered silicon carbide.

Keywords: Corrosion, Engines, Erosion, Structural Ceramics, Silicon Carbide, Creep, SiAlON, Silicon Nitride

82. Microstructural Characterization of Silicon Carbide and Silicon Nitride Ceramics for Advanced Heat Engines (WBS No. 3114) FY 1992 $200,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: T. A. Nolan, (615) 574-0811

The purpose of this work is to determine the microstructure of both monolithic and composite ceramics and to relate that microstructure to mechanical properties and material performance. Specifically, the materials of interest are silicon carbides and silicon nitrides developed by U.S. manufacturers as part of this program and the ATTAP. A major objective is to use electron microscopy and surface chemistry to characterize the chemistry, crystallography, and morphology of phases present with particular emphasis on the structure and chemistry of grain boundaries and other interfaces.

A second major objective is to relate those microstructural observations to available mechanical test data produced by other participants in the ATTAP and Ceramic Technology programs. Ceramic specimens from foreign sources are also characterized to provide comparative information on microstructural properties.

Keywords: Silicon Carbide, Silicon Nitride, Microstructure, Chemical Analysis, Mechanical Properties, Scanning Electron Microscopy

83. Project Database (WBS No. 3117) FY 1992 $240,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: B. L. Keyes, (615) 574-5113

This task involves efforts to develop and maintain a computer data base of mechanical property data generated in the Ceramic Technology Project. The data base system is currently composed of a loosely-connected framework of commercially available programs. However, data can be easily transferred by electronic means to a variety of other programs. Techniques have been developed that allow data as compiled on a variety of computers using several different software programs to be transferred directly into the data base electronically with no manual transcription of the data. Several techniques for data output in useful formats (tabular and graphical) have also been developed. The system has been designed to provide maximum flexibility to allow the addition of other data as the data base grows.

Reports containing mechanical properties and material characterization information on silicon nitride, silicon carbide, Al_2O_3- and zirconia-based, and transformation-toughened ceramics as well as test data files on brazed specimen torsion strength and torsion fatigue and tribology data have been issued.

Keywords: Database, Mechanical Properties, Structural Ceramics

84. Fracture Behavior of Toughened Ceramics (WBS No. 3213) FY 1992 $290,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: P. F. Becher, (615) 574-5157

The purpose of this work is to characterize the high-temperature strength, toughness, and creep response of advanced structural ceramics for heat engine applications. Studies will be aimed at providing fundamental insights into the processes responsible for toughness and time-dependent strength degradation of materials such as silicon carbide, silicon nitride, and dispersion-toughened and whisker-reinforced ceramics. Particular emphasis is given to understanding the effects of environment, temperature, and how the materials can be improved by systematic control of microstructure and composition.

Keywords: Toughened Ceramics, Silicon Carbide, Silicon Nitride, Alumina

85. Cyclic Fatigue of Toughened Ceramics (WBS No. 3214) FY 1992 $220,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: K. C. Liu, (615) 574-5116

The objective of this task is to develop and demonstrate the capability of performing uniaxial tension-tension dynamic fatigue testing of structural ceramics at elevated temperature. The effort includes (1) design, fabrication, and demonstration of a load-train column capable of concentric load transfer between grip and specimen at high temperature; and (2) development of the baseline information on the tensile fatigue behavior of structural ceramics at room and elevated temperatures.

Keywords: Cyclic Fatigue, High Temperature Properties, Toughened Ceramics, Tensile Testing, Silicon Nitride

86. Tensile Stress Rupture Development (WBS No. 3215) FY 1992
$300,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: K. C. Liu, (615) 574-5116

The objective of this task is to develop the test capability for performing uniaxial tensile stress-rupture and creep tests on candidate structural ceramics. Existing data requirements specify that such data be developed in the range of 1260°C to 1371°C. These data bases are to be generated using cylindrical bar specimens of materials such as silicon nitride with the tests conducted in an air environment. Ten standard uniaxial level arm creep frames have been modified to incorporate the specimen load train with a self-aligning feature and closed-loop control capability. Time-dependent strain deformation occurring in the gage section will be measured during the test using a laser-based optical strain extensometer.

Keywords: Creep, Silicon Nitride, High Temperature Properties, Tensile Testing, Time-Dependent

87. Rotor Materials Data Base (WBS No. 3216) FY 1992
$200,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: M. K. Ferber, (615) 576-0818

The goal of this research program is to systematically study the tensile strength of silicon nitride ceramics as a function of temperature and time in an air environment. The research involves both test development and measurement of the creep/fatigue properties. The test development (alumina tensile specimens) phase will focus upon the establishment of procedures required for accurate measurement of specimen deflection using existing mechanical extensometers. In the second phase, stress-rupture data will be generated for silicon nitride specimens by measuring fatigue life at constant stress. The time-dependent deformation will also be monitored during testing so that the extent of high-temperature creep may be ascertained. Tested samples will be thoroughly characterized using established ceramographic, SEM, and TEM techniques. The resulting stress-rupture data will be used to examine the applicability of a generalized fatigue-life (slow crack growth) model.

Keywords: Alumina, Creep, Engines, High Temperature Service, Structural Ceramics, Tensile Testing, Predictive Behavior Modeling, Silicon Nitride, Scanning Electron Microscopy

88. Toughened Ceramics Life Prediction (WBS No. 3217) FY 1992
$217,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
NASA - Lewis Research Center Contact: Stanley R. Levine, (216) 433-3276

The purpose of this effort is to understand the room-temperature and high-temperature behavior of toughened ceramics as the basis for developing a life prediction methodology. A major objective is to understand the relationship between microstructure and mechanical behavior within the bounds of a limited number of materials. A second major objective is to determine behavior as a function of time and temperature. Specifically, strength and reliability, fracture toughness, slow crack growth, and creep behavior will be determined as a function of temperature for the as-manufactured material. The same properties will also be evaluated after long-time exposure to various high-temperature isothermal and cyclic environments. These results will provide input for parallel materials development and design methodology programs. Resultant design codes will be verified.

Keywords: Creep, Fracture Toughness, High Temperature Properties, Life Prediction, Silicon Nitride, Time-Dependent

89. Life Prediction Methodology (WBS No. 3222) FY 1992
$0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: C. R. Brinkman, (615) 574-5106
Allison Gas Turbine Division Contact: N. J. Provenzano, (313) 230-3150

The objective of this effort is to develop and demonstrate the necessary nondestructive examination (NDE) technology, material data base, and design methodology for predicting the useful life of structural ceramic components of advanced heat engines. The analytical methodology will be demonstrated through confirmatory testing of ceramic components subject to thermal-mechanical loading conditions similar to those anticipated to occur in actual vehicular service. The project addresses fast fracture, slow crack growth, creep, and oxidation failure modes.

Keywords: Creep, Failure Analysis, Failure Testing, Oxidation, Life Prediction, Nondestructive Evaluation, Silicon Nitride

90. Life Prediction Methodology (WBS No. 3223) FY 1992
$544,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: C. R. Brinkman, (615) 574-5106
Garrett Auxiliary Power Division Contact: John Cuccio, (602) 220-3600

The objective of this effort is to develop the methodology required to adequately predict the useful life of ceramic components used in advanced heat engines. Various specimen geometries will undergo comprehensive testing under both uniaxial and multiaxial loads at different environmental conditions to determine the strength-controlling flaw distributions and to identify various failure mechanisms. NDE techniques will be correlated with failure analyses of the test specimens to help determine the flaw distributions. This information will be used to develop the flaw distribution statistical model and material behavior models for fast fracture, slow crack growth, creep deformation, and oxidation. As subroutines, these models will be integrated with stress and thermal analyses into a failure risk integration analytical tool to predict the life of ceramic components. The methodology developed will be verified by analytically predicting the life of several ceramic components and testing these components under stress and temperature conditions encountered in ceramic turbine engines. Correlation of the NDE data will be used to predict the flaw populations/sizes in the verification components.

Keywords: Creep, Failure Analysis, Failure Testing, Life Prediction, Nondestructive Evaluation, Oxidation, Silicon Nitride

91. Environmental Effects in Toughened Ceramics (WBS No. 3314) FY 1992
$326,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: Victor J. Tennery, (615) 574-5123
University of Dayton Contact: N. L. Hecht, (513) 229-4341

The objective of this task is to characterize the mechanical behavior and environmental degradation processes operative in candidate AGT ceramics using flexural and uniaxial tensile testing and extensive pre- and post-test characterization of physical properties and microstructure.

Flexural strength is being measured over a wide range of stressing rates, temperatures, and atmospheric conditions to quantitatively determine relevant fatigue parameters.

Keywords: Fatigue, Engines, Structural Ceramics, Environmental Effects, Alumina, Zirconia, Diesel Combustion, Tensile Testing, Time-Dependent, Transformation-Toughened

92. High Temperature Tensile Testing (WBS No. 3412) FY 1992
$218,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
North Carolina A&T State University Contact: J. Sankar, (919) 334-7620

The objective of this research is to test and evaluate the mechanical behavior of a sintered Si_3N_4 in uniaxial tension at temperatures up to 1200°C. Currently, the emphasis is on analyzing the influence of precycling on the creep behavior and the tensile strength of GTE SNW-1000 at 1200°C, and to compare these results with previously obtained creep data for that same material. Microstructural/microchemical analysis of the fracture surfaces using scanning electron microscopy (SEM) and energy-dispersive spectral analysis (EDS) is an integral part of this effort.

Keywords: Creep, Fracture, Silicon Nitride, Structural Ceramics, Tensile Testing

93. Standard Tensile Test Development (WBS No. 3413) FY 1992
$125,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
National Institute of Standards and Technology (NIST) Contact: S. M. Wiederhorn, (301) 975-5772

This project is concerned with the development of test equipment and procedures for measuring the strength and creep resistance of ceramic materials at elevated temperatures to assist in the development of a reliable data base for use in the structural design of heat engines for vehicular applications.

Inexpensive techniques for tensile creep are being developed at NIST. These methods use self-aligning test fixtures and simple grinding procedures for specimen preparation. Once the test methods have been fully analyzed, a methodology for assuring long-term reliability of structural ceramics at elevated temperatures will be developed.

Keywords: Creep, High Temperature Properties, Structural Ceramics, Tensile Testing, Test Procedures

94. Development of a Fracture Toughness Microprobe (WBS No. 3415) — FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: W. C. Oliver, (615) 576-7245
Rice University Contact: G. M. Pharr, (713) 527-8101

The objective of this effort is to develop a technique for measuring fracture toughness in thin films and small volumes on a spatially-resolved basis. The Mechanical Properties Microprobe will be used to develop the technique.

Keywords: Fracture Toughness, Structural Ceramics, Testing

95. Non-Destructive Evaluation (WBS No. 3511) — FY 1992 $360,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: D. J. McGuire, (615) 574-4835

The purpose of this program is to develop nondestructive evaluation (NDE) techniques in order to identify approaches for quantitative determination of conditions in ceramics that affect the structural performance. High-frequency ultrasonics and radiography are being used to detect, size, and locate critical flaws and to nondestructively determine selected physical or mechanical properties (e.g., density, elastic properties, fracture toughness) of the host material. Materials to be incorporated include monolithic ceramics such as Si_3N_4 and SiC as well as ceramic composites and the studies will address problems unique to various fabrication techniques such as sintering, hot isostatic pressing, gel casting, etc.

Keywords: NDE, Radiography, Structural Ceramics, Ultrasonics

96. Computed Tomography (WBS No. 3515) — FY 1992 $108,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
Argonne National Lab Contact: W. A. Ellingson, (312) 972-5068

The overall purpose of this program is to develop X-ray computed tomographic (XCT) imaging for characterizing structural ceramic materials relative to density distributions and the presence of voids, inclusions, and cracks and, further, to relate the detected variations to performance or processing variations. Currently, this technique is being used to study density distributions and other possible variations in composite green-state (as-cast) pressure slip-cast billets supplied by Garrett Ceramic Components Division of Allied-Signal Aerospace Company.

Garrett will then perform destructive analysis of the as-cast billets and their findings will be correlated with the 3D X-ray microtomography data.

Keywords: Computed Tomography, Nondestructive Evaluation, Silicon Carbide, Silicon Nitride, Structural Ceramics, Green State

97. Nuclear Magnetic Resonance Imaging (WBS No. 3516) — FY 1992 $65,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
Argonne National Laboratory Contact: W. A. Ellingson, (312) 972-5068

The purpose of this work is to determine the feasibility of using nuclear magnetic resonance (NMR) systems (spectroscopy and imaging) to: (a) measure the distribution of organic binder/plasticizer (B/P) systems in various stages of the injection molding of Si_3N_4 parts; and (b) study slips and slip-mold interactions during composite slip-casting processes.

Currently the effort is focused on relating NMR image data to slip casting of SiC_w/Si_3N_4 in conjunction with Garrett Ceramic Components Division of Allied-Signal Aerospace Company. Destructive analysis will be very important and Garret's technology for these destructive data will continue to be used. Work on NMR imaging methods will be continued as well, primarily with the addition of slice-selection methods.

Keywords: Binder, Nondestructive Evaluation, Nuclear Magnetic Resonance, Silicon Nitride

98. Additives for High-Temperature Liquid Lubricants — FY 1992 $100,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
JPL Contact: C. M. Moran, (818) 354-2982

Current additive technology is inadequate in that the additives generally available are designed to be effective at temperatures well below those encountered in the LHRE. They perform well at temperatures below 200°C. Volatility as well as both thermal and oxidative stability are some of the areas requiring improvement. Dinitrile compounds are in one chemical family that possesses the potential of meeting some of the desired properties. This project was initiated at JPL during the second quarter of FY 1991. The purpose of this research is to synthesize and characterize unique additives for liquid lubricants. The additives selected should lead to significant improvements in the major tribology task areas related to the reduction of friction and wear at high temperatures.

Keywords: Lubricants, Additives, Oils, Friction, Wear, Engines, High Temperature

99. Friction & Wear Mechanisms in *In Situ*-Reinforced Ceramics FY 1992
$125,000

DOE Contact: J. M. Perez, (202) 586-8060
ORNL Contact: Peter Blau, (615) 574-1514

Previous work has identified the friction and wear mechanisms of silicon nitride- and alumina-based whisker composites. This new effort extends this analysis to a new class of *in situ*-toughened ceramics developed by subcontractors in the Ceramics Technology for Advanced Heat Engines Program. Tests and analysis will be performed to compare their high-temperature friction and wear behavior with that of other ceramics and to identify the dominant friction and wear mechanisms that control that behavior. During the year, a CRADA will be instituted between ORNL and the developer of these materials.

Keywords: Lubrication, Ceramic Composites, Wear, Friction, Wear Transitions, Design Guidelines

100. Carbon-Based Materials for Engine Applications FY 1992
$125,000

DOE Contact: J. M. Perez, (202) 586-8060
ORNL Contact: Peter Blau, (615) 574-1514

Carbon-based materials offer a number of attractive advantages for friction and wear applications, having been used for many years in such applications as pumps, seals, and bearings. However, their introduction into advanced automotive and truck technology has been relatively slow. Part of the reason for this is a lack of focused R&D into the elevated-temperature tribological properties of carbon-based materials. We propose developing baseline data to explore the tribological potential of these materials under temperatures and lubricated environments typical of advanced transportation applications.

Keywords: Ceramics, Carbon, Graphite, Engines, Friction, Wear

101. Advanced Materials/Additive Interactions FY 1992
$150,000

DOE Contact: J. M. Perez, (202) 586-8060
NIST Contact: Stephen Hsu, (301) 921-2113

Ceramic materials are being used increasingly in various applications requiring superior tribological properties related to wear resistance under severe environmental conditions. Data obtained at NIST and those reported in the literature indicate that the wear rate and the friction coefficient of ceramics are generally too high to achieve material conservation or energy conservation. Thus, to successfully utilize ceramics, lubricants must be developed to control friction and wear in these systems. Interactions between various synthetic base oils, additive and ceramics will be studied at ambient and elevated temperatures using various testing and analytical resources. Specifically, the role of the ceramic surfaces on the tribochemical reactions leading to the formation of lubricating films, the effect of the substrate on the thermo-oxidative chemistry of the lubricant, and the effect of lubricant chemical structure will be determined and the competitive or additive interactions established. Analytical techniques considered will include SEM, EDX, FTIR, GPC, TEM and NMR.

Keywords: Ceramics, Ceramic Coatings, Lubrication, Friction, Wear

102. <u>Liquid Lubricants for Heat Engines</u> — <u>FY 1992</u> $150,000

DOE Contact: J. M. Perez, (202) 586-8060
NIST Contact: Stephen Hsu, (301) 921-2113

Successful implementation of advanced heat engines, such as the low heat rejection engine (LHRE), is hindered by the lack of stable lubricants and additives for high temperature applications. Current engine technology is the result of years of materials and lubricants research and development. Extensive experience on metals, alloys, lubricants and additives has evolved from this process. However, extension of this experience to new technological areas has been slow and at times Edisonian. A major factor is that most conventional lubricant applications involve regimes of 200°C or lower and the extension of the knowledge gained to the temperature requirements for future advanced systems is lacking. The LHRE may have a top-ring-reversal temperature as high as 425°C. The need is for lubricants with the capability to effectively survive the severe thermo-oxidative environment, minimize deposit formation and still control friction and wear. A critical component to success in this area is the development of information and an understanding of high temperature science involving new chemical additives and lubricants.

Keywords: High Temperature Liquid Lubricants, Tribology, Friction, Wear

103. <u>Advanced Lubricants for Engine Particulate Control</u> — <u>FY 1992</u> $150,000

DOE Contact: J. M. Perez, (202) 586-8060
NIST Contact: Stephen Hsu, (301) 921-2113

The U.S. Environmental Protection Agency (USEPA), starting in 1968 as part of its overall strategy to improve air quality, has reduced the contribution of diesel-engine emissions to the atmosphere through the implementation of increasingly more stringent regulations with time. The scenario has been well- documented in numerous publications. The regulations invoked for 1991 and 1994 reflect reductions in both the gaseous and particulate emissions, with the latter more difficult to achieve. In engines of the 1970's, the fuel was a major contributor to the particulates under many operating conditions. As the efficiency of the engines has improved through the use of electronic fuel injection and design changes, the contribution of the fuels to the particulate chemistry has diminished. Therefore, the lubricant and its additive components have become the major contributor to the organic fraction of particulates. Reduction or elimination of this fraction will significantly improve engine emissions. It is the objective of this project to explore concepts that would enable a two-thirds reduction in the organic fraction of diesel particulates.

Keywords: High Temperature Liquid Lubricants, Tribology, Emissions

104. <u>Lubricious Oxide Coatings</u> — <u>FY 1992</u> $150,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Wear Sciences Corporation (WSC) Contact: M. Peterson, (301) 261-2342

This project will attempt to define the principles of wear protection of sliding surfaces through the formation, in situ, of durable, hard but lubricious oxide films. Binary alloys will be prepared with solute elements which could form protective oxides based on several different approaches: (1) formation of a hard, compacted, wear-debris layer; (2) internal oxidation; (3) stress-free films; (4) increased oxidation; and (5) spall inhibition. Binary alloys of nickel, iron, copper, and titanium with selected additions of hard oxide formers (e.g. Si, Be, Ti, Ta, Zr, Nb, Al, In, Ge, V, and Cr) will be fabricated and evaluated in terms of their tribological performance and microstructural behavior. This research will involve a collaborative effort between WSC, ANL, Colorado State University (CSU), and the Institute of Metal Research (Shenyang, China).

Keywords: Surface Modification, Coatings, Solid Lubricants, Friction, Lubricious Coatings

105. <u>Alternative Fuels-Compatible Materials</u> — <u>FY 1992</u> $0

DOE Contact: J. M. Perez, (202) 586-8060
ORNL Contact: Ralph McGill, (615) 574-4077

A new Alternative Fuels-Compatible Materials Program is being initiated. The objective of this program is to develop an industrial technology base in advanced materials for engines and vehicles to enable the optimization of the use of alternative fuels and to ensure that the infrastructure necessary to utilize these vehicles might be realized. Improved materials of all types, when available, should be exploited so that their inherently superior properties would make the greatest contribution to improved energy efficiency, reduced environmental degradation, and greater utilization of alternative fuels. A tentative program addressing the need for materials that are compatible with alternative fuels has been defined through several workshops conducted with industry by DOE and ORNL. Some of the critical needs identified at these workshops are for lean-burn catalysts, fuel system durability, reduction of emissions, lubrication of engine components, and materials compatibility. Various alternative fuels were considered at the workshops, but the focus of the program would be primarily on alcohol and natural gas fuels.

Keywords: Alternative Fuel Vehicles, Fuels/Materials Compatibility, Fuels/Lubricant Compatibility

Technology Transfer and Management Coordination

106. Management and Coordination (WBS No. 111) FY 1992 $950,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832

The objective of this effort is to assess the ceramic technology needs for advanced automotive heat engines, formulate technical plans to meet these needs, and prioritize and implement a long-range research and development program.

Keywords: Advanced Heat Engines, Structural Ceramics, Management, Coordination, AGT, Diesel

107. International Exchange Agreement (IEA) (WBS No. 4115) FY 1992 $400,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: Victor J. Tennery, (615) 574-5123

The purpose of this effort is to assist and encourage international cooperation in the development of voluntary standard methods for determining mechanical, physical, and structural properties of advanced ceramic materials. There are four tasks in the Annex II agreement, "Cooperative Program on Ceramics for Advanced Heat Engines and Other Conservation Applications," (1) technical information exchange, (2) ceramic powder characterization, (3) ceramic chemistry, and (4) ceramic material property measurements. Research for subtasks 2, 3, and 4 has been completed. Participants in Annex II include the United States, the Federal Republic of Germany, Sweden and, most recently, Japan.

Two new subtasks, Subtask 5 - Tensile and Flexural Properties of Ceramics and Subtask 6 - Advanced Ceramic Powder Characterization, have been added.

Keywords: IEA, Powder Characterization, Mechanical Properties

108. Standard Reference Materials (WBS No. 4116) FY 1992 $150,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
National Institute of Standards and Technology (NIST) Contact: S. Malghan, (301) 975-5772

This project is directed toward a critical assessment and modeling of ceramic powder characterization methodology and toward the establishment of an international basis for standard materials and methods for the evaluation of powders prior to processing. There are three areas of emphasis: (1) to divide, certify, and distribute five ceramic powders for an international round-robin on powder characterization; (2) to provide reliable data on physical (dimensional), chemical, and phase characteristics of two silicon nitride powders (a reference powder and a test powder); and (3) to conduct a statistical assessment and modeling of round-robin data. The round-robin is to be conducted through the auspices of the International Energy Agency.

Keywords: IEA, Reference Material, Powder Characterization

109. Mechanical Property Standardization (WBS No. 4121) FY 1992 $100,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
National Institute of Standards and Technology (NIST) Contact: G. Quinn, (301) 975-5765

The purpose of this effort is to develop test methods in support of the Advanced Materials Development and the Advanced Turbine Technology Applications Programs.

Keywords: Mechanical Properties, Test Procedures

110. Diamond Coatings for Tribology FY 1992 $70,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: Fred Nichols, (708) 252-8292

Much progress has been made in depositing hard diamond and diamond-like-carbon (DLC) films. Existing diamond-coating processes, while capable of producing true diamond films with hardness comparable to that of natural diamonds, result in films which are quite rough and too abrasive to be utilized in sliding- and rolling- contact applications. It is apparent that the production of diamond films suitable for tribological applications will require a thorough assessment of the deposition technology. ANL will organize and conduct a workshop which will bring together the leading researchers and potential users of thin diamond films, especially in applications relevant to transportation technologies. It is expected that, following the workshop, the program will be initiated by means of an RFP.

Keywords: Surface Modification, Coatings, Friction, Wear, Ion Assisted Deposition, Diamond Films, Hard Coatings

Device or Component Fabrication, Behavior or Testing

111. Advanced Coating Technology (WBS No. 1311) — FY 1992 $175,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: D. P. Stinton, (615) 574-4556

The objective of this project is to develop an adherent coating that will prevent sodium corrosion of silicon nitride, silicon carbide, or other ceramics used as components in gas turbine engines. The specific coating composition must be compatible with these materials during both coating application and in the operating environment of a gas turbine engine. The effects of the combustion environment on the microstructure, wear, strength, and adherence of these coatings must be minimal.

Keywords: Coatings, Chemical Vapor Deposition, Engines, Silicon Carbide, Silicon Nitride, Structural Ceramics, Corrosion

112. Coatings to Reduce Contact Stress Damage of Ceramics (WBS No. 1313) — FY 1992 $66,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. R. Johnson, (615) 576-6832
Boston University Contact: V. K. Sarin, (617) 353-2842

The objective of this effort is to develop oxidation/corrosion-resistant, high toughness, adherent coating configurations for silicon-based ceramic substrates for use in advanced gas turbine engines. It is anticipated that a compositionally tailored and structurally engineered multilayered coating configuration with each layer designed to fulfill certain requirements will be developed. The conceptual coating configuration will be composed of an interfacial layer, transition layer, and a protective outer layer.

Keywords: Adherence, Coatings, Contact Stress, Oxidation, Modeling, Corrosion Resistance, Structural Ceramics

113. Wear Resistant Coatings (WBS No. 1331) FY 1992 $224,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. P. Stinton, (615) 574-4556
Caterpillar Contact: M. H. Hazelkorn, (309) 578-6624

The following wear-resistant coatings were selected in Phase I: plasma-sprayed high carbon iron-molybdenum, plasma-sprayed chromia-silica, and low temperature arc vapor deposited (LTAVD) chrome nitride. A plasma-sprayed carbon iron-molybdenum and a plasma-sprayed chromia-silica were identified as wear-resistant piston-ring coatings. The three main technical tasks for Phase II are: (1) further optimization of the LTAVD chrome nitride and cast iron porcelain enamel wear coatings; (2) process scale-up of wear-resistant plasma coatings for cylinder-liner applications; and (3) simulated engine testing.

Keywords: Coatings, Engines, Friction, Structural Ceramics, Wear

114. Wear Resistant Coatings (WBS No. 1332) FY 1992 $0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. P. Stinton, (615) 574-4556
Cummins Contact: Malcolm Naylor, (812) 377-7713

The goal of this program is to develop wear-resistant coatings for application to metallic components of low-heat-loss diesel engines; specifically, piston rings and cylinder liners. Coated ring samples were tested against a conventional pearlitic grey cast iron liner material using a high temperature reciprocating wear-test rig. For lowest wear under boundary-lubricated conditions, high velocity oxy-fuel (HVOF) Cr_3C_2-20% NiCr and WC-12% Co cermets, low temperature arc vapor deposited (LTAVD) CrN, and plasma-sprayed chromium oxides were the most promising candidates. Plasma-sprayed Cr_2O_3 and Al_2O_3-ZrO materials exhibited excellent wear resistance in unlubricated tests and at extremely high temperatures with synthetic oil. Although most of the coatings evaluated showed higher rates of wear with high-soot, engine-tested oil, metallic materials were found to be much more sensitive to soot/oil degradation. A Phase II is planned.

Keywords: Adherence, Coatings, Engines, Friction, Metals, Structural Ceramics, Thermal Conductivity, Wear

115. Thick Thermal Barrier Coating Systems for Low Heat Rejection Diesel Engines (WBS No. 1342)

FY 1992
$151,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. P. Stinton, (615) 574-4556
Caterpillar Contact: M. B. Beardsley, (309) 578-8514

The objective of this effort is to advance the fundamental understanding of thick thermal barrier coating (TTBC) systems for application to low-heat-rejection diesel engine combustion chambers. Areas of TTBC technology that will be examined include: powder characteristics and chemistry; bond coat composition; coating design, microstructure, and thickness as they affect properties, durability, and reliability; and TTBC "aging" effects (microstructural and property changes) under diesel engine operating conditions.

Keywords: Coatings, Structural Ceramics

116. Active Metal Brazing PSZ-Iron (WBS No. 1411)

FY 1992
$220,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: D. Ray Johnson, (615) 576-6832
ORNL Contact: M. L. Santella, (615) 574-4805

The objective of this task is to develop strong, reliable joints containing ceramic components for applications in advanced heat engines. The overall emphasis is on studying the brazing characteristics of silicon nitride and silicon carbide. The techniques of direct brazing as well as vapor coating ceramics to circumvent wetting problems will be applied to these materials. Currently, the specific areas of emphasis are to: (1) continue the study of high-temperature brazing of silicon nitride, and the effects of thermal aging on joint strength; and (2) initiate a study of the brazing characteristics of sintered α-SiC.

Keywords: Metals, Structural Ceramics, Joining/Welding, Brazing, Silicon Carbide, Silicon Nitride

117. Metal-Ceramic Joints AGT (WBS No. 1412)

FY 1992
$0

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: M. L. Santella, (615) 574-4805
GTE Contact: S. Kang, (617) 890-8460

The goal of this work was to demonstrate analytical tools for use in designing ceramic-to-metal joints including the strain response of joints as a function of the mechanical and physical properties of the ceramic and metal, the materials used in producing the joint, the geometry of the joint, externally imposed stresses both mechanical and thermal in nature, temperature, and the effects on joints exposed for long times at high temperature in an oxidizing (heat engine) atmosphere. A general methodology was developed to optimize the joint geometry and material systems for 650°C applications. Failure criteria were derived to predict the fracture of the braze and ceramic. Extensive finite element analyses (FEA) were performed to examine various joint geometries and to evaluate the effect of different interlayers on the residual stress state. The results showed that the measured strength of the joint reached 30-80% of the strength predicted using FEA. Potential braze alloys were developed and evaluated for the high-temperature application of ceramic-metal joints.

Keywords: Engines, Joining/Welding, Metals, Structural Ceramics, AGT, Metal-Ceramic, Physical/Mechanical Properties, Predictive Behavior Modeling

118. Ceramic-Ceramic Joints AGT (WBS No. 1421) — FY 1992 $254,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: M. L. Santella, (615) 574-4805
Norton Contact: G. J. Sundberg, (508) 351-7908

The purpose of this program is to develop techniques for producing reliable ceramic-ceramic joints and analytical modeling to predict the performance of the joints under a variety of environmental and mechanical loading conditions. The purpose of the Phase II effort is to develop joining technologies for HIPed Si_3N_4 with 4 wt % Y_2O_3 and for a siliconized SiC (NT-230) for various geometries including butt joins, curved joins, and shaft-to-disk joins. In addition, more extensive mechanical characterization (MOR at 22°C and 1370°C, stress rupture at 1370°C, high-temperature creep, tensile tests at 22°C, and spin tests) of silicon nitride joins will be performed to enhance the predictive capabilities of the analytical/numerical models for structural components in advanced heat engines.

Keywords: Engines, Joining/Welding, Metals, Silicon Carbide, Silicon Nitride

119. Next-Generation Grinding Wheel (WBS No. 1531) — FY 1992 $70,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: P. J. Blau, (615) 574-5377
Industry Contact: TBD

This new RFP is aimed at the engineering design and development of a next-generation, superabrasive grinding wheel specifically tailored for the cylindrical grinding of silicon nitride and other advanced structural ceramic parts for automotive and truck engine applications. The intent of this effort is to significantly reduce manufacturing cost of ceramic parts and to enhance the competitiveness of U.S. industry by providing an optimized grinding wheel for ceramics.

Keywords: Cost Effective Ceramics, Machining, Silicon Nitride, Structural Ceramics, Surface Characterization and Treatment

120. High Speed Grinding (WBS No. 1532) FY 1992 $230,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: P. J. Blau, (615) 574-5377
Eaton Contact: TBD

The purpose of this new effort is to develop a single step, rough finishing process suitable for producing high-quality silicon nitride ceramic parts at high material removal rates and at substantially lower cost than traditional, multi-stage grinding processes.

Keywords: Cost Effective Ceramics, Machining, Silicon Nitride, Structural Ceramics, Surface Characterization and Treatment

121. Chemically Assisted Grinding of Ceramics (WBS No. 1533) FY 1992 $150,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: P. J. Blau, (615) 574-5377
NIST Contact: Steven M. Hsu, (301) 975-6119

The purpose of this new effort is to increase the rate of machining and simultaneously reduce the surface roughness of ceramics through controlled surface modification (i.e., by adjusting the cutting fluid chemistry) of the surface grinding and finishing process.

Keywords: Cost Effective Ceramics, Machining, Silicon Nitride, Structural Ceramics, Surface Characterization and Treatment

122. Grindability Test (WBS No. 1542) FY 1992 $70,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: P. J. Blau, (615) 574-5377
Industry Contact: TBD

The objective of this new RFP is to develop and validate an inexpensive, bench-top method to assess the relative grindability of ceramics in a manner that can help manufacturers define, in advance, the operations necessary to grind and finish newly developed ceramics for automotive and truck engine applications.

Keywords: Cost Effective Ceramics, Machining, Silicon Nitride, Structural Ceramics, Surface Characterization and Treatment

123. Grinding Consortium (WBS No. 1543) FY 1992 $150,000

DOE Contact: Robert B. Schulz, (202) 586-8051
ORNL Contact: P. J. Blau, (615) 574-5377
NIST Contact: Said Jahanmir, (301) 975-6871

The purpose of this new effort is to develop guidelines and recommendations for grinding optimization of advanced structural ceramics to achieve minimum cost and maximum reliability. This effort will involve development of a database to provide information on the relationship between machining parameters and ceramic specimen surface finish and modulus of rupture (MOR). It will also involve assessing the usefulness of using thermal-wave methods to detect machining damage in ceramics.

Keywords: Database, Cost Effective Ceramics, Machining, Silicon Nitride, Structural Ceramics, Surface Characterization and Treatment

124. Fluid-Film Bearings Model FY 1992 $69,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Univ of Pittsburgh Contact: Professor A. Z. Szeri, (412) 624-9775

This subcontract consists of a three-year project to develop a simplified, yet accurate model of fluid-film lubrication under thermohydrodynamic conditions, with significant elastic and thermal distortion. The end product will include two user-friendly, comprehensive computer programs, one for journal bearings and one for thrust bearings, to be utilized interactively by designers on a personal computer such as the IBM PC/AT or IBM/PS2, or compatibles. The journal-bearing problem will include multiple-pad bearings, either fixed or pivoted, and the thrust-bearing code will cover sector-shaped pads. In addition to static performance parameters, the programs will also calculate linearized stiffness and damping coefficients in a form suitable for rotor-stability analyses. Continual liaison will be maintained with ACTIS, Inc., to insure compatibility for incorporation of these codes into the ACTIS family.

Keywords: Tribology, Bearings, Computer Models, Hydrodynamic Lubrication, Lubrication

125. Wear Mechanism Modeling | FY 1992
$115,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Cambridge University Contact: M. F. Ashby, 0-223-33-2-2622

The rate of wear when two surfaces slide, and the mechanisms responsible for it, depend on bearing pressure and sliding velocity, and on numerous bulk and surface properties of the mating materials. Frictional heating is an important factor in determining the mechanism of wear, and—unlike the wear rate itself—it is relatively easy to calculate. The unified approach to this problem previously developed in this activity and published as the user friendly, PC-based computer software T-MAPS will be further analyzed and applied to various sliding pairs utilized in the tribology program at ANL. As new insight and new questions are generated, these are fed back for incorporation into revision of the T-MAPS software at Cambridge. Currently, the lifetime for contact of one asperity with sliding-partner surface is being further refined due to feedback from ANL and other investigators. A final report will be prepared.

Keywords: Metals, Ceramics, Friction, Wear, Engines

126. Instantaneous Friction Torque | FY 1992
$25,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Wayne State University Contact: Naeim Henein, (313) 577-3887

A three-year project involving a new technique to evaluate instantaneous friction torque (IFT) in an operating, reciprocating internal-combustion engine, is in its third year at Wayne State University (WSU). This Pressure-Angular Velocity (P-ω) technique is based on in-situ measurements of combustion pressure (P) and shaft angular velocity (ω) during both steady-state and transient operation of the engine. It is the only currently known method for measuring IFT under actual operating conditions. The final phase of the work will consist of in-situ operating-engine tests of solid-lubricant-coated components. Experiments will be conducted in a single-cylinder gasoline engine where cycle-to-cycle variations are often significant. The goal is to determine the effect on engine friction and wear of low-friction, high-temperature, self-lubricating coatings prepared by Ion-Beam-Assisted Deposition (IBAD).

Keywords: Friction, Torque, Engines, Combustion, Modeling

127. Scale Effects in Friction Simulations FY 1992
$62,000

DOE Contact: J. M. Perez, (202) 586-8060
ORNL Contact: Peter Blau, (615) 574-1514

Friction simulations for accurate screening of advanced materials requires attention to such details as contact-stress history, thermal history, contact arrangement, chemical environment, surface preparation, sliding velocity, and more. Previous work demonstrated how important testing-machine characteristics can be and how wear particles can sometimes accommodate interfacial shears so as to dominate frictional behavior. The project will study effects of contact aspect ratio and pressure distribution, to determine sensitivity of frictional behavior (including running-in) to macro-contact geometry, and strategies for selection of testing procedures (or equipment design) for effective simulations. In addition, more attention will be paid to vibrational characteristics of friction-testing machines and how these need to be characterized to better simulate the behavior of actual components.

Keywords: Friction, Friction Models, Scale Effects, Friction Mechanisms, Ceramics, Composites

128. Thermomechanical Wear Model for Ceramics FY 1992
$0

DOE Contact: J. M. Perez, (202) 586-8060
ORNL Contact: P. J. Blau, (615) 574-1514
GA Inst Tech Contact: Ward Winer, (404) 894-3270

FY 1992 marks the final year of this subcontract which has resulted in the development of a thermomechanical model for wear and surface damage of ceramics. The initial model is being extended to permit lubrication effects to be incorporated. The final product of this work will be a series of thermomechanical wear maps for engineering ceramics (alumina, silicon nitride, zirconia, and silicon carbide) which identify combinations of contact pressure, velocity (thermal parameter), and time for satisfactory wear performance.

Keywords: Ceramics, Wear, Friction, Lubrication

129. Cylinder Kit Model Development FY 1992
$75,000

DOE Contact: J. M. Perez, (202) 586-8060
ANL Contact: F. A. Nichols, (708) 252-8292
Compu-Tec Engineering Contact: L. J. Brombolich, (314) 532-4062

The research will be divided into three areas of effort. Phase I will consist of the analytical development of the RING and PISTON programs. Phase II will include a literature search to obtain any data that may be pertinent to the verification of the RING program. In addition, any algorithms that may be used in RING will be investigated. A database of cylinder-kit parameters will be assembled to provide guidelines for future analyses. Data previously developed as well as data generated during this subcontract will be obtained from piston and piston-ring manufacturers, oil companies, liner manufacturers and engine manufacturers. Phase III will consist of engine testing and verification of the RING computer program. Ford Motor Co. is supporting Compu-Tec in this phase of the work. It is anticipated that the three phases will be conducted essentially concurrently.

Keywords: Engines, Diesels, Cylinder Kit, Friction, Piston Rings, Lubrication

Office of Propulsion Systems

The Office of Propulsion Systems is comprised of the Advanced Propulsion Division and the Electric and Hybrid Propulsion Division. Programs supported by this office are focused on developing, with industry through cost-shared contracts, the technologies that will lead to the production and introduction of advanced heat engine propulsion systems, and electric and hybrid vehicles in the nation's transportation fleet. Materials activities of the Office of Propulsion Systems focus on integration of materials into components and testing of subsystems for advanced vehicle propulsion systems.

Advanced Propulsion Division

The Advanced Propulsion Division has two major programs: Light Duty Engine Development focused on gas turbines through the Advanced Turbine Technology Applications Project (ATTAP) and Heavy Duty Engine Development focused on diesel engines through the Heavy Duty Transport Technology (HDTT) project. Materials activities supported by this Office and managed through the NASA Lewis Research Center for component and coating applications are included in this report. The DOE contacts are: Thomas Sebestyen, (202) 586-9727 for ATTAP and John Fairbanks, (202) 586-8012 for the HDTT project.

Materials Properties, Behavior, Characterization or Testing

130. Ceramic Durability Evaluation AGT — FY 1992 $195,000

DOE Contact: Thomas Sebestyen, (202) 586-9727
NASA Contact: Sunil Dutta, (216) 433-3282
Garrett Turbine Engine Contact: Nancy Campbell, (602) 220-7006

The objective of the program is to evaluate commercially available structural and glass ceramic material specimens exposed to combustion products at temperatures up to 2500°F for periods up to 3,500 hours. In 1989, Kyocera SN-251 silicon nitride and Carborundum TiB_2 and silicon carbide were tested.

Keywords: Silicon Carbide, Silicon Nitride, Erosion, Corrosion, High Temperature, Long Life, Automobile Engine Environment, Gas Turbines

Device or Component Fabrication, Behavior or Testing

131. Thick Thermal Barrier Coatings — FY 1992, $25,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: M. Murray Bailey, (216) 433-3416
Cummins Contact: Thomas M. Yonushonis, (812) 377-7078

Design and demonstration of the durability of thick thermal barrier coatings with low thermal conductance for use in low heat rejection diesel engines is the objective of the project. Zirconia-based coating systems will be developed and applied to metal engine parts for evaluation in a single cylinder engine rig. Completed 100 hours of engine testing and final report in process. No improvement in performance because duration of combustion was extended.

Keywords: Coatings, Oxide Ceramics, Diesel Engines

132. Thick Thermal Barrier Coatings — FY 1992, $25,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: M. Murray Bailey, (816) 433-3416
Caterpillar Contact: H. J. Larson, (309) 578-6549

Zirconia thermal barrier coating (TBC) systems are being developed and applied to diesel engine parts for evaluation in a single cylinder engine rig. Initial evaluation of engine tests indicated deterioration by combustion possibly attributed to TBC coating porosity and surface roughness. Impermeable sealing outerlayers that are aerodynamically smooth, coupled with combustion parameters modified for hot wall effects, produced a four percent fuel economy gain. Final report has been produced.

Keywords: Coatings, Oxide Ceramics, Diesel Engines

133. Advanced Diesel Engine Component Development Project — FY 1992 $260,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: Richard Barrows, (216) 433-3388
Detroit Diesel Corporation Contact: Theodor Freiheit, (313) 592-7224

The objective of the project is to develop advanced technology diesel engine components and integrate these into a test bed engine to demonstrate reduced emissions and improved fuel economy. Advanced ceramic and metallic materials are being investigated and used in structural, insulative, and tribological component applications.

Keywords: Structural Ceramics, Low Heat Rejection Diesel Engines, Thermal Barrier Coatings, Component Designs, Composite Materials

134. Advanced Piston and Cylinder Component Development — FY 1992 $1,200,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: J. J. Notardonato, (216) 433-3908
Caterpillar Inc. Contact: H.J. Larson, (309) 578-6549

The objective of the project is to develop advanced technology diesel engine components and integrate these into a test bed engine to demonstrate reduced emissions and improved fuel economy. Advanced ceramic and metallic materials are being investigated and used in structural, insulative, and tribological component applications.

Keywords: Structural Ceramics, Low Heat Rejection Diesel Engines, Thermal Barrier Coatings, Component Designs, Composite Materials

135. Advanced Piston and Cylinder Component Development — FY 1992 $1,000,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: J. J. Notardonato, (216) 433-3908
Cummins Engine Contact: T. Yonushonis, (812) 377-7078

The objective of the project is to develop advanced technology diesel engine components and integrate these into a test bed engine to demonstrate reduced emissions and improved fuel economy. Advanced ceramic and metallic materials are being investigated and used in structural, insulative and tribological component applications.

Keywords: Structural Ceramics, Low Heat Rejection Diesel Engines, Thermal Barrier Coatings, Component Designs, Composite Materials

136. High Temperature Solid Lubricant Coatings FY 1992
$50,000

DOE Contact: John W. Fairbanks, (202) 586-8066
NASA Contact: Hal Sliney, (216) 433-6055
Case Western Reserve University Contact: Joseph Prahl, (216) 368-2000

High temperature wear resistant sleeve bearing systems for use at operating temperatures of up to 500°C in diesel engines are being developed and evaluated. Powder metallurgy forms of wear resistant coatings containing solid lubricants for reduced friction are being developed. Current efforts involve development consistent with the process steps that are commercially competitive.

Keywords: Wear, Coatings, Diesel Engines, Tribology

137. Advanced Turbine Technology Applications Project (ATTAP, AGT-5) FY 1992
$6,300,000

DOE Contact: Thomas Sebestyen, (202) 586-9727
NASA Contact: Paul Kerwin, (216) 433-3409
General Motors, Allison Gas Turbine Division, Contact: Phil Haley, (317) 230-2272

Advanced structural ceramic materials are being developed for hot flow path components of an automotive gas turbine engine designed for operation at 2500°F. Efforts include material characterization, process development, and component design and test. In 1989, design of a 2500°F axial turbine stage was completed. Prototype ceramic components for dimensional and properties characterization have been fabricated.

Keywords: Structural Ceramics, Component Design, Silicon Carbide, Rig and Engine Testing, Silicon Nitride, Gas Turbine Engines

138. Advanced Turbine Technology Applications Project (ATTAP, AGT-101) FY 1992
$5,000,000

DOE Contact: Thomas Sebestyen, (202) 586-9727
NASA Contact: Thomas N. Strom, (216) 433-3408
Garrett Auxiliary Power Division Contact: Jay Smyth, (602) 220-3360

Advanced structural ceramic materials are being developed for hot flow path components for an automotive gas turbine engine designed for operation at 2500°F. The project combines an integrated design, fabrication, and test approach with component technology to be verified in an engine environment. Fabrication of prototype components is underway. A study to quantify rotor impact damage has been completed which has led to a new mixed flow rotor design. This strengthens the (new) rotor and eliminates particle traps.

Keywords: Structural Ceramics, Component Design, Fabrication, Component Test, Gas Turbine Engines

Electric and Hybrid Propulsion Division

The Electric and Hybrid Propulsion Division has three major programs: Battery Development, Fuel Cells Development, and Systems Development for electric vehicles. The DOE contact is Kenneth Heitner, (202) 586-2341, for Battery Development; Pandit Patil, (202) 586-8055, for Fuel Cells Development; and Albert Landgrebe, (202) 586-1483, for Exploratory Research in support of Batteries and Fuel Cells.

Materials Preparation, Synthesis, Deposition, Growth or Forming

139. Corrosion-Resistant Coatings for High-Temperature, High-Sulfur Activity Applications — FY 1992 $132,000

DOE Contact: A. Landgrebe, (202) 586-1483
Illinois Institute of Technology Contact: J. R. Selman, (312) 567-3037

This research project explores electrodeposition and chemical vapor deposition techniques used to prepare corrosion-resistant coatings for high temperature batteries. The deposition of molybdenum and Mo_2C by electrochemical deposition in molten salt was optimized in order to obtain reproducible thicknesses and smooth surface morphology. Researchers observed that complete removal of moisture from the electrolysis bath is necessary to obtain a reproducible, high quality coating. Coatings of an even better quality were obtained with a bath containing non-Li alkali molybdates and carbonates. Through utilizing these coatings, researchers hope to obtain long-term endurance in Na/S cells.

Keywords: Electrodeposition, Chemical Vapor Deposition, Corrosion, Coatings and Films

140. Improved Container Electrode Coating for Na/S Battery Systems — FY 1992 $105,000

DOE Contact: A. Landgrebe, (202) 586-1483
Environmental Research Institute of Michigan Contact: T. Hunt

The objective of this new project is to develop improved corrosion-resistant coatings for high-temperature secondary batteries by sputter-deposition techniques. Preliminary studies at Ford Motor company indicated that sputter-coated TiN on Al was resistant to attack in a polysulfide melt during a 72-hour test. Reactive sputtering will be used for the preparation of the majority of coatings that are being examined. However, experiments also will be conducted to understand the stress regimes on coatings obtained by radio frequency sputtering, because control of the deposition parameters for reactive sputtering appears to be more demanding. A reactive sputtering system is being assembled at ERIM to duplicate the quality of coatings obtained at Ford, and to evaluate the corrosion-resistant properties of TiN for extended periods in polysulfide melts.

Keywords: Sputter-Deposition, Corrosion, Coatings and Films

Materials Structure and Composition

141. New Battery Materials — FY 1992, $78,000

DOE Contact: A. Landgrebe, (202) 586-1483
Stanford University Contact: R. Huggins, (415) 723-4110

An effort is underway to characterize high-sodium-activity alloys and low-melting-point molten salts for sodium metal chloride cells. This research seeks to improve the removal of the high-impedance solid electrolyte. A number of alloy compositions were screened for their ability to act as fast, reversible sodium-conducting electrodes. The use of a family of alkali halide ammoniate salts for sodium metal chloride cells was also investigated.

Keywords: Batteries, Alloys

142. Spectroscopic Studies of Passive Films on Alkali and Alkaline Earth Metals in Nonaqueous Solvents — FY 1992, $55,000

DOE Contact: A. Landgrebe, (202) 586-1483
Case Western Reserve University Contact: D. G. Scherson, (216) 368-5186

Research efforts have focused on preparing and characterizing submonolayer deposits of alkali and alkaline earth metals on metal surfaces and developing the understanding of passive surface layers in rechargeable alkali batteries. The activation of carbon dioxide induced by the presence of alkali metal atoms adsorbed on an otherwise inert substrate was examined with an array of surface analytical methods. The analytical techniques in this study included work function measurements, low energy electron diffraction, Auger electron, X-ray photoelectron XPS, high resolution electron energy loss, and thermal desorption spectroscopies. Researchers used *in situ* spectroscopic techniques and thermal analysis to study the Li/organic electrolyte and Li/poly(ethyleneoxide) interfaces. Interactions between Li and tetrahydrofuran, and the formation of Li-O and Li-CO_3 species, were detected.

Keywords: Surface, Structure, Films, Batteries

143. *In situ* Spectroscopic Applications to the Study of Rechargeable Lithium Batteries — FY 1992, $86,000

DOE Contact: A. Landgrebe, (202) 586-1483
Case Western Reserve University Contact: D. G. Scherson, (216) 368-5186

Initiated in 1991, this project uses *in situ* spectroscopic techniques to investigate charge/discharge reactions of Li at Li/SPE interfaces. Techniques have been developed and implemented to investigate the Li electrode in PEO electrolytes under conditions of direct relevance to rechargeable Li/polymer battery technology. Preliminary cyclic voltammetry studies of Au in contact with $LiClO_4$- poly(ethylene oxide) electrolyte at 55°C showed evidence for underpotential deposition (UPD) of Li.

Keywords: Electrode, Electrolytes, Batteries

144. Raman Spectroscopy of Electrode Surface in Ambient-Temperature Lithium Secondary Battery — FY 1991, $47,000

DOE Contact: A. Landgrebe, (202) 586-1483
Jackson State University Contact: H. Tachikawa, (601) 968-2171

Researchers at Jackson State are characterizing the surface layers on Li electrodes in nonaqueous electrolytes by electrochemical and Raman spectroscopic methods to provide information which is useful for improving the performance and cycle life of Li secondary batteries. Researchers evaluated the electrochemical properties of C_{60} fullerene as an electrode material that may be useful in rechargeable Li cells. The cyclic voltammograms of C_{60} in $LiClO_4$/polyethylene glycol 400 dimethyl ether (PEG400DME) indicated five redox peaks which suggested the formation of C^{-}_{60}, C^{2-}_{60}, C^{3-}_{60}, C^{4-}_{60}, and C^{5-}_{60}. These anions dissolve in PEG400DME.

Keywords: Surface, Structure, Batteries

145. Battery Materials: Structure and Characterization FY 1992
$100,000

DOE Contact: A. Landgrebe, (202) 586-1483
Brookhaven National Laboratory Contact: J. McBreen, (516) 282-4513

This project attempts to elucidate the molecular aspects of materials and electrode processes in batteries and to use this information to develop electrode and electrolyte structures with improved performance and extended life. Work during the year included extended X-ray absorption fine structure (EXAFS) and x-ray absorption near-edge spectroscopy (XANES) studies of mossy Zn deposits that form in alkaline zincate electrolytes. The Zn that deposits at -65 mV (*vs* Zn wire reference electrode) is oriented with the c-axis parallel to the lines of current, whereas the deposit at -45 mV has a random orientation like that of a Zn foil.

Keywords: Electrodes, Morphology

146. Zinc/Air Battery Development for Electric Vehicles FY 1992
$50,000

DOE Contact: A. Landgrebe, (202) 586-1483
Metal Air Technology Systems International Contact: R. Putt, (404) 876-8203

During the past year it was demonstrated that zinc loadings greater than 100 mAh/cm^2 can be attained in a reticulated electrode structure. A process was developed for pre-plating copper substrate on a reticulated electrode structure. The process yielded a dense, uniform, and continuous zinc deposit.

Keywords: Batteries, Electrode

147. Polymeric Electrolytes for Ambient-Temperature Lithium Batteries FY 1992
$50,000

DOE Contact: A. Landgrebe, (202) 586-1483
University of Pennsylvania Contact: G. Farrington, (215) 898-6642

University of Pennsylvania researchers have investigated polymeric electrolytes formed by radiation-polymerization of various oligomers that contain different compositions of plasticizer (ethylene carbonate, EC and propylene carbonate, PC) and 1 M $LiAsF_6$. Polymeric electrolytes with greater than or equal to 50 wt% PC in mixtures with EC appear to exhibit acceptable electrochemical (reversible Li redox process) and physiochemical properties (ionic conductivity greater than 8×10^4 at room temperature, glass-transition temperature of -94°C, amorphous structure from -90 to 150°C) for use in rechargeable Li cells.

Keywords: Polymers, Batteries

Materials Properties, Behavior, Characterization or Testing

148. Corrosion, Passivity, and Breakdown of Alloys Used in High-Energy-Density Batteries — FY 1992 $30,000

DOE Contact: A. Landgrebe, (202) 586-1483
Johns Hopkins University Contact: J. Kruger, (301) 338-8937

The objective of this project is to investigate the phenomena of passivation and its breakdown on metals and alloys in nonaqueous solvents for rechargeable Li batteries. Researchers have observed that iron and 1018 carbon steel display an extensive and stable passive region in $LiAsF_6$/dimethoxyethane (DME). In a nominally dry $LiAsF_6$/DME solution (less than 100-ppm H_2O), the breakdown potentials of iron and carbon steel are 1300 mV (*vs* saturated calomel electrode, SCE) and 1050 mV, respectively. The adsorption of DME and the formation of carbon-based polymer film are believed to be responsible for passivation.

Keywords: Passivation, Metals: Ferrous, Batteries

149. Advanced Chemistry and Materials for Fuel Cells — FY 1992 $100,000

DOE Contact: A. Landgrebe, (202) 586-1483
Brookhaven National Laboratory Contact: J. McBreen, (516) 282-4513

The purpose of this project is to increase the understanding of electrocatalysis on a molecular level and to apply this knowledge to fuel cells. Researchers are utilizing x-ray absorption spectroscopy (XAS) to investigate the properties of Pt/C and several of its alloys with Cr, Co, and Ni. The results indicate that alloying with Ni has a large effect on the d character of Pt, whereas Cr has little effect. Nickel forms a solid solution with Pt, with the Ni atoms substituting at Pt sites.

Keywords: Fuel Cells, Catalysts, Oxygen Reduction

150. Electrocatalysts for Oxygen Electrodes — FY 1992 $150,000

DOE Contact: A. Landgrebe, (202) 586-1483
Case Western Reserve University Contact: E. Yeager, (216) 386-3626

The objective of this research is to develop more effective electrocatalysts for O_2 reduction and generation which have high activity and long-term stability. Various electrocatalysts, including the transition-metal macrocycles and oxide catalysts, were evaluated to identify stable catalysts with much higher activity for both monofunctional and bifunctional air electrodes. Researchers observed that the catalytic activity for the reduction of O_2 at cobalt tetrasulfonated phthalocyanine (CoTsPc) adsorbed on ordinary pyrolytic graphite (OPG) in alkaline solution is enhanced by approximately 60 mV in the presence of alcohols. Further, the presence of methanol has no short-term deleterious effect on the kinetics for O_2 reduction on CoTsPc/OPG, which also exhibits negligible catalytic activity for methanol oxidation.

Keywords: Catalysts, Polymers, Metals Surface, Composites, Batteries

151. <u>Novel Concepts for Oxygen Electrodes in Secondary Metal/Air Battery</u> — <u>FY 1992</u> $125,000

DOE Contact: A. Landgrebe, (202) 586-1483
Eltech Research Corporation Contact: E. Rudd

Researchers are investigating the viability of graphitized carbon blacks and metal oxides as electrocatalyst supports in bifunctional air electrodes for electrically rechargeable Zn/air cells. Graphitized carbon blacks of Monarch 120 and Shawinigan acetylene black, and the metal oxides of $NiCo_2O_4$, Co_3O_4, $Pb_2Ru_2O_7$ and $Pb_2Ir_2O_7$, have been prepared. Electrochemical tests will be underway shortly in small cells at Metal Air Technology Systems International (MATSI).

Keywords: Electrodes, Electrocatalysts, Batteries

<u>Device or Component Fabrication, Behavior or Testing</u>

152. <u>Proton-Exchange-Membrane Fuel Cells for Vehicles</u> — <u>FY 1992</u> $1,400,000

DOE Contact: A. Landgrebe, (202) 586-1483
Los Alamos National Laboratory Contact: S. Gottesfeld, (505) 667-0853

Los Alamos National Laboratory conducts three major proton-exchange-membrane (PEM) fuel cell projects. These projects seek to: (1) develop better gas diffusion electrodes; (2) measure and model the mass transport properties and conductivity of proton-conducting membranes; and (3) measure the operating characteristics of single PEM fuel cells and determine the conditions providing optimal performance. Researchers have found that processing membrane-electrode assemblies (MEA) with membranes that contain the Na^+ form rather than the H^+ form permits the use of higher processing temperatures (185 *vs* 135°C). Researchers have also characterized some of the H_2O-management properties of Nafion 117, membrane C, and an experimental Dow membrane. In addition, researchers have resolved the problem of membrane puncture that was observed in small fuel cells that contain thin membranes such as the 2-mil thick Nafion membrane by using a thin Teflon gasket in the membrane/electrode/gasket assembly.

Keywords: Fuel Cells, Separators, Electrodes

153. Solid Polymer Electrolytes for Rechargeable Batteries — FY 1992 $97,000

DOE Contact: A. Landgrebe, (202) 586-1483
SRI International Contact: D. MacDonald, (415) 859-3195

The objective of this project is to develop advanced ion-conducting polymers that can be used as SPEs in high-energy, rechargeable solid-state batteries. Researchers have developed a Li-ion conducting PEO-type polymer in which oxygen is replaced by sulfur. The best-performing polymer electrolyte, obtained from sulfur-substituted PEO (16.7% S) and tetraethylorthosilicate with a plasticizer, exhibited a conductivity of 7.5×10^{-4} ohm^{-1} cm^{-1} in a Li/Li cell.

Keywords: Polymers, Electrolytes, Batteries

154. Electrochemical Energy Storage — FY 1992 $1,800,000

DOE Contact: A. Landgrebe, (202) 586-1483
Lawrence Berkeley Laboratory Contact: F. McLarnon

The major thrust of this program is to evaluate promising electrochemical couples for advanced batteries for electric vehicles. Exploratory research was carried out on Zn/NiOOH and Na/metal oxide polymerization cells. Novel components for various versions of rechargeable Li, Na, and Zn cells were also investigated. Research topics include: Zn/KOH/NiOOH cells studies; electrochemical properties of solid-state sodium/polymer cells; surface morphology of metals in electrodeposition; high-temperature cell research; analysis and simulation of electrochemical systems; surface layers on batteries; application of photothermal deflection spectroscopy to electrochemical interfaces; electrode kinetics and electrocatalysis of methanol electrooxidation; effect of electrocatalyst and electrolyte composition on methanol/air fuel cell performance; transport in zinc-air cells and mathematical modeling; and development of electrically rechargeable zinc/air cells.

Keywords: Electrocatalyst, Batteries, Fuel Cells, Polymers

Office of Alternative Fuels

The Office of Alternative Fuels has three major programs: Biofuels Production, Alternative Fuels Utilization, and the Alternative Motor Fuels Act (AMFA) fleet test program. Materials technologies for alternative fuels are being addressed by the Office of Transportation Materials and other DOE offices. The DOE contact for biomass is Richard Moorer, (202) 586-5350, and the DOE contacts for alternative fuels are John Russell, Richard Wares, or Steve Goguen, (202) 586-8053.

Materials Properties, Behavior, Characterization or Testing

155. Cold-start Assist Materials — FY 1992 $120,000

DOE Contract: S. Goguen, (202) 586-8053
Oak Ridge National Laboratory Contact: R. Graves, (615) 574-2036

This project examines a wide variety of materials to determine their exothermic properties during phase change. The heat released would be utilized to vaporize alcohol automotive fuels to enhance their cold-starting characteristics.

Keywords: Phase Change Materials, Alcohol Fuels

156. CNG Adsorbents Demonstration — FY 1992 $300,000

DOE Contact: M. Gurevich, (202) 586-8053
Brookhaven National Laboratory Contact: James Wegrzyn, (516) 282-7917

This project screens and evaluates materials for their effectiveness in adsorbing natural gas. An adsorbent placed in a storage vessel could hold sufficient CNG to reduce working pressure to 500 psi. This would permit introduction of complex geometry (shaped) automotive tanks configured to take advantage of interior vehicle "dead space" as well as reducing overall weight requirements. Net result would be increased range.

Keywords: Natural Gas Adsorbent Materials, Alternative Fuels

OFFICE OF UTILITY TECHNOLOGIES

	FY 1992
Office of Utility Technologies - Grand Total	$40,426,000
Office of Solar Energy Conversion	$23,400,000
Photovoltaic Energy Technology Division	$23,400,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$18,000,000
Amorphous Silicon for Solar Cells	4,900,000
Polycrystalline Thin Film Materials for Solar Cells	9,100,000
Deposition of III-V Semiconductors for High-Efficiency Solar Cells	4,000,000
Materials Properties, Behavior, Characterization or Testing	$ 3,000,000
Materials and Device Characterization	3,000,000
Device or Component Fabrication, Behavior or Testing	$ 2,400,000
High-Efficiency Crystal Silicon Solar Cells	2,400,000
Office of Renewable Energy Conversion	$ 627,000
Geothermal Division	$ 627,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 97,000
Thermally Conductive Composites for Heat Exchangers	97,000
Materials Properties, Behavior, Characterization or Testing	$ 530,000
Advanced High Temperature Geothermal Well Cements	120,000
Advanced High Temperature Chemical Systems for Lost Circulation Control	160,000
Corrosion Mitigation in Highly Acidic Steam Condensates	100,000
High Temperature Chemical Bonding Systems	150,000

OFFICE OF UTILITY TECHNOLOGIES

	FY 1992
Office of Energy Management	$16,399,000
Advanced Utility Concepts Division	$16,399,000
Materials Properties, Behavior, Characterization or Testing	$ 330,000
High Temperature Composite Phase Change Material	200,000
Geochemistry Dynamics Associated with Groundwater Heating	130,000
Device or Component Fabrication, Behavior or Testing	$ 180,000
Phase Change Thermal Storage for Domestic Water Heating	90,000
Phase Change Thermal Storage in Building Materials	90,000
Superconductivity Systems Program (National Laboratory Projects)	$15,889,000
Device or Component Fabrication, Behavior or Testing	$15,889,000
High-Temperature Superconductor Development for Electric Power Applications	4,385,000
Characterization and Development of High-Temperature Superconductors	600,000
Electrodeposition of High-Temperature Superconductors	700,000
Bulk and Thin-Film Materials Process Research for Practical High-Temperature Superconductor Development	1,000,000
High-Temperature Superconductor Wire and Component Development for Electric Power Applications	4,200,000
Conductor Development Processing of Wires and Tapes, Magnet Design and Testing, Applications Development for High-Temperature Superconductors	5,004,000

OFFICE OF UTILITY TECHNOLOGIES

Office of Solar Energy Conversion

Photovoltaic Energy Technology Division

The National Photovoltaics Program sponsors high-risk, potentially high-payoff research and development in photovoltaic energy technology that will result in a technology base from which private enterprise can choose options for further development and competitive application in U.S. electrical markets. The objective of materials research is to overcome the technical barriers currently limiting the efficiency and cost of photovoltaic cells. Theoretical conversion efficiency of photovoltaic cells is limited by the portion of the solar spectrum to which the cell's semiconductor material can respond, and by the extent to which these materials can convert each photon to electricity. The practical efficiency is constrained by the amount of light captured by the cell, the cell's uniformity, and a variety of loss mechanisms for the photo-generated carriers. Cost is affected by the expense and amount of materials required, the complexity of processes for fabricating the appropriate materials, and the complexity and efficiency of converting these materials into cells.

Materials Preparation, Synthesis, Deposition, Growth or Forming

157. Amorphous Silicon for Solar Cells — FY 1992 $4,900,000

DOE Contact: Richard King, (202) 586-1693
NREL Contact: Werner Luft, (303) 231-1452

This project performs applied research upon the deposition of amorphous silicon alloys to improve solar cell properties. Efficient solar energy conversion is hindered by improper impurities or undesired structure in the deposited films and the uniformity of the films over large (1000 cm^2) areas. The films are deposited by plasma enhanced chemical vapor deposition (glow discharge), thermal chemical vapor deposition and sputtering. The long term goal of this effort is to develop the technology for 12 percent efficient solar cells with an area of about 1000 cm^2. Achieving that goal should enable amorphous silicon to be a cost-effective electrical generator.

Keywords: Amorphous Materials, Coatings and Films, Semiconductors, Chemical Vapor Deposition, Sputtering and Solar Cells

158. Polycrystalline Thin Film Materials for Solar Cells FY 1992
$9,100,000

DOE Contact: Richard King, (202) 586-1693
NREL Contact: Kenneth Zweibel, (303) 231-7141

This project performs applied research upon the deposition of $CuInSe_2$ and CdTe thin films for solar cells. Research centers upon improving solar cell conversion efficiency by depositing more nearly stoichiometric films, by controlling interlayer diffusion and lattice matching in heterojunction structures and by controlling the uniformity of deposition over large (1000 cm^2) areas. The films are deposited by chemical and physical vapor deposition, electrodeposition and sputtering. The long term goal for this effort is to develop the technology for 15 percent efficient solar cells with areas of about 1000 cm^2. Achieving this goal would enable polycrystalline thin film material to be a cost-effective electrical generator.

Keywords: Coatings and Films, Semiconductors, Chemical Vapor Deposition, Physical Vapor Deposition, Electrodeposition, Sputtering and Solar Cells

159. Deposition of III-V Semiconductors for High-Efficiency Solar Cells FY 1992
$4,000,000

DOE Contact: Richard King, (202) 586-1693
NREL Contact: John Benner, (303) 231-1396

This project performs applied research upon deposition of III-V semiconductors for high efficiency solar cells, both thin film for flat plate applications and multilayer cells for concentrator applications. Research centers upon depositing layers precisely controlled in terms of composition, thickness and uniformity and studying the interfaces between the layers. The materials are deposited by chemical vapor deposition, liquid phase epitaxial growth and molecular beam epitaxial growth. The long term goal of this area is to develop 35 percent efficient concentrator cells and 24 percent 100 cm^2 one-sun cells for flat plate applications. Achieving these goals would enable systems using these technologies to be cost-effective electrical generators.

Keywords: Semiconductors, Chemical Vapor Deposition, Solar Cells (Liquid Phase Epitaxial Growth, Molecular Beam Epitaxial Growth)

Materials Properties, Behavior, Characterization or Testing

160. Materials and Device Characterization

FY 1992
$3,000,000

DOE Contact: Richard King, (202) 586-1693
NREL Contact: Larry Kazmerski, (303) 231-1115

This project measures and characterizes materials and device properties. The project performs surface and interface analysis, electro-optical characterization and cell performance and material evaluation to study critical material/cell parameters such as impurities, layer mismatch and other defects that limit performance and lifetime. Techniques that are used include deep level transient spectroscopy, electron beam induced current, secondary ion mass spectroscopy, scanning electron microscopy and scanning transmission electron microscopy.

Keywords: Semiconductors, Nondestructive Evaluation, Surface Characterization, Microstructure and Solar Cells

Device or Component Fabrication, Behavior or Testing

161. High-Efficiency Crystal Silicon Solar Cells

FY 1992
$2,400,000

DOE Contact: Richard King, (202) 586-1693
NREL Contact: John Benner, (303) 231-1396
SNLA Contact: David Hasti, (505) 844-8161

This project performs applied research upon crystal silicon devices to improve solar-to-electric conversion efficiency. The project employs new coatings and/or dopants and other treatments to reduce electron-hole recombination at cell surfaces or in the bulk material. Control of point defects in crystalline silicon are being studied by a variety of techniques.

Keywords: Semiconductors, Solar Cells

Office of Renewable Energy Conversion

Geothermal Division

The primary goal of the geothermal materials program is to ensure that the private sector development of geothermal energy resources is not constrained by the availability of technologically and economically viable materials of construction. This requires the performance of long-term high risk co-sponsored materials R&D.

Materials Preparation, Synthesis, Deposition, Growth or Forming

162. Thermally Conductive Composites for Heat Exchangers

FY 1992
$97,000

DOE Contact: R. LaSala, (202) 586-4198
BNL Contact: L. E. Kukacka, (516) 282-3065

This project is investigating thermally conductive polymer-based composites for use as corrosion and scale-resistant liner materials for shell and tube heat exchangers in binary geothermal processes or for bottoming cycles in multi-stage flash plants. Corrosion and scaling on the brine side of carbon steel tubing in shell and tube heat exchangers have been major problems in the operation of geothermal processes. Compared to the cost of high alloy steels, a considerable economic benefit could result from the utilization of a proven corrosion resistant polymer concrete material if sufficient heat transfer and anti-fouling properties can be derived. The work consists of determinations of the effects of compositional and processing variables on the thermal and fouling properties of the composite, and measurements of the physical and mechanical properties after exposure to hot brine in the laboratory and in plant operations..

Keywords: Composites, Polymers, Corrosion, Heat Transfer, Scale-Resistant, Fabrication Technology

Materials Properties, Behavior, Characterization or Testing

163. Advanced High Temperature Geothermal Well Cements

FY 1992
$120,000

DOE Contact: R. LaSala, (202) 586-4198
BNL Contact: L. E. Kukacka, (516) 282-3065

Lightweight (<1.2 g/cc) chemically and thermally resistant well cements are needed to reduce the potential for lost circulation problems during well completion operations and to ensure long-term well integrity. Material designed for temperatures $>400°C$ will be needed as higher temperature resources are developed. Cements resistant to brines containing high concentrations of CO_2 at temperatures $>150°C$ are also needed. Emphasis is being placed on high temperature rheology, phase chemistry, and the mechanical, physical, and chemical resistance properties of the cured materials. Retarding admixtures required to maintain pumpability during placement operations are also being identified.

Keywords: Cements, Material Degradation, Strength, Transformation, Bulk Characterization, Drilling, Carbonation, Retarders

164. Advanced High Temperature Chemical Systems for Lost Circulation Control

FY 1992
$160,000

DOE Contact: R. LaSala, (202) 586-4198
BNL Contact: L. E. Kukacka, (516) 282-3065

The cost of correcting lost circulation problems occurring during well drilling and completion operations constitutes 20 to 30 percent of the cost of a geothermal well. The objective of the program is to develop advanced high temperature chemical systems which upon curing will yield an expandable high strength brine-resistant cementitious material. Emphasis is being placed upon high temperature rheology, phase chemistry, and the mechanical, physical and chemical resistance properties of the cured material. Optimization of the formulations with respect to various placement technologies is also being conducted.

Keywords: Cement, Pumpable Slurries, Strength, Transformation, Bulk Characterization

165. Corrosion Mitigation in Highly Acidic Steam Condensates

FY 1992
$100,000

DOE Contact: R. LaSala, (202) 586-4198
BNL Contact: L. E. Kukacka, (516) 282-3065

Increased HCl gas concentrations in the steam produced from geothermal wells at The Geysers in Northern California have resulted in severe corrosion problems in casings in the upper regions of wells where condensation may occur, in the well-head and transmission piping, and on turbine blades. The objective of the program is to optimize and field test polymer matrix composites for utilization as corrosion resistive liners on carbon steel components exposed to low pH steam condensates at temperatures up to ~200 °C. Emphasis is being placed on polymer and composite composition, metal surface modification, installation procedures and techniques for joining lined pipe sections.

Keywords: Polymers, Polymer Matrix Composites, Acid, Durability, Fabrication Techniques, Field Tests

166. High Temperature Chemical Bonding Systems

FY 1992
$150,000

DOE Contact: R. LaSala, (202) 586-4198
BNL Contact: L. E. Kukacka, (516) 282-3065

The unavailability of hydrolytically (300°C) stable chemical coupling systems that will bond high temperature elastomers to metal reinforcing substrates is constraining the development of high temperature tools needed for the drilling and completion of geothermal wells. The objective of this program is to develop advanced bonding systems consisting of elementally modified metal surfaces, elastomers and hydrothermally stable polyaryl-type polymeric adhesives which will chemically bond with each to form stable interfaces. Specific end-use applications include drillpipe protectors, rotating head seals, blow-out preventors, and downhole drillmotors.

Keywords: Elastomers, Chemical Bonding, Metals, Interfacial Characteristics, Hydrothermal Stability

Office of Energy Management

Advanced Utility Concepts Division

The Advanced Utility Concepts Division supports research and development of advanced energy storage and electrochemical conversion systems that will facilitate the substitution of renewable energy sources for fossil fuels—measures that will increase the reliability and efficiency of the energy economy. The goal is to provide reliable, inexpensive devices to mitigate the temporal and spatial mismatches between energy supply and energy demand. The research is divided into four subprograms: Superconductivity Systems, Utility Battery Storage, Thermal Storage, and Hydrogen Energy.

Materials Properties, Behavior, Characterization or Testing

167. High Temperature Composite Phase Change Material — FY 1992 $200,000

DOE Contact: Dr. Eberhart Reimers, (202) 586-4563
PNL Contact: Landis Kannberg, (509) 375-3919
Mississippi State University Contact: Professor G. A. Adebiyi, (601) 325-3260

The objective of the work is to develop high temperature sensible/latent heat storage media for use in a packed bed regenerator for the capture and reuse of waste heat. The near term application of this technology is recovery of heat currently exhausted into the air by flue gases in high-temperature industrial processes. The composite material consists of a ceramic matrix (sponge) in which a phase change material (salt eutectic) is imbibed forming a composite phase change material (CPCM). When the salt melts, capillary forces retain the salt in the ceramic matrix, providing form-stability to the PCM. Samples of conventional sensible storage material and the CPCM have been prepared and testing of the sensible material in a packed bed configuration has been undertaken. Testing with the CPCM will follow.

Keywords: Heat Storage, Phase Change Materials, Composites

168. Geochemistry Dynamics Associated with Groundwater Heating FY 1992
$130,000

DOE Contact: Dr. Eberhart Reimers, (202) 586-4563
PNL Contact: Landis Kannberg, (509) 375-3919
PNL Technical Contact: E. A. Jenne, (509) 376-4412

The objective of this effort is to obtain the equilibrium and kinetic data required to accurately predict important geochemical reactions and permeability reduction in an aquifer during thermal energy injection, storage and recovery. Models of solubility equilibria were substantially improved through accounting for temperature dependence on geochemical equilibria. Batch experiments were conducted to determine the impact of fines on geochemical reactions, focusing specifically on identification of the specific solid forms that are most influential in determining geochemical behavior. Mineral forms characterized include K-feldspars, quartz, chalcedony (a variety of poorly crystalline quartz), calcite dolomite and pyrite. Mineral forms still under investigation include other feldspars, amorphic silica and sepiolite.

Keywords: Minerals, Permeability, Aquifer

Device or Component Fabrication, Behavior or Testing

169. Phase Change Thermal Storage for Domestic Water Heating FY 1992
$90,000

DOE Contact: Dr. Eberhart Reimers, (202) 586-4563
PNL Contact: Landis Kannberg, (509) 375-3919
University of Florida - Gainsville Contact: D. Yogi Goswami, (904) 392-0851

The objective of this work is to develop new thermal energy storage prototypes that double the energy density of conventional electrically heated storage water heaters. The development effort includes the identification and development of phase change materials (PCM) and their encapsulating materials where appropriate. Several concepts are under development including use of cross-linked polyethylene and pentaerythritol as solid-solid PCMs. Encapsulation in lined and unlined pouches are being studied, including sealing methods such as ultrasonic welding, adhesives and thermal welding. Corrosion studies will be conducted on encapsulated materials. Theoretical models will be used to design prototype units that will then be constructed and tested.

Keywords: Phase Change Materials, Encapsulation, Corrosion

170. Phase Change Thermal Storage in Building Materials FY 1992
$90,000

DOE Contact: Dr. Eberhart Reimers, (202) 586-4563
PNL Contact: Landis Kannberg, (509) 375-3919
University of Dayton Contact: I. O. Salyer, (513) 229-2213

The objective of this effort is to develop and adapt low-cost, effective phase change systems that melt and freeze in the temperature range 23-25°C for building materials. The near-term emphasis is on incorporating these materials in plasterboard. Crystalline alkyl hydrocarbon materials (paraffin) have been identified as promising PCMs for wallboard applications. The paraffin is imbibed into interstices of the gypsum. Evaluation of critical performance factors includes characterization of the quantities of paraffin that can be imbibed, internal distribution of paraffin, effect of thermal cycling, propensity for leakage, combustibility and smoke evolution during combustion. Alternative methods for introducing the PCM into the plasterboard production process have been investigated, including imbibition in high-density polyethylene and dry mixing with high-surface-area silica. Performance has been sufficiently promising to persuade U.S. Gypsum to try a short plant production run with this material.

Keywords: Building Materials, Phase Change Materials, Paraffin

171. High-Temperature Superconductor Development for Electric Power Applications FY 1992
$4,385,000

DOE Contact: J. Daley, (202) 586-1165
Argonne National Laboratory Contact: R. Poeppel, (708) 252-5118

A parallel approach is employed to (1) increase J_C in an applied magnetic field; (2) improve mechanical properties, flexibility, and chemical stability; (3) develop composite conductors integrated with device design strategies; and (4) interact with industry and academia to insure technology transfer. Work is continuing on wire development using bulk, thick- and thin-film techniques. BSCCO(2223) tape has been produced by the powder-in-tube method and yielded critical currents of 37,000-51,000 A/cm^2 at 77 K and 150,000 A/cm^2 at 4.2 K in self-field. In addition to conventional post-deposition annealing at high temperatures, novel low-temperature processing methods are evaluated using optical *in situ* analyses combined with extensive *ex situ* analyses. Neutron diffraction techniques are being used to examine internal strains (stresses) in YBCO to provide feedback for better wire processing techniques. A low-temperature melt-growth process has been developed to fabricate YBCO pellets with minimized contamination from crucible materials which was common was in quench-melt growth process. Extensive work with private industry continued through collaborative efforts with Reliance Electric, Intermagnetics General, General Electric, Conductus, American Superconductor, and Commonwealth Research

Keywords: Superconductors, Bulk Conductors, Thick Films, Magnetic Bearings

172. Characterization and Development of High-Temperature Superconductors FY 1992 $600,000

DOE Contact: J. Daley, (202) 586-1165
Brookhaven National Laboratory Contact: D. Welch, (516) 282-3517

Present efforts are focused on the following areas: (1) the fabrication of textured $YBa_2Cu_3O_2$ (YBCO)-based conductors by continuous zone-partial-melting methods; (2) the development of mono- and multi-filament 2223 Bi-cuprate tapes (Intermagnetics General); (3) microstructural and electromagnetic properties characterization of materials; and (4) the design and operational issues of HTS magnets (Massachusetts Institute of Technology). AC losses of superconductors in non-ideal geometries are studied both experimentally and theoretically. Work is continuing to develop scalable processing techniques for thick-film composite YBCO tape. Textured YBCO composite tape has been successfully grown at 960 to 970°C at heat zone speeds of 25 mm/h. Critical transition temperature for the tape was 90 K at 77 K while critical current density was 104 A/cm^2 and 500 A/cm^2 for $H=0$ and 1 T, respectively.

Keywords: Superconductors, Composites, Tapes, Characterization

173. Electrodeposition of High-Temperature Superconductors FY 1992 $700,000

DOE Contact: J. Daley, (202) 586-1165
National Renewable Energy Laboratory Contact: R. McConnell, (303) 231-1019

This program has two objectives: to coordinate technology transfer activities leading to industrial partnerships for the purpose of aiding the commercialization of HTS products or services, and to perform conductor development activities. To foster technology transfer, a workshop on material issues for Tl-oxide superconductors was held in order to exchange information with industry and focus conductor development activities. Conductor development activities are dedicated to increasing the critical current density of superconductor coatings on long lengths of prototype conductors through a systematic study of substrates, buffer layers, post coating annealing and processing. An innovative electrodeposition process is being used to manufacture long coated wires and tapes from YBaCuO (YBCO), Bi(Pb)SrCaCuO (BSCCO), and TlBaCaCuO (TBCCO). The best result to date is 32,000 A/cm^2 in 0 T at 76 K and 10,000 A/cm^2 in 0.5 T at 76 K for electrodeposited TBCCO on an Ag foil. Collaborative work on the electrodeposition of thallium films is being conducted in conjunction with General Electric.

Keywords: Superconductors, Wires, Tapes, Electrodeposition

174. <u>Bulk and Thin-Film Materials Process Research for Practical High-Temperature Superconductor Development</u>

FY 1992
$1,000,000

DOE Contact: J. Daley, (202) 586-1165
Sandia National Laboratory Contact: T. Bickel, (505) 845-9301

The four research tasks underway are: process research on the material synthesis of HTS conductors, investigation on the synthesis and processing of thallium-based HTS thin and thick films, process development and characterization of HTS wire and tape, and cryogenic design of an HTS motor. These research efforts are focused toward the achievement of practical devices using scalable and manufacturable processing techniques. Studies continue on the effects of ceramic processing parameters on precursor powders and subsequently their effects on conductor fabrication. Conductor R&D includes work on thin- and thick-TBCCO film production through deposition and *ex situ* sintering and annealing, and both low and high density powder-in-tube production methods. The research program collaborates extensively with Nuclear Metals, Inc., HTS research programs at other national laboratories.

Keywords: Superconductor, Deformation, Thallium, Thick Film

175. <u>High-Temperature Superconductor Wire and Component Development for Electric Power Applications</u>

FY 1992
$4,200,000

DOE Contact: J. Daley, (202) 586-1165
Los Alamos National Laboratory Contact: D. Peterson, (505) 665-3030

The program has provided substantial advances in the synthesis and processing of yttrium-, bismuth-, and thallium-based HTS systems. Conductor development efforts have focused on the powder-in-tube processing approach using Bi-based materials. A concentric tube conductor design, "tube-within-tube," has been demonstrated as a feasible alternative to powder-in-tube processing. Precursor powders and bulk shapes continue to be synthesized using various techniques, stoichiometries, phase assemblages, and particle sizes to optimize superconducting characteristics and refine processing techniques. Evaluations continue on the effects that silver doping, mechanical deformations, and processing parameters have on various conductor characteristics. The introduction of flux pinning sites to HTS materials through doping and neutron irradiation were performed to investigate conductor performance in external magnetic fields. Work was completed on YBCO thin-film techniques, demonstrating YBCO pulse laser deposited films on sapphire substrates with J_C values >2 MA/cm^2.

Collaborative development projects with private industry continue with the following companies: American Superconductor, DuPont-Hewlett Packard, Nuclear Metals, Ceracon, Cryopower Associates and HiT_C Superconco.

Keywords: Superconductors, Wire, Tape, Thin Film, Deposition

176. Conductor Development, Processing of Wires and Tapes, Magnet Design and Testing, and Applications Development for High-Temperature Superconductors

FY 1992
$5,004,000

DOE Contact: J. Daley, (202) 586-1165
Oak Ridge National Laboratory Contact: R. Hawsey, (615) 574-8057

Research activities include development of: improved powder-in-tube conductors; thin- and thick-film conductors; techniques for production of high purity, homogenous, morphologically controlled precursor powders; metallic substrates and barriers compatible with melt-processing; rapid melt-texturing processes applicable to long lengths of conductor; various application device designs; and technology transfer activities through collaborative efforts with the private sector. Precursor development activities are focused on aerosol pyrolysis methods. Projects based on Bi-based materials concentrate on fabricating high J_C and long length conductors by the powder-in-tube process. Activities for TBCCO conductors have focussed on the development of practical substrates for TBCCO film processing. Grain boundaries and grain morphology are studied to enhance J_C through improved flux pinning and control of microstructure parameters. Tests performed on thick YBCO deposits and powder-in-tube conductors using a silver/palladium alloy indicate the alloy is chemically compatible for both YBCO and BSCCO. Work with private industry, including HTS coil development and testing activities, continued in conjunction with American Superconductor, Intermagnetics General, Reliance Electric, Superconductivity, Inc., and Westinghouse.

Keywords: Superconductors, Wire, Tape, Motor, Magnet

OFFICE OF ENERGY RESEARCH

	FY 1992
Office of Energy Research - Grand Total	$298,503,860
Office of Basic Energy Sciences	$273,741,659
Division of Materials Sciences	$261,500,000
Division of Engineering and Geosciences	$ 7,957,659
Engineering Research	$ 5,109,659
Materials Properties, Behavior, Characterization or Testing	$ 7,976,097
Bounds on Dynamic Plastic Deformation	137,000
In-Flight Measurement of the Temperature of Small, High Velocity Particles	493,000
Intelligent Control of Thermal Processes	530,000
Multivariable Control of the Gas-Metal/Arc Welding Process	158,000
Metal Transfer in Gas-Metal Arc Welding	122,300
Modeling and Analysis of Surface Cracks	196,000
Thermal Plasma Processing of Materials	141,928
Transport Properties of Disordered Porous Media from the Microstructure	70,676
Inelastic Deformation and Damage at High Temperature	134,000
Energy Changes in Transforming Solids	175,000
Optical Techniques for Superconductor Characterization	134,678
Effective Elastic Properties and Constitutive Equations for Brittle Solids Under Compression	59,798
Investigation of PACVD Protective Coating Process Using Advanced Diagnostic Techniques	179,995
Elastic-Plastic Fracture Analysis Emphasis on Surface Flaws	430,000
Continuous Damage Mechanics - Critical States	60,000
Quantitative Non-Destructive Evaluation of High Temperature Superconducting Materials	0
Development of Measurement Capabilities for the Thermophysical Properties of Energy-Related Fluids	551,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Division of Engineering and Geosciences (continued)	
Engineering Research (continued)	
Materials Properties, Behavior, Characterization or Testing (continued)	
Flux Flow, Pinning and Resistive Behavior in Superconducting Networks	69,500
An Investigation of the Effects of History Dependent Damage in Time Dependant Fracture Mechanics	94,027
Mixing and Settling in Continuous Metal Production	135,000
Application of Magnetomechanical Hysteresis Modelling to Magnetic Techniques for Monitoring Neutron Embrittlement and Biaxial Stress	99,912
Stability and Stress Analysis of Surface Morphology of Elastic and Piezoelectric Materials	98,695
Micromechanical Viscoplastic Stress-Strain Model with Grain Boundary Sliding	48,500
A Micromechanical Viscoplastic Strain-Strain Model with Grain Boundary Sliding	48,500
Modeling of Thermal Plasma Processes	232,000
Nondestructive Evaluation of Superconductors	200,000
Momentum and Heat Transfer Processes in Viscoelastic Fluids	73,391
Pulse Propagation in Inhomogeneous Optical Waveguides	83,537
Low Resistivity Ohmic Contacts Between Semiconductors and High-Tc Superconductors	98,488
The Evolution of a Hele-Shaw Interface and Related Problems in Dendritic Crystal Growth	66,116
Degenerate Four-Wave Mixing as a Diagnostic of Plasma Chemistry	95,328
Effect of Forced and Natural Convection on Solidification of Binary Mixtures	93,290

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Division of Engineering and Geosciences (continued)	
Geosciences Research	$2,848,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 351,000
Interaction Between Mineral Surfaces and Ions in Solution	100,000
Mineral Hydrolysis Kinetics	124,000
Transition Metal Catalysis in the Generation of Natural Gas	127,000
Materials Structure and Composition	$ 521,000
Three-Dimensional Imaging of Drill Core Samples Using Synchrotron Computed Microtomography	75,000
Grain Boundary Transport and Related Processes in Natural Fine-Grained Aggregates	141,000
IR Spectroscopy and Hydrogen Isotope Geochemistry of Hydrous Silicate Glasses	90,000
Crystal Chemistry of Hydroxyl and Water in Silicate Minerals	93,000
Investigation of Ultrasonic Wave Interactions with Fluid-Saturated Porous Rocks	59,000
Zircons and Fluids: An Experimental Investigation with Applications for Radioactive Waste Storage	63,000
Materials Properties, Behavior, Characterization or Testing	$1,976,000
Modification of Fracture Transport Properties of Rocks by Mechanical and Chemical Processes	180,000
Oxygen and Cation Diffusion in Oxide Minerals	180,000
Acoustic Emissions and Damage in Geomaterials	150,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Division of Engineering and Geosciences (continued)	
Geosciences Research (continued)	
Materials Properties, Behavior, Characterization or Testing (continued)	
Transport Properties of Fractures	129,000
Cation Diffusion Rates in Selected Silicate Minerals	187,000
Isotopic Tracer Studies of Diffusion in Silicates and Geological Transport Processes Using Actinide Elements	345,000
Experimental Measurement of Thermal Conductivity in Silicate Liquids	109,000
New Method for Determining Thermodynamic Properties of Carbonate Solid-Solution Minerals	83,000
Development of Synchrotron X-Ray Microspectroscopic Techniques and Application to Low Temperature Geochemistry	105,000
High Resolution TEM/AEM and SEM Studies of Fluid Rock Interactions Using Cu, Ag, Se, Cr and Cr-Bearing Solutions and Phyllosilicates	134,000
Thermodynamics of Minerals Stable Near the Earth's Surface	130,000
Poroelasticity of Rock	244,000
Division of Advanced Energy Projects	$ 4,284,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 2,840,000
Metallic Multilayer and Thin Film Fabrication	337,000
Synthesis of New High Performance Lubricants and Solid Lubricants	272,000

OFFICE OF ENERGY RESEARCH (Continued)

		FY 1992
Division of Engineering and Geosciences (continued)		
Division of Advanced Energy Projects (continued)		
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)		
Design of Materials with Photonic Band Gaps		299,000
High-Flux, Large-Area Carbon-Cluster Beams for Thin Film Deposition and Surface Modification		445,000
Novel Composite Coatings for High Temperature Friction and Wear Control		428,000
Synthesis of Advanced Composite Ceramic Precursor Powders by the Electric Dispersion Reactor		179,000
New Ion Exchange Materials for Environmental Restoration and Waste Management		430,000
Development of an Ion Replacement Electrorefining Method		450,000
Materials Properties, Behavior, Characterization or Testing	$	658,000
A Study of Potential High Band-Gap Photovoltaic Materials for a Two Step Photon Intermediate Technique in Fission Energy Conversion		294,000
Nonlinear Optics in Doped Fibers		364,000
Device or Component Fabrication, Behavior or Testing	$	786,000
Research on Magnet Replicas and the Very Incomplete Meissner Effect	$	341,000
Ultrafast Molecular Electronic Devices		445,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program	$24,762,201*
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 9,430,225
Phase I Projects:	
On-Line Diagnostics for Fullerene Synthesis	49,951
Development of High-Selectivity, High-Permeability, Flexible Plasma-Polymer Membranes	50,000
Development of a Novel Reverse-Osmosis Membrane with High Rejections for Organic Compounds	49,954
Selective Ceramic Membranes from Hybrid Templates	50,000
A New Class of Ionomer Membranes	50,000
Stoichiometric Precursors for Preparation of Advanced Metal Oxide Solid Electrolytes	49,996
A Thin Solid Film Electrochemical Oxygen Partial Pressure Sensor for Control of Low Vacuum Inert Gas Processes	50,000
Novel Durable Sol-Gel Fiber-Optic Sensors for Use in Elevated Temperature and/or Pressure Environments	50,000
Development of a Process to Synthesize Tubular Fullerenes	50,000
Molecular-Sieve-Modified Polymeric Membranes for Gas and Vapor Separation	50,000
A Low Cost Process for Manufacture of Fullerenes	40,569
Continuous Production of Fullerenes from Hydrocarbon Precursors	49,927
Preparation of Low-Density Microcellular Materials from Fullerenes	49,720
A Novel Fabrication Method for Diamond Composites	50,000
In Situ Surface Elemental Analysis/Process Control at Millitorr Pressure During Superconductor Film Deposition	50,000

*Includes 73 new Phase I and 20 new Phase II projects initiated in FY 1992 and 23 Phase II projects initiated in FY 1991. The funding shown for each Phase II project is the total allocated for the duration of the project (up to two years).

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Phase I Projects: (continued)	
Control of Thin Film Microstructures by Gasdynamic Energy Deposition	50,000
A Generic Chemical Processing Technology for the Production of Nanostructured Composite Materials	49,823
Fast-Pulse Hot Pressing with Fine-Scale Adiabatic Heating	48,972
Production of Controlled Microstructure Nanophase Ceramic Powders	50,000
Ductile High-Temperature Superconducting Alloys	49,804
New Gadolinium-Boron Compounds for Neutron Capture Therapy	49,986
Lead Sulfate Scintillators for Use in Position Emission Tomography	50,000
Refractory Metal Coatings on Carbon/Carbon Composites for First Wall Applications	49,970
A Thermal Composite Plasma Facing Material	49,649
A Niobium-Tin Multifilamentary Composite Superconductor with Artificial Copper (Bronze) Inclusions	49,996
A Porous Metal Heat Exchanger to Cool a Microwave Cavity	49,999
Development of Silicon Carbide Ceramic Composites for Fusion Reactor Applications	50,000
Addition of Silicon to the Copper Between Niobium-Titanium Filaments to Reduce Interdiffusion	49,986
Improved Critical Current Density at High Magnetic Flux in Niobium-titanium-tantalum Superconductors by Artificial Pinning Centers	49,986
Radiation Damage Resistant Silicon for Particle Physics Detectors	50,000
Epitaxial Wide Bandgap Semiconductor Films for Improved Detectors and Electronics	50,000
A Hydrogenated Amorphous Silicon Particle Detector	50,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Phase I Projects: (First Year) (continued)	
Epitaxial Silicon Carbide Growth by Plasma Enhanced Chemical Vapor Deposition	50,000
Development of High-Field Chevrel Phase Superconducting Wires Using Low Temperature Processing	49,996
Indium Iodide Photoconductive Detectors for Use in Scintillation Spectroscopy	50,000
Casting of Metallic Nuclear Fuel Pins	49,758
Enabling Materials Technology for Nuclear Propulsion	50,000
Jet Vapor Deposition of Corrosion Resistant Coatings on Critical Molten Carbonate Fuel Cell Components	50,000
Lithium-Graphite Intercalation Anodes	50,000
Phase II Projects: (First Year)	
Graphite and Metal Oxide Catalyst Supports for Rechargeable Oxygen Electrodes	500,000
Development of Hollow-Fiber Modules for the Purification of Natural Gas	493,602
Inexpensive Pathways for the Synthesis of p-Boronophenylalanine and New Boron Containing Agents	500,000
An Improved Method of Introducing Additional Alloying Elements into Nb_3Sn in Internal-Tin Processes	499,991
Phase II Projects: (Second Year)	
Single CrystalMolybdenum Mirrors for High Power Vacuum Ultraviolet and X-ray Radiation	499,026
Ultrathin Metal Membranes	500,000
Changes in Niobium Tin Conductors Made by the Internal Tin Method to Improve Magnet Performance	500,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Phase II Projects: (Second Year) (continued)	
Alternative Fabrication Processes for Ultrafine Filament, Metal Matrix Microcomposites	500,000
A Novel Carbon First Wall Material	499,573
Development of Radiation-Resistant Copper Matrix Composites	500,000
Development of New Radiation Resistant Large Stokes Shifted Intermolecular Proton Transfer Fluorescent Composites	500,000
More Economical Low Loss Niobium Tin for Pulsed Field Applications, Made by a Modified Internal Tin Process	499,991
A New Semiconductor Photosensor for Scintillation Spectroscopy	500,000
Magnetically Enhanced Plasma Deposition of Intrinsic Amorphous Hydrogenated Silicon Layers in Roll-to-Roll Systems	500,000
A Novel Precursor Yarn for Advanced Automotive Composite Structures	500,000
Materials Properties, Behavior, Characterization or Testing	$6,643,610
Phase I Projects:	
Pseudospectral Quantum Chemistry for Periodic Systems	50,000
Improved Scintillator Materials for Medical Applications	50,000
A Carbon-Carbon Repair System for the First Wall of the International Thermonuclear Experimental Reactor	50,000
Aluminum-Wettable Coatings on Graphite for Improved Brazed Joints	50,000
Photon-Stimulated Desorption from Cold Surfaces	49,138
Radiation Hard Diamond Transistors for the Superconducting Super Collider	50,000

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Materials Properties, Behavior, Characterization or Testing (continued)	
Phase I Projects: (continued)	
Large Area Distributed Electronics on Glass by Ion Implantation of Silicon-Germanium Alloys	49,945
Development of Methods to Substantially Reduce Photodesorption from Storage Ring Photo Absorbers	50,000
Silicides for Thermoelectric Conversion	50,000
Ceramic Matrix Composites for Magnetohydrodynamic Air Preheaters	49,930
Novel Energy-Efficient Membrane System for the Recovery of Volatile Organic Contaminants from Industrial Process Gases	49,907
Polymer Electrolytes for High Voltage Batteries	50,000
A Polymer Electrolyte for Lithium Batteries	49,997
Phase II Projects: (First Year)	
Thermophotovoltaic Cogenerators for Advanced Integrated Appliances	499,737
Spontaneous Natural Gas Oxidative Dimerization Across Mixed Conducting Ceramic Membranes	499,996
A Membrane Process for Hot-Gas Cleanup and Decomposition of H_2S to Elemental Sulfur	499,425
In-Situ FT-IR Diagnostics for Coal Liquefaction Processes	499,675
Deep Hole Drilling in Refractory Metals Using Abrasive Waterjets	497,035
Improved Properties in Nb 46.5 Weight % Ti Materials for the SSC by Reducing the Separation Between Filaments	499,988
Multifilamentary Nb_3Sn Superconducting Wire Using APC, Composite Filaments with Improved High Field Performance	499,905
A Novel Joining Method for Graphite and Carbon-Carbon Composites	499,266

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Materials Properties, Behavior, Characterization or Testing (continued)	
Phase II Projects: (Second Year)	
Utilization of Fluidized Bed Residuals for Producing Synthetic Aggregate by Vacuum Extrusion	499,885
An Evaluation of Erosion Resistance in a Simulated Fluidized Bed Combustor Environment of Coatings Applied Using a Hypersonic Spray Process	500,000
A Novel, Robust, High Repetition, High Brightness Cathode	499,937
An All-Solid-State Titanium-Doped Sapphire Laser Source for Production of Polarized Electrons	499,844
Device or Component Fabrication, Behavior, or Testing	$8,688,366
Phase I Projects:	
Nondestructive Measurement of Residual Stresses in Power Plant Piping Welds Using Magnetoelastic Techniques	49,329
Digital Processing Electronics for X-ray Detector Arrays	50,000
A Cold/Thermal Beam Bender Using Capillary Optics to Increase the Number of End-Guide Instrument Positions	50,000
A Thomson-Scattering Plasma Diagnostic for Materials Testing	50,000
Eddy-Current Nondestructive Testing Methods for On-Line Detection of Cable Manufacturing Defects	50,000
Low-Cost Microstrip Detectors on Conductivity-Modified Kapton	49,828
Development of a Low Cost Method for the Fabrication of Fine Filament Niobium-Titanium Conductor	49,991
A Gallium Arsenide Heterojunction Bipolar Transistor for High-Speed Electronic Instrumentation	49,878

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Device or Component Fabrication, Behavior, or Testing (continued)	
Phase I Projects: (continued)	
High Strength Mono- and Multifilament High Temperature Superconductors for High Field Applications	49,974
Fabrication of Niobium-Aluminum Superconducting Strands Using Mechanical Alloying	49,987
Novel Detector for Neutron Spectroscopy	50,000
Acoustic Sensor Development for Use in Robotics	49,900
A Uranium Carbide/Rhenium Thermionic Fuel Element	48,933
Durable, Low Cost Ceramic Materials for Use in Hot Gas Filtration Equipment	50,000
An Innovative Device to Enhance Jet Pulse Cleaning of Ceramic Candle Filters	46,347
Microwave Joining of Silicon Carbide for Fabrication of Ceramic Heat Exchanger Assemblies	49,952
A Torsional Rheometer for Measuring the Properties of Coal and Coal Slurries	49,694
High Cation Mobility Lithium Polymer Batteries	50,000
A Real-Time X-ray Detector	50,000
Multiplexed Thermopile-Based Infrared Imaging Arrays	46,364
Novel Ultrasonic Thermometers with Non-Metal Sensing Elements for Hostile Environments	46,524
Phase II Projects: (First Year)	
Metal Hydride Refrigerators	499,935
A Catalytic Membrane Reactor for Facilitating the Water-Gas Shift Reaction at High Temperature	492,117
A Single Manifold, Radial Flow, Solid Oxide Fuel Cell	499,402
Ceramic/Metal Elements for MHD Sidewalls	499,991
Fabrication of Tungsten and Tungsten-Molybdenum Alloy Tubing	498,555

OFFICE OF ENERGY RESEARCH (Continued)

	FY 1992
Small Business Innovation Research Program (continued)	
Device or Component Fabrication, Behavior, or Testing (continued)	
Phase II Projects: (First Year) (continued)	
A Distributed Fiber Optic Sensor for Reversible Detection of Atmospheric CO_2	499,954
An Electron Bombarded Semiconductor Device	499,756
Silicon Junction Diode Absolute Radiometers for Plasma Diagnostics in the Soft X-ray and Vacuum Ultraviolet Spectral Region	263,960
Phase II Projects: (Second Year)	
A Ceramic Filter for Removal of Particulates from Flue Gas	499,990
A Ceramic Filter for Removal of Particulates from Hot Gas Streams	499,985
Development of a Flue Gas Desulfurization Process Using a Novel Regenerative Hydrogen Bromide System	499,927
Perovskite Solid Electrolytes for Intermediate Temperature Fuel Cells	499,990
Development of Flexible Super-Lattice Artificial Crystals for Use in Fixed-Source Johann X-ray Spectrometers with Application to Analytical Transmission Electron Microscopy	500,000
An Efficient X-ray Wavelength Spectrometer for Improved Elemental Analysis on the Analytical Electron Microscope	399,601
A Fiber Optic Long Counter	499,086
Advanced High-Concentration High-Efficiency Tandem Photovoltaic Assemblies	499,416

OFFICE OF ENERGY RESEARCH

The Director of Energy Research is responsible for three major outlay programs: Basic Energy Sciences, High Energy and Nuclear Physics, and Fusion Energy. The Director of Energy Research also advises the Secretary on DOE physical research programs, university-based education and training activities, grants, and other forms of financial assistance. The Director also carries out additional duties assigned to the Office related to basic and advanced research, and monitors the well-being and management of the multiprogram laboratories under the jurisdiction of the Department.

Four multiprogram and seven single-purpose laboratories are administratively assigned to the Office of Energy Research. The multiprogram facilities are Argonne National Laboratory, Oak Ridge National Laboratory, Brookhaven National Laboratory, and Lawrence Berkeley Laboratory. The single-purpose or specialized laboratories are the Bates Linear Accelerator Facility at the Massachusetts Institute of Technology, the Ames Laboratory at the Iowa State University, the Fermi National Accelerator Laboratory, the Notre Dame Radiation Laboratory, the Princeton University Plasma Physics Laboratory, the Michigan State University Plant Research Laboratory, and the Stanford Linear Accelerator Center. The multiprogram laboratories conduct significant research activities for other DOE programs (Conservation, Nuclear, etc.) and other Federal agencies, while the seven specialized laboratories are funded almost totally by the Office of Energy Research.

The Office of Energy Research conducts materials research in the following offices and divisions:

- Office of Basic Energy Sciences: Division of Engineering and Geosciences; Division of Materials Sciences; Division of Advanced Energy Projects
- Office of Health and Environmental Research: Division of Physical and Technologies Research
- Office of Fusion Energy
- Small Business Innovation Research Program

Office of Basic Energy Sciences

Division of Materials Sciences

This basic research program has several roles. One is to increase the understanding of materials properties, behavior, and phenomena in those classes of materials that either presently or in the future might be important to the mission of the Department of Energy. Another concerns the development of new forefront analytical instruments and facilities that are used to probe the structure and behavior of matter. Thus this program carries a major responsibility for many of the nation's premier research facilities including several neutron sources, a synchrotron radiation source, processing facilities, and frontier electron microscopes. Some of the materials research has a specific relationship to an identified energy technology (e.g., photovoltaic phenomena for solar energy conversion, fast-ion diffusion for solid electrolytes in fuel cells and batteries); some is related to many energy technologies simultaneously (e.g., hydrogen embrittlement, corrosion, high temperature structural metals and ceramics); and some is important to fundamental understanding of new experimental and theoretical research tools.

This research is conducted at DOE laboratories, universities, and to a lesser extent at industrial laboratories by metallurgists, ceramists, solid state physicists, and materials chemists in about 100 different institutions.

There are three subprograms:

- Metallurgy and Ceramics seeks to understand the synergistic relationship between properties/behavior, structure, and processing parameters of materials.

- Solid State Physics is concerned with understanding the interactions of electrons, atoms, and defects and their role in determining the structure and properties of condensed matter.

- Materials Chemistry focuses on understanding the chemical properties of materials and their relationship to composition, structure, and specimen environment.

The operating funds for FY 1992 for the Division of Materials Sciences were $261,500,000. This was allocated to 443 projects. The approximate funding distribution for FY 1992 was:

	$ (Millions)
Materials Preparation, Synthesis, Deposition, Growth or Forming	27.0
Materials Structure and Composition	28.3
Materials Properties, Behavior, Characterization or Testing	84.2
Device or Component Fabrication, Behavior or Testing	--
Facilities	122.0

The DOE contact for this Division is Iran Thomas, (301) 903-3427. For specific detailed information, the reader is referred to DOE publication Materials Sciences Programs Fiscal Year 1992 (DOE/ER-0581P dated February 1993). This publication contains summaries of all funded programs at DOE laboratories, summaries of all Small Business Innovation Research programs; major user facilities (descriptive information); other user facilities; cross-cutting indices; investigators, materials, techniques, phenomena, environment. Limited copies may be obtained by calling (301) 903-3427.

Division of Engineering and Geosciences

Materials research in the Division of Engineering and Geosciences is sponsored by two different research programs, as described below.

The BES Engineering Research Program was started in 1979 to help resolve the numerous serious engineering issues arising from efforts to meet U.S. energy needs. The program supports fundamental research on broad, generic topics in energy related engineering—topics not as narrowly scoped as those addressed by the shorter term engineering research projects sponsored by the various DOE technology programs. Special emphasis is placed on projects which, if successfully concluded, will benefit more than one energy technology.

The broad goals of the BES Engineering Research Program are: (1) To extend the body of knowledge underlying current engineering practice so as to create new options for enhancing energy savings and production, for prolonging useful equipment life, and for reducing costs without degradation of industrial production and performance quality; and (2) To broaden the technical and conceptual base for solving future engineering problems in the energy technologies. The DOE contact for this Program is Oscar P. Manley, (301) 903-5822.

The BES Geosciences Research Program supports research that is fundamental in nature and of long-term relevance to one or more energy technologies, national security, energy conservation, or the safety objectives of the Department of Energy. It is also concerned with the extraction and utilization of such resources in an environmentally acceptable way. The purpose of this program is to develop geoscience or geosciences-related information relevant to one or more of these Department of Energy objectives or to develop the broad, basic understanding of geoscientific materials and processes necessary for the attainment of long-term Department of Energy goals. In general, individual research efforts supported by this program may involve elements of several different energy objectives. The DOE contact for this Program is William C. Luth, (301) 903-5829.

Engineering Sciences Research

Materials Properties, Behavior, Characterization or Testing

177. Bounds on Dynamic Plastic Deformation

FY 1992
$137,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Argonne National Laboratory Contact: C. K. Youngdahl, (312) 972-6149

Analytical studies are being performed to develop load correlation parameters which can be used in approximating or bounding the dynamic plastic deformation of structures. In many applications where the load is transmitted to the structure through a fluid, details of the load history and spatial distribution significantly affect the final plastic deformation. The objective of the program is to devise load correlation parameters based on various weighted integrals of the time-space load distributions which can be used to characterize the effects of the load without resorting to detailed numerical analysis. These load correlation parameters have three important uses: to perform design and safety analyses of structures over a wide range of design variables and loadings; to validate computer programs which have a nonlinear dynamic plasticity capability; and to correlate experimental simulations with actual or predicted events. The dynamic plastic deformation of some basic structural configurations will be analyzed for loadings which vary both in magnitude and region of application with time. Load correlation parameters will be hypothesized and their usefulness in predicting final plastic deformation will be determined. The analyses will be based initially on a rigid, perfectly plastic material model and small deformation response, but will be extended to include strain hardening, and initial elastic response period, and large deformation interactions.

Keywords: Plastic Deformation

178. In-Flight Measurement of the Temperature of Small, High Velocity Particles

FY 1992
$493,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Idaho National Engineering Laboratory Contact: J. R. Fincke, (208) 526-2031

The measurement of temperature, velocity, enthalpy, and species concentration in high temperature gases such as weakly ionized thermal plasmas has considerable importance in the areas of plasma thermal spray and the thermal plasma synthesis of materials. In particular, the dynamics of the plasma, the interaction of the plasma with its surroundings and the behavior of particles immersed in the plasma surrounding it are important in the understanding, development and optimization of plasma process that involve fine powders. Laser based measurement techniques have been developed at this laboratory and are being applied to the study of thermal plasmas. In addition to the laser techniques enthalpy probes coupled to a mass spectrometer also provide temperature, velocity and concentration information. The experimental data produced is used to benchmark the modeling work done under a related program in "Modeling of Thermal Plasma Processes" (see J. Ramshaw, Idaho National Engineering Lab).

Keywords: Plasma Processing, Particle/Plasma Interaction

179. <u>Intelligent Control of Thermal Processes</u> <u>FY 1992</u> $530,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Idaho National Engineering Laboratory Contacts: H. B. Smartt, (208) 526-8333 and J. A. Johnson, (208) 526-9021

This project addresses intelligent control of thermal processes as applied to materials processing. Intelligent control is defined as the combined application of process modeling, sensing, artificial intelligence, and control theory to process control. The intent of intelligent control is to produce a good product without relying on post-process inspection and statistical quality control procedures. The gas metal arc welding process is used as a model system; considerable fundamental information on the process has been developed at INEL and MIT during the past six years. Research is being conducted on an extension of the fundamental process physics, application of neural network-like dynamic controllers and signal/image processors, and development of noncontact sensing techniques.

Tasks include physics of nonlinear aspects of molten metal droplet formation, transfer, and substrate thermal interaction; understanding substrate thermal interaction; understanding the relationship of neural network structure and associated learning algorithm to model development and learning dynamics in neural networks with the objective of obtaining a fundamental understanding of network transfer functions; and advanced sensing, including the propagation and interaction of ultrasound in metallic solid and liquid media.

Keywords: Welding, Ultrasonic Sensing, Optical Sensing

180. Multivariable Control of the Gas-Metal Arc Welding Process — FY 1992 $158,000

DOE Contact: Oscar P. Manley, (301) 903-5822
MIT Contact: David E. Hardt, (617) 253-2429

The Gas-Metal Arc Welding Process (GMAW) is a highly productive means for joining metals and is being used increasingly for structures and pressure vessels. The overall objective of this work is to examine the problem of simultaneous regulation of all real-time attributes of a weld. Past work has established the viability of independent control of thermal characteristics and the present work is examining the geometric aspects of weld pool control.

One objective of this work is to develop basic process modeling and control schemes to allow independent regulation of the weld bead width and height. A control model relating wirefeed and travel speed to width and height was developed using transfer function identification techniques applied to a series of step welding tests. We are developing a control system to independently regulate the weld bead width and the width of the heat affected zone. Initial work is concentrating on simulation of wide seam welding using an analytical heat transfer model as well as a finite difference process model. A key issue in the problem is the strong coupling between the inputs (current and travel speed) and the outputs. The use of high frequency transverse motion of the torch is being investigated as a means of overcoming this coupling. Once the control latitude is increased, a two variable control scheme based on both video and infrared sensing will be implemented.

Finally, the depth of penetration of a weld is the most important indicator of weld strength, yet it is the one variable that is essentially impossible to measure directly. A real-time depth estimator has been developed based on solution of an inverse heat transfer problem. Surface temperature measurements from the top and bottom of the weld have shown accurate and rapid convergence and development of a depth control system based on this estimator is now being processed.

This project is a collaborative program with INEL.

Keywords: Welding, Control

181. Metal Transfer in Gas-Metal Arc Welding — FY 1992 $122,300

DOE Contact: Oscar P. Manley, (301) 903-5822
MIT Contact: T. W. Eagar and J. Lang, (617) 253-3229

The present research is part of a cooperative progrram among faculty at MIT and staff at the Idaho National Engineering Laboratory (INEL) to develop a sound understanding of the arc welding process and to develop sensing and control methods that can be used to automate the gas-metal arc process.

The research during the current year has reviewed methods of filtering the voltage and current waveforms during pulsed current welding in order to extract signals which can be used to control the process. A new process control system has been developed and integrated with the welding equipment. Work has begun to study methods of mechanically controlling droplet detachment from the welding electrode.

Keywords: Welding, Control

182. Modeling and Analysis of Surface Cracks FY 1992
$196,000

DOE Contact: Oscar P. Manley, (301) 903-5822
MIT Contacts: David M. Parks, (617) 253-0033 and F. A. McClintock, (617) 253-2219

This research focuses on the analysis of ductile crack initiation, growth and instability in part-through surface-cracked plates and shells. The overall approach consists of careful calculations of crack front stress and deformation fields, and correlation of cracking with experimental observations being conducted at the Idaho National Engineering Laboratory. Recently, significant progress has been achieved in developing and applying a two-parameter description of crack front fields.

Simplified engineering applications of surface crack analysis are being developed in the context of the line-spring model. Specific enhancements include improved elastic-plastic procedures for the practically important case of shallow surface cracks, as well as simple methods for calculating the T-stress along surface cracks fronts.

Detailed elastic-plastic stress analyses of cracked structural geometries provide a basis for interpreting experimental observations, for quantitatively assessing inherent limitations of nonlinear fracture mechanics methodology, and for extending these boundaries through development of two-parameter characterization of crack tip fields. Simplified but accurate analytical methods are also under development for analysis of surface-cracked plates and shells. Emphasis is placed on better understanding complex three-dimensional features of elastic-plastic crack tip fields.

Keywords: Fracture

183. Thermal Plasma Processing of Materials FY 1992
$141,928

DOE Contact: Oscar P. Manley, (301) 903-5822
University of Minnesota Contact: E. Pfender, (612) 625-6012

The objective of this research project is to study analytically and experimentally specific thermal plasma processes for materials treatment. Processes of interest include the synthesis of ultrafine ceramic powders and of films.

During the past year our efforts have concentrated on characterizing the thermal plasma chemical vapor deposition (TPCVD) process of diamond films onto various substrates. Modeling of the situation close to the substrate indicates extremely steep temperature and concentration gradients pointing to the important of thermal diffusion.

Very high diamond deposition rates up to 1 mm/hr have been observed with a plasma reactor with recirculation eddies. A series of diagnostic studies have been initiated to facilitate an understanding of the main reasons for the observed high deposition rates.

Keywords: Plasma Processing, Plasma Diagnostics

184. Transport Properties of Disordered Porous Media from the Microstructure
FY 1992
$70,676

DOE Contact: Oscar P. Manley, (301) 903-5822
Princeton University Contact: S. Torquato, (609) 258-4600

This research program is concerned with the quantitative relationship between transport properties of a disordered heterogeneous medium that arise in various energy-related problems (e.g., thermal or electrical conductivity, trapping rate, and the fluid permeability) and its microstructure. Attention will be focused on studying the effect of porosity, spatial distribution of the phase elements, interfacial surface statistics, anisotropy, and size distribution of the phase elements, on the effective properties of models of both unconsolidated media (e.g., soils and packed beds of discrete particles) and consolidated media (e.g., sandstones and sintered materials).

Both theoretical and computer-simulation techniques have been employed to quantitatively characterize the microstructure and compute the transport properties of disordered media.

Keywords: Disordered Media

185. Inelastic Deformation and Damage at High Temperature FY 1992
$134,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Rensselaer Polytechnic Institute Contact: Erhard Krempl, (518) 266-6432

A combined theoretical and experimental investigation is performed to study the biaxial deformation and failure behavior of engineering alloys under low-cycle fatigue conditions at elevated temperature. The purpose is to characterize the material behavior in mathematical equations which are ultimately intended for use in inelastic stress analysis and life prediction. Creep-fatigue interaction and ratchetting are of special concern. The long-term goal is the development of a finite element program that can directly calculate the life-to-crack initiation of a component under a given load history.

Keywords: Fracture, Damage

186. Energy Changes in Transforming Solids FY 1992
$175,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Stanford University Contacts: George Herrmann, David M. Barnett, (415) 723-4143

Heterogenization techniques developed with DOE support. The methods have been extended to provide a number of universal formulae valid for the average stresses between two holes or inclusions, for inclusions with imperfect interfaces, and for stresses in cylindrical and plane layered media. A new methodology to establish conservation laws for dissipative systems has been advanced, and a theory of stressed solids prone to damage has been formulated based on the Gibbs free energy.

Another portion of our research has as its objective the development of further understanding of subsonic and supersonic surface waves and interfacial and bulk waves in anisotropic linear elastic solids. New investigations of so-called "generalized surface waves" and Stoneley waves in pre-stressed anisotropic bimaterials have also been undertaken.

Keywords: Stress Analysis, Materials Science

187. Optical Techniques for Superconductor Characterization FY 1992
$134,678

DOE Contact: Oscar P. Manley, (301) 903-5822
Stanford University: G. S. Kino, (415) 497-0205

The aim of this project is to develop a photothermal microscope for noncontact testing of materials. Techniques of this kind are particularly well suited to the determination of thermal parameters, and anisotropy of small samples.

One example of the work is the measurement of high temperature superconductors over a range of temperatures from room temperature through the critical temperature T_c down to 20°K. A modulated laser beam, focused to less than 1 m diameter, impinges through a sapphire window onto a sample of Bi-Ca-Sr-Cu-O in a helium cryostat and periodically modulates its temperature. This process excites a thermal wave, which can be detected by the variation in reflected signal amplitude of a second focused laser beam, due to the change of refractivity with temperature. The sample can be rotated under the beams and the thermal diffusion coefficient, its anisotropy and its magnitude can be measured from the phase delay of the thermal wave. By measuring the amplitude of the thermal wave, material phase changes associated with superconductivity can be measured. A pronounced peak in amplitude is seen at the critical temperature T_c. Even stronger effects of this type are observed with charge density waves in a variety of materials.

Keywords: Nondestructive Evaluation, Acoustic Sensors

188. Effective Elastic Properties and Constitutive Equations for Brittle Solids Under Compression FY 1992
$59,798

DOE Contact: Oscar P. Manley, (301) 903-5822
Tufts University Contact: Mark Kachanov, (617) 628-5000, ext. 2821

The knowledge of effective elastic properties of solids with cracks appears to be of increasing engineering importance. Extensive microcracking in structural elements working under conditions of high temperatures or irradiation, microcracking in composite materials under fatigue conditions may noticeably reduce the stiffness of the material and make it anisotropic. Understanding and prediction of these changes are essential for proper design and strength and lifetime assessments.

A new approach to many cracks problems based on interrelating the average tractions on individual cracks is introduced. Its advantages are that it yields simple analytical results which are quite accurate up to very high crack densities and that it can be applied to crack arrays or arbitrary geometry. Relation between deterioration of elastic properties and "damage" is discussed.

Keywords: Fracture, Elasticity

189. Investigation of PACVD Protective Coating Process Using Advanced Diagnostic Techniques — FY 1992 $179,995

DOE Contact: Oscar P. Manley, (301) 903-5822
United Technologies Research Center Contact: W. C. Roman, (203) 727-7590

The research objective is the comprehensive experimental investigation of the fundamental nonequilibrium reactive plasma assisted chemical vapor deposition (PACVD) process as applied to hard face coatings (e.g., TiB_2 or diamond). Nonintrusive laser diagnostics (e.g., laser induced fluorescence (LIF) and coherent anti-Stokes Raman spectroscopy (CARS) are being used to probe gas phase species, concentrations and rotational temperatures *in situ*. Detailed coating characterization is accomplished using Auger, Ion Scattering and secondary ion mass spectroscopies (AES, ISS and SIMS) and complementary techniques. In addition, coating characteristics such as smoothness, adhesion (UTRC custom built pin-on-disc apparatus) and hardness (state-of-the-art nanoindenter apparatus) are measured. Gas phase spectroscopy is interpreted through chemical kinetic modelling and will be correlated to coating characteristics thus providing a predictive capability that is severely lacking in the present science base of advanced protective coatings.

Keywords: Coatings, Plasma Diagnostics

190. Elastic-Plastic Fracture Analysis Emphasis on Surface Flaws — FY 1992 $430,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Idaho National Engineering Laboratory Contact: W. G. Reuter, (205) 526-0111

The objective is to improve design and analytical techniques for predicting the integrity of flawed structural components. The research is primarily experimental, with analytical evaluation guiding the direction of experimental testing. Tests are being conducted on a material (a modified ASTM A-710) exhibiting a range of fracture toughness but essentially constant yield and ultimate tensile strength. As test temperature increases, the specimen configuration-fracture toughness relationship complies initially with requirements for linear elastic-fracture mechanics and extends beyond the range of a J-controlled field. Presently, compact tension and bend specimens are being used to develop state-of-the-art fracture mechanics.

Metallographic techniques are being used to measure crack tip opening displacement and remaining ligament size for comparison with analytical models. Other techniques including microphotography and the replicating of the crack tip region, for future metallographic examination, are being used to complement the above measurements to identify limits and capabilities of each technique. Moire interferometry techniques are being used to evaluate and quantify the deformation in the crack region. These data are being used to experimentally measure J and CTOD for standard (CT and SENB) specimens as well as for specimens containing surface cracks.

The above tests have been supplemented by using specimens fabricated from aluminum (dimple rupture only) and titanium. The titanium specimens are being used to study the fracture behavior and the ability of existing models to predict failure for weldments. Moire interferometry techniques are being used to study the local constitutive behavior and the fracture process at the crack tip region of the weldment.

Keywords: Fracture, Metals: Ferrous

191. Continuous Damage Mechanics - Critical States

FY 1992
$60,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Arizona State University Contact: D. Krajcinovic, (602) 965-8656

The research during the fourth, and last, year of the research was focused almost entirely on the two tasks: (a) response of microcrack weakened solids in the vicinity of the critical state, and (b) initial exploration of the use of Preisach model in fatigue analyses.

The studies of critical states were concentrated on fundamental issues such as the determination of the proximity parameter, universal parameters, order parameter and differences between the elastic and traditional (conduction) percolation problems. It was demonstrated that the second order phase (connectivity) transition takes place only in stress (load) controlled conditions. In contrast, localization (emergence of shear bands) of the deformation occurs in the strain (displacement) controlled tests.

Initial exploration of the Preisach model were focused on ductile behavior using parallel bar models. Important conclusions were related to the thermodynamics of the process, including differences between locked-in and dissipated work.

Keywords: Metals: Ferrous, Fracture, Fatigue, Creep

192. Quantitative Non-Destructive Evaluation of High Temperature Superconducting Materials FY 1992

$ 0

DOE Contact: Oscar P. Manley, (301) 903-5822
Northwestern University Contact: J. D. Achenbach, (312) 491-5527

The work on this project is concerned with applications of the scattered field approach to the detection and characterization of cracklike flaws. The work is both analytical and numerical in nature.

The efficacy of ultrasonic methods to detect and characterize a crack depends on topographical features of the crack faces, the presence of inhomogeneities in the crack's environment, and on the mechanical properties in the near-crack region. In this work the effects on the scattered ultrasonic field of various features of fatigue and stress corrosion cracks, such as partial crack closure, the presence of microcracks and microvoids, and near-tip zones of different mechanical properties have been investigated. Most of the results have been obtained by formulating a set of singular integral equations for the fields on the boundaries of the scattering obstacles. These equations have been solved numerically by the boundary element method, and the scattered fields have subsequently been obtained by using representation integrals.

For the configurations examined in this work, crack closure has the most significant effect on far-field scattering.

Keywords: Non-Destructive Evaluation, Superconductors, Scattering

193. Development of Measurement Capabilities for the Thermophysical Properties of Energy-Related Fluids FY 1992

$551,000

DOE Contact: Oscar P. Manley, (301) 903-5822
National Institute of Standards and Technology Contact: R. Kayser, (301) 975-2483 and J. M. H. Sengers, (301) 975-2463

The major objective of this project is to develop state-of-the-art experimental apparatus that can be used to measure the thermophysical properties of a wide range of fluids and fluid mixtures important to the energy, chemical, and energy-related industries and to carry out carefully selected benchmark measurements on key systems. The research is being done jointly by two groups within the Thermophysics Division of the NIST Chemical Science and Technology Laboratory; one group is located in the Gaithersburg, MD, laboratories and the other at the Boulder laboratories. The specific measurement capabilities to be developed in this project include new apparatus for transport properties (thermal conductivity and viscosity), for thermodynamic properties (pressure-volume-temperature data and enthalpy), for phase equilibria properties (vapor-liquid equilibria, coexisting densities, and dilute solutions), and for dielectric properties (dielectric constant). These new apparatus will extend significantly the existing state of the art for properties measurements and make it possible to study a wide range of complex fluid systems (e.g., highly polar, electrically conducting, and reactive fluids) under conditions which have been previously inaccessible. This project also includes benchmark experimental measurements on systems containing alternative refrigerants, on aqueous solutions, and on carefully selected systems with species of diverse size and polarity that are important to the development of predictive models for energy-related fluids.

Keywords: Thermophysical Properties, Mixtures, Fluids, High Temperature, High Pressure

194. Flux Flow, Pinning, and Resistive Behavior in Superconducting Networks

FY 1992
$69,500

DOE Contact: Oscar P. Manley, (301) 903-5822
University of Rochester Contact: S. Teitel, (716) 275-4039

The motion of vortex structures, in response to applied currents, is a major source of resistance in superconducting networks in magnetic fields. Systems of interest include regular Josephson junction arrays and type II superconductors, such as the new granular high T_c ceramics. Numerical simulations of finite temperature, current carrying networks will be carried out to provide a characterization of vortex response in non-equilibrium situations. For periodic networks, current-voltage (I-V) characteristics will be computed and compared with experimental results. The effects on resistivity of transitions from pinned to unpinned or to melted vortex structures, will be investigated. For disordered networks, the effects of pinning in producing metastable vortex structures leading to glassy behavior will be explored.

To date, simulations have been carried out for the "fully frustrated" two dimensional regular Josephson junction array. I-V characteristics were computed and reasonable agreement found with experiment. Behavior was explained within a simple physical model, in which correlations between vortices is crucial for producing the critical excitations leading to vortex flow resistance.

Keywords: Flux Flow, Pinning, Vortex Motion, Superconductors

195. An Investigation of the Effects of History Dependent Damage in Time Dependent Fracture Mechanics

FY 1992
$94,027

DOE Contact: Oscar P. Manley, (301) 903-5822
Battelle Memorial Institute Contact: F. Brust, (614) 424-5034

The demands for structural systems to perform reliably under severe operating conditions continue to increase. Modern energy production facilities experience degradation and damage because they operate in severe high-temperature environment where time dependent straining and damage may lead to structural failures. The goal of this research is to study the high temperature damage and failure processes and to further develop a method for predicting this behavior in an effort to increase structural life. In particular, we focus on time dependent damage which occurs under history-dependent loading conditions, i.e., transient conditions.

The types of time dependent (creep) damage considered in this program include: sustained load creep, variable load creep, and variable load creep with thermal gradients. During the first year of this study, the implications of using Norton's creep law on various integral parameters used to characterize crack tip phenomena were evaluated as a function of time. Other constitutive laws for time dependent materials such as those of Murakami and Ohno are being implemented into the finite element code. In addition, constitutive property data and high temperature creep crack growth data are being obtained on stainless steel. These experiments will be used to verify analytical predictions and characterize time and history dependent damage during crack nucleation and growth.

The results from this work will be used by practicing engineers to enhance the life of high temperature structural systems during the design phase.

Keywords: Fracture Mechanics, History Dependent Damage, High Temperature

196. Mixing and Settling in Continuous Metal Production

FY 1992
$135,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Dartmouth College Contact: H. Richter, (603) 646-2707

For reasons of both energy conservation and environmental protection, tonnage oxygen has become an essential component in many ferrous and nonferrous smelting and refining operations. Innovative reactors such as the QSL converter for direct lead bullion production are also suitable for direct coppermaking and a modification of the reactor is also very attractive for steelmaking.

The research objectives of the work are to learn about the behavior of gas and particulate matter in turbulent liquids, the nature and paths of liquids and particulate entrainment into the plumes, and separation phenomena including travel to and behavior in the settling zones. Such knowledge is of fundamental value in designing reactors for continuous, direct metalmaking.

The new information will predict bath mixing, heat and mass transfer and settling parameters under the variety of operating conditions which will prevail in continuous, direct metalmaking oxygen reactors of the future. Therefore, it is proposed to study gas injection through submerged injectors, and also particulate addition from above, into a liquid bath consisting of two immiscible layers of liquids having roughly the same viscosities as slag and matte or metal and with roughly comparable density ratios.

Keywords: Bath Mixing, Heat and Mass Transfer, Settling

197. Application of Magnetomechanical Hysteresis Modelling to Magnetic Techniques for Monitoring Neutron Embrittlement and Biaxial Stress — FY 1992 $99,912

DOE Contact: Oscar P. Manley, (301) 903-5822
Southwest Research Institute Contact: M. Sablik, (512) 522-3342

The project objective is to study the effects of neutron embrittlement and biaxial stress on signals from various magnetic measurement techniques in steels. It is expected that interaction between experiment and modeling will lead to design of efficient magnetic measurement procedures for monitoring neutron embrittlement and biaxial stress. Project research is important for safety monitoring in the nuclear power and gas industries.

Magnetic measurement techniques to be assessed are: (1) magnetic hysteresis loop measurement of properties like coercivity and permeability; (2) magabsorption, which measures the impedance of an rf coil brought close to a magnetic sample; (3) Barkhausen noise analysis; (4) magnetically induced velocity change (MIVC) of an ultrasonic wave; and (5) harmonic analysis of an ac magnetic hysteresis loon. The model of Sablik *et al.* for magnetic hysteresis and uniaxial stress effects on magnetic properties will be extended to conditions of biaxial stress and neutron embrittlement. The effects of these conditions on magnetic probe signals (1)-(5) will be modeled and compared to experiment. In the case of neutron embrittlement, measurements will be made on steel samples characterized by Charpy tests after previous exposure to various neutron fluences.

Keywords: Magnetic NDE Techniques, Neutron Embrittlement, Biaxial Stress

198. Stability and Stress Analysis of Surface Morphology of Elastic and Piezoelectric Materials

FY 1992
$98,695

DOE Contact: Oscar P. Manley, (301) 903-5822
Stanford University Contact: H. Gao, (415) 725-2560

The goal of this research is to investigate the mechanical effects of surface morphology of elastic dielectric and piezoelectric materials. In particular, the project will study the stability of a flat surface against diffusional perturbations and the stress concentration caused by slightly undulating surfaces.

The surface morphology of materials will be studied by using a unified perturbation procedure based on the notion of thermodynamic forces and the energy momentum tensor. The thermodynamic forces on material inhomogeneities such as interfaces and inclusions are a measure of the rate at which the total energy of a physical system varies with the configurational change of these inhomogeneities. Within the general methodology, any type of material and loading condition can be studied as long as the proper forces can be identified. By using corresponding material conservation laws discovered previously, a systematic analysis of surfaces of piezoelectric solids will be made. Preliminary studies have shown that under sufficiently large stresses, surfaces of materials become unstable against a range of diffusional perturbations bounded by two critical wave lengths. Even a slight undulation caused by these unstable diffusional perturbations, such as micro-level bumps and troughs, can result in a significant stress concentration along a material surface. These concentrations may lead to mechanical failures along the surface and may have more consequences for piezoelectric materials where the deformation is coupled to an applied electric field. There are also suggestions that the stress distributions in a body may be sensitive to the surface morphology.

Keywords: Stress Analysis, Surface Morphology, Elastic, Dielectric, Piezoelectric Materials

199. Micromechanical Viscoplastic Stress-Strain Model with Grain Boundary Sliding

FY 1992
$48,500

DOE Contact: Oscar P. Manley, (301) 903-5822
University of Connecticut Contact: E. H. Jordan, (203) 486-2371

The first part of this project has focused on developing and experimentally verifying methods of predicting the deformation response of polycrystalline metals from models of single crystal deformation, based on crystallographic slip. In the ongoing research, the goal is to try to predict the degree of heterogeneity of deformation and verify these predictions experimentally. The existing self-consistent model is to be completed by a second model based on periodicity which is expected to be both more realistic and more computationally burdensome. The degree of heterogeneity of deformation will be studied by the different experimental techniques. Neutron diffraction experiments are planned in which diffraction from a few grains at a time is studied to determine lattice strains in individual grains. Many grains will be surveyed to get statistical measure of heterogeneity of grains including no surface grains. The Moire strain analysis will also be done on large grained samples. The material studied is the same ones used in the first phase, so that all the single crystal mechanical properties are accurately known. The data collected will prove a unique complete set of data to test the ability of the models in this program and other models with respect to their ability to predict the degree of heterogeneity of deformation. comparison of more data and the neutron defraction data will also provide insight into the difference between surface grain behavior and interior grain behavior. Developing models that realistically predict grain to grain heterogeneity and verifying those models is a basic element in modeling mechanical behavior. Heterogeneity is particularly important to fatigue in which the most unfavorably oriented grain is the site of failure.

Keywords: Micromechanical, Viscoplasticity, Grain Boundary, Crystallographic Slip, High Temperature, Experiments

200. A Micromechanical Viscoplastic Stress-Strain Model with Grain Boundary Sliding — FY 1992 $48,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Engineering Science Software, Inc., Contact: K. P. Walker, (401) 231-3182

This project is joint with the University of Connecticut project described above. See the previous paragraph for a description.

Keywords: Micromechanical, Viscoplasticity, Grain Boundary, Crystallographic Slip, High Temperature, Constitutive Model

201. Modeling of Thermal Plasma Processes — FY 1992 $232,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Idaho National Engineering Laboratory Contacts: J. D. Ramshaw, (208) 526-9240 and C. H. Chang, (208) 526-2886

Optimization of thermal plasma processing techniques requires a better understanding of the space- and time-resolved flow and temperature distributions in the plasma plume and of the interaction between the plasma and a particulate phase. This research is directed toward the development of a comprehensive computational model of thermal plasma processes and plasma-particle interactions capable of providing such information. The model is embodied in the LAVA computer code for two- or three-dimensional transient or steady state thermal plasma simulations. LAVA uses a rectangular mesh with an excluded volume function to represent geometrical obstructions and volume displaced by particles. Simple highly vectorizable numerics are utilized, with rapid steady state and low-speed flow options. The plasma is represented as a multicomponent fluid governed by the transient compressible Navier-Stokes equations. Real gas physics is allowed for by temperature-dependent specific heats and transport properties. Multicomponent diffusion is calculated in a self-consistent effective binary diffusion approximation, including ambipolar diffusion of charged species. Both k-epsilon and subgrid-scale turbulence models are included. Dissociation, ionization, and plasma chemistry are represented by means of general kinetic and equilibrium chemistry routines. Discrete particles interacting with the plasma will be represented by a stochastic particle model similar to that previously used to model liquid sprays. This model allows for spectra of particle sizes, shapes, temperatures, etc., thereby capturing the important statistical aspects of the problem. It will include sub-models for the various plasma-particle and particle-particle interaction processes, including melting, evaporation, condensation, nucleation, agglomeration, and coalescence.

Keywords: Plasma Processing, Optimization, Computational Model

202. Nondestructive Evaluation of Superconductors FY 1992
$200,000

DOE Contact: Oscar P. Manley, (301) 903-5822
Idaho National Engineering Laboratory Contact: K. L. Telschow, (208) 526-1264

The purpose of this task is to perform fundamental research which will lead to the development and application of new nondestructive evaluation (NDE) techniques and devices for the characterization of high-temperature superconducting materials. In the near future, application of these new superconductors will require NDE methods for evaluating the properties of wires, tapes and coatings. Microstructural and, particularly, superconducting properties must be measured noninvasively in a manner capable of providing spatial information so that fabrication processes can be optimized. Although the fabrication of these ceramic materials is being pursued by many different techniques at present, there is enough similarity in the different superconducting materials and the fabricated forms to begin research into NDE measurement techniques. In FY89 this project began identifying techniques that can determine critical superconducting properties on a local scale. This has resulted in the use of AC induced currents in conjunction with DC transport currents to determine critical currents and dissipation locally. The analysis of these measurements is being carried out with the aid of the London and "Critical State" models for supercurrent flow in these materials. These results are being correlated with material microstructure information and other measurement techniques.

Keywords: NDE, Superconductors

203. Momentum and Heat Transfer Processes in Viscoelastic Fluids FY 1992 $73,391

DOE Contact: Oscar P. Manley, (301) 903-5822
University of Illinois at Chicago Contact: J. P. Hartnett, (312) 966-4490

The goal of the research is to study the fluid mechanical and heat transfer behavior of viscoelastic aqueous polymer solutions. The ultimate objective is to provide a basis for predicting the performance of such fluids.

Recent studies of the heat transfer performance of viscoelastic aqueous polymer solutions have been directed to the pool boiling behavior of such solutions. The heat transfer coefficients of aqueous hydroxyethy cellulose (Natrosol) solution boiling on horizontal wires were found to be considerably higher than the values found for water alone, whereas the pool boiling performance of other polymer solutions (including polyacrylamide solutions) was inferior to that of water. A distinguishing characteristic of the Natrosol solutions is their lower surface tension values. Accordingly an investigation of the influence of two different surfactants (i.e., agents which reduce surface tension) on the pool boiling behavior of aqueous polyacrylide solutions was carried out. When the surfactant polyoxyethylene sorbitan monoleate (Tween 80) was added to the aqueous polyacrylamide solution the surface tension decreased by 10 to 20 percent but the boiling performance was not affected. In contrast, the presence of sodium lauryl sulfate (SLS) resulted in a similar decrease in surface tension but a significant increase in the boiling heat transfer coefficient was recorded relative to the pool boiling performance of deionized water. On the basis of these experimental results it can be concluded that lowering the surface tension of an aqueous polymer solution does not guarantee enhancement of the boiling heat transfer performance.

Keywords: Heat Transfer, Aqueous Polymer Solutions

204. Pulse Propagation in Inhomogeneous Optical Waveguides FY 1992 $83,537

DOE Contact: Oscar P. Manley, (301) 903-5822
University of Maryland Contact: C. Menyuk, (301) 455-3501

Our research, which was originally focused on light propagation in inhomogeneous optical fibers, has broadened in scope to include studies of solid state rib waveguides and Y-junctions which are used to guide and switch light. The work on optical fibers is divided into two research projects.

The first project concerns long-distance communication using solutions. We have been particular concerned with the effects of randomly varying birefringence, and we have shown that its effect is benign. From the basic equations we were able to show from an appropriate ordering expansion that the nonlinear Schrodinger equation is the lowest order equation and, hence, we expect its behavior to dominate the soliton evolution even in a highly birefringent fiber, as long as the birefringence is rapidly varying. We have also studied optical fiber soliton switches based on trapping and dragging. To do the work on optical fibers, we have collaborated with scientists at AT&T Bell Laboratories. The first solid state project was to find the effect of a quantum well on the propagation characteristics of a rib waveguide. Using a planar guide as reference, we were able to show that the effect of the real geometry is qualitatively small but can have a significant quantitative effect.

The second solid-state project is to determine the effect of dry-etching on the mode-holding characteristics of the device. As a consequence of the etching, the height at which the junction splits can vary. We showed that the rounding has a very small effect, in contrast to blunting which occurs when the materials are chemically wet etched.

Keywords: Optical Fibers, Pulse Propagation, Inhomogeneities, Imperfections

205. Low Resistivity Ohmic Contacts Between Semiconductors and High-T_c Superconductors

FY 1992
$98,488

DOE Contact: Oscar P. Manley, (301) 903-5822
National Institute of Standards and Technology Contacts: J. Moreland, (303) 497-3641 and J. W. Ekin, (303) 497-5448

The purpose of this project is to fabricate and characterize high-T_c superconductor/semiconductor contacts. Developing a method for optimizing the current capacity of such contact will extend the application of high-T_c superconductors to hybrid superconductor/semiconductor technologies. These technologies include integrated circuit interconnects (both on-chip and package) and proximity superconductor/semiconductor/superconductor SNS Josephson junctions. Presently, these are among the most promising high-T_c superconductor applications, but an essential first step is the development of reliable, stable, ohmic contacts between semiconductors and the high-T_c oxide superconductors.

The initial phase of this program is to determine the compatibility of various metals and alloys (Au and Al alloys and W, for example) as contact materials for superconducting YBCO and other high T_c materials. Once a good combination has been established, patterned YBCO/normal metal contacts will be deposited onto semiconductor wafer surfaces. We have purchased a sputter co-deposition system for YBCO thin films and have adapted three other vacuum systems for contact deposition including two sputtering systems and an evaporator.

Keywords: High-Tc Superconductors, Semiconductors, Contact, Low Resistivity

206. <u>The Evolution of a Hele-Shaw Interface and Related Problems in Dendritic Crystal Growth</u> <u>FY 1992</u>
$66,116

DOE Contact: Oscar P. Manley, (301) 903-5822
Ohio State University Contact: S. Tanveer, (614) 292-4972

A cell consisting of two parallel plates separated by a thin layer of liquid, the so-called Hele-Shaw cell, serves as a model of a porous medium. For example, one can readily observe the displacement of a more viscous fluid by a less viscous one, such as is taken advantage of in secondary oil recovery methods.

Most mathematical models of the displacement process studied to date have dealt with steady states and their stability. Under those conditions solutions can be obtained even if the surface tension at the interface between the two fluids is ignored. As to the initial value problem, it has been found that ignoring surface tension leads to an ill-posed problem in the sense that nonphysical cusps form at the interface in a finite time. Experimentally it is found that when the surface tension is small no steady state is reached and the interface continues to deform into a finer and finer fractal-like structure.

Recent work by the proposer has revealed that it is possible to imbed the ill-posed problem into a well-posed one so as to clarify what happens when the surface tension tends to zero. For the proposed research detailed calculations will be carried out to examine how the singularities in the model equations are related to the evolution of the shape of the interface. Second, the results obtained for the Hele-Shaw cell will be extended to study the time evolution of the surface of a growing crystal with dendrites. Third, statistics of the observed patterns will be related to the statistical distribution of singularities in the model equations. Next, more general boundary conditions will be considered to conform to a broader class of physically realistic situations. Finally, some intrinsically nonlinear aspects of dendritic growth will be examined.

Keywords: Crystal Growth, Dendrites

207. <u>Degenerate Four-Wave Mixing as a Diagnostic of Plasma Chemistry</u> <u>FY 1992</u>
$95,328

DOE Contact: Oscar P. Manley, (301) 903-5822
Stanford University Contact: R. Zare, (415) 723-3062

A need exists for *in situ* nonintrusive diagnostics for probing trace and highly reactive radical intermediates in nonequilibrium plasma used for chemical vapor deposition. We propose applying a novel nonlinear spectroscopic technique, degenerate four-wave mixing (DFWM). The DFWM signal is a coherent scattered beam at frequency which is generated by the nonlinear response of the medium to the interaction of three incident waves at the same frequency. The signal is enhanced by a resonant transition and offers a form of Doppler-free spectroscopy with extremely high spectral, spatial, and temporal resolution. Signal detection is remote and does not suffer from background interference from the bright plasma source. In addition, the phase conjugate nature of the signal eliminates optical aberration. The environment we propose to study is an atmospheric-pressure rf-inducively-coupled plasma and the target radicals include CH, CH_2, C_2, C_2H, and CH_3 that are important in plasma synthesis of diamond thin films. The spatial sensitivity of DFWM will be used to study the coupling of gas-phase and gas-surface chemistry by measuring temperature and concentration profiles. The proposed research will advance diagnostic techniques for plasma environments and provide a better understanding of the plasma chemistry of diamond synthesis.

Keywords: Plasma, Four-Wave Mixing

208. <u>Effect of Forced and Natural Convection on Solidification of Binary Mixtures</u>

FY 1992
$93,290

DOE Contact: Oscar P. Manley, (301) 903-5822
Purdue University Contact: F. Incropera

This study deals with the influence of combined convection mechanisms on the solidification of binary systems. A major accomplishment of research performed to date has been the development and numerical solution of a continuum model, which uses a single set of equations to predict transport phenomena in the liquid, "mushy" (two-phase), and solid regions of the mixture. Calculations have been performed for two-dimensional, aqueous salt solutions involving forced convection, thermo/solutal natural convection, and/or thermo/diffusocapillary convection. The calculations have revealed a wide variety or rich and robust flow conditions, including important physical features of the solidification process which have been observed experimentally but have heretofore eluded prediction. These features include double-diffusive layering in the melt, development of an irregular liquidus front, remelting of solid, development of flow channels in the mushy region, and the establishment of characteristic macrosegregation patterns (regions of significantly different composition) in the final solid.

The primary objective of current studies is to determine the manner in which externally imposed forces influence thermo-solutal convection in the mushy and liquid regions during solidification of a binary mixture. A special goal is to determine means by which the forces may be used to offset or dampen thermo/solutal convection, thereby reducing macrosegregation and attendant casting defects. Separate consideration is being given to the effects of magnetic and centrifugal forces on solidification in binary metallic alloys and aqueous salt solutions, respectively. Predictions based on the continuum model are being compared with measurements obtained for metallic (Pb-Sn) and aqueous (NH_4 Cl-H_2O) systems.

Keywords: Solidification, Convection, Binary Alloys, Salt Solutions, Magnetic Fields, Centrifugal Forces

Geosciences Research

Materials Preparation, Synthesis, Deposition, Growth or Forming

209. Interactions between Mineral Surfaces and Ions in Solution — FY 1992 $100,000

DOE Contact: W. C. Luth, (301) 903-5822
LBL Contact: D. L. Perry, (510) 486-4819

The research involves fundamental studies to determine the basic surface chemistry of common minerals (both synthetic and natural) and the chemical reactions of metal ions with the mineral surfaces. It encompasses (1) basic spectroscopy of natural minerals and their synthetic counterparts, (2) spectroscopic studies of metal ions that have been adsorbed onto the mineral surfaces, (3) syntheses and spectroscopy of model compounds that form in metal ion-mineral reactions, and (4) spectroscopy of organic compounds that have been chemisorbed on synthetic and natural minerals. X-ray photoelectron and Auger spectroscopic techniques have been used to study the surfaces and their reactions with metal ions in solution.

Keywords: Surface Chemistry, Minerals, Photoelectron Spectroscopy, Auger Spectroscopy

210. Mineral Hydrolysis Kinetics — FY 1992 $124,000

DOE Contact: W. C. Luth, (301) 903-5822
SNL Contact: H. R. Westrich (505) 844-9092

The research involves measuring dissolution kinetics of orthosilicate minerals and incorporating these data in molecular dynamics computer simulations of the solid-liquid interface. A suite of orthosilicate minerals has been assembled, either at one atmosphere or obtained commercially as natural minerals for dissolution measurements. This suite includes both alkaline-earth (Be, Mg, & Ca) and transition metal end members (Mn, Fe, Co, & Ni). Both pH-stat and batch techniques have been used for dissolution measurements after extensive sample preparation and characterization. Measurements suggest that dissolution rates of compositionally distinct orthosilicate minerals scale similarly to rates of water exchange around the corresponding dissolved, divalent cation.

Keywords: Dissolution Kinetics, Orthosilicate Minerals

211. Transition Metal Catalysis in the Generation of Natural Gas FY 1992
$127,000

DOE Contact: W. C. Luth, (301) 903-5822
Rice University Contact: F. Mango, (713) 527-4880

Light hydrocarbons in petroleum (C_5-C_9) and natural gas (C_1-C_4) are conventionally viewed as thermolytic products formed in the Earth from the progressive thermal breakdown of kerogen and oil. Alternatively, transition metals, activated under the reducing conditions of diagenesis, can be proposed as catalysts in the generation of light hydrocarbons. Transition metals (Ni, V, Ti, Co, Fe), in kerogenous sedimentary rocks, petroporphyrins, and as pure compounds, will be reduced under diagenic conditions and analyzed for catalytic activity. If the hypothesis is correct, kerogenous transition metals should become catalytically active under the reducing conditions of diagenesis and catalyze the conversion of paraffins into the light hydrocarbons seen in petroleum.

Keywords: Transition Metals, Catalytic Activity, Petroleum

Materials Structure and Composition

212. Three-Dimensional Imaging of Drill Core Samples Using Synchrotron Computed Microtomography FY 1992
$75,000

DOE Contact: W. C. Luth, (301) 903-5822
BNL Contact: K. W. Jones, (516) 282-4588
State University of New York, Stony Brook Contact: W. Lindquist, (516) 632-8361

The synchrotron makes feasible use of high resolution computed microtomography (CMT) for non-destructive measurements of the structure of different types of drill core samples. The work is carried out using bending magnet radiation from the National Synchrotron Light Source for measurements below X-ray energies of 40-50 keV. A superconducting wiggler source is used for large samples that require higher X-ray energies. The spatial resolution of the measurement can be adjusted to fit the objectives of a specific experiment. The best resolution obtained to date is about 2 μm. Measurements have been made on a number of large rocks which clearly show the inhomogeneities, pores, and cracks present. It has been shown that the system can be used to produce images of a crack structure in three dimensions. Analysis of the data is done using the distribution of measured attenuation coefficients and 2-point correlation structure to determine porosity and other information relevant to the rock structure.

Keywords: Microtomography, Non-Destructive Evaluation, Pore Structure, Crack Structure, Drill Cores, Rocks

213. Grain Boundary Transport and Related Processes in Natural Fine-Grained Aggregates

FY 1992
$141,000

DOE Contact: W. C. Luth, (301) 903-5822
Brown University Contact: R. A. Yund (401) 863-1931

The objective of this study is direct measure of diffusional transport rates in rocks and how the rates vary with mineralogy and microstructure, as well as temperature and pressure. Techniques for experimentally determining diffusional transport rates in fine-grained, monomineralic aggregates using the ion microprobe (SIMS), have been developed and used to study natural quartz aggregates. In addition, we have demonstrated the important effect of fluid composition on the equilibrium microstructure, and on the nature and rate of diffusional transport, in quartz aggregates. The effective diffusion rate (bulk diffusivity) of oxygen in the aggregates is four to five orders of magnitude greater in samples with an interconnected fluid due to transport by ionic diffusion through the static fluid which is much faster than true grain boundary diffusion.

Keywords: Diffusion, Rocks, Quartz, Microstructure

214. IR Spectroscopy and Hydrogen Isotope Geochemistry of Hydrous Silicate Glasses

FY 1992
$90,000

DOE Contact: W. C. Luth, (301) 903-5822
California Institute of Technology Contact: E. Stolper, (818) 356-6504

The focus of this project is the combined application of infrared spectroscopy and stable isotope geochemistry to the study of hydrogen- and oxygen-bearing species dissolved in silicate melts and glasses. The partitioning of hydrogen isotopes (D and H) between water vapor and rhyolitic silicate melt and glass was measured at temperatures of 550-850°C and pressures up to 2000 bars. The solubility of water in rhyolitic melts and the speciation of water (i.e., the proportions of dissolved water molecules and hydroxyl groups) in such melts have also been measured, with careful attention to the temperature dependence of speciation and the kinetics of interconversions between species. The uniqueness of this approach stems from the ability to measure the concentration profiles of separate species, which allows the problem to be treated in terms of distinct diffusing, reacting species. These results have been used as a basis for understanding "self diffusion" of oxygen in silicate minerals and glasses.

Keywords: Infrared Spectroscopy, Geochemistry, Silicate Minerals, Glasses, Silicate Liquids, Diffusion, Water

215. Crystal Chemistry of Hydroxyl and Water in Silicate Minerals

FY 1992
$93,000

DOE Contact: W. C. Luth, (301) 903-5822
University of Colorado Contact: J. R. Smyth, (303) 492-5521

Hydrogen in the form of hydroxyl and molecular water plays an important role in the chemical and physical properties of silicate minerals. D/H and $^{16/18}O$ ratios used as geothermometers to evaluate geothermal and petroleum reservoirs are controlled by hydroxyl stability; the stability of hydroxyl-bearing silicates (such as omphacite, wadsleyite (β-Mg_2SiO_4), rutile and stishovite) may control melting and magma generation; the hydration of various cations substituting into the abundant natural zeolite and clinoptilolite may control sorption and migration of cations and radionuclides. This project investigates the crystal chemistry of OH and H_2O substitution in silicate minerals by use of X-ray and neutron diffraction methods combined with IR spectroscopy and interprets and generalizes the results using an electrostatic model for these mineral structures.

Keywords: Silicate Minerals, Hydroxyl, Water, Geotemperature, Isotope Ratios, Crystal Chemistry, Infrared Spectroscopy

216. Investigation of Ultrasonic Wave Interactions with Fluid-Saturated Porous Rocks

FY 1992
$59,000

DOE Contact: W. C. Luth, (301) 903-5822
Ohio State University Contact: L. A. Adler

The research involves ultrasonic wave propagation in fluid-filled porous materials. A novel technique based on direct generation of surface waves by edge excitation was employed on both synthetic and natural rocks. Low-frequency (100-500kHz) shear transducers were used in pitch-catch mode to launch and receive ultrasonic surface waves. Reflecting the importance of surface tension, practically closed pore boundary conditions can prevail on the free surface of a water-saturated rock for completely open pores.

Keywords: Porous Materials, Rocks, Surface Waves

217. Zircons and Fluids: An Experimental Investigation with Applications for Radioactive Waste Storage — FY 1992 $63,000

DOE Contact: W. C. Luth, (301) 903-5822
Virginia Polytechnic Institute Contact: A.K. Sinha, (703) 231-5580

Current research is aimed at providing a comprehensive physical and chemical data base for $ZrSiO_4$ (zircon) that integrates measurements of solubility, alpha damage and development of fractures induced due to both radiation and temperature enhanced stresses. New analytical data that bear on the chemical stability of $ZrSiO_4$ in varying fluid compositions. Solubility studies include both batch and mixed-flow reactor experiments. The calculated value of dissolution activation energy (ΔE_a) of ~30 K cal suggests surface detachment reactions as the rate limiting process. Current research efforts also include development of techniques for measurement of Zr by isotope dilution methods to permit measurement of Zr concentrations in solutions at ppb levels. The continued integration of physical and chemical data will provide a comprehensive model for the stability of $ZrSiO_4$ as a host for radioactive waste.

Keywords: Zircon, Irradiation Damage, Solubility

Materials Properties, Behavior, Characterization or Testing

218. Modification of Fracture Transport Properties of Rocks by Mechanical and Chemical Processes — FY 1992 $180,000

DOE Contact: W. C. Luth, (301) 903-5822
LLNL Contact: W. B. Durham (510) 422-7046

It is widely accepted that the movement of fluid through the crust is controlled not by the bulk permeability of the medium but by the network of joints (fractures) that pervade the medium. The goal of this project is to understand the physics of fluid flow in individual fractures to better constrain models of flow in fracture networks. Topography of the individual surfaces on a coarse as well as microscopic scale are characterized using a profilometer having extremely high positional accuracy in the plane of the fracture. Fracture aperture and fracture permeability are measured as a function of confining pressure (as high as 160 MPa). Other relevant parameters: fractures slightly offset from their mated positions, the effects of various kinds of wear (e.g., sandblasting, abrasion) and the effect of grain size by using different rock types can also be measured.

Keywords: Fluid Flow, Fractures

219. Oxygen and Cation Diffusion in Oxide Minerals FY 1992
$180,000

DOE Contact: W. C. Luth, (301) 903-5822
LLNL Contact: F. J. Ryerson, (510)422-6170
UCLA Contact: K. D. McKeegan, (310) 825-3580

This project concerns the experimental determination of diffusion coefficients, under a variety of environmental conditions, for minerals of importance to contemporary problems. The results of these investigations support efforts to constrain the physical properties of minerals as a function of chemical environment, such as the variation in plastic deformation rate as a function of the oxygen fugacity (f_{O2}), by determining relationships between point defect chemistry and diffusive transport. Work is proceeding in the following areas: (1) the role of f_{O2} and silica activity on the diffusion of oxygen in olivine; (2) the role of f_{O2} and silica activity on the diffusion of silicon in olivine; (3) determination of activation volumes for oxygen, silicon, and magnesium diffusion in olivine; and (4) oxygen diffusion in diopside, spinel, hibonite, and melilite solid solutions.

Keywords: Diffusion, Minerals, Plastic Deformation

220. Acoustic Emissions and Damage in Geomaterials FY 1992
$150,000

DOE Contact: W. C. Luth, (301) 903-5822
SNL Contact: D. J. Holcomb, (505) 844-2157

A fundamental parameter in many geophysical and geotechnical problems is the state of stress in the rock mass of interest. The standard techniques for determining in situ stress have limitations such as cost, limited depth range or reliance on unproven models. A method that promises to circumvent many of these difficulties has been developed as a result of research on damage and acoustic emissions in rock. Using a phenomenon known as the Kaiser effect, which acts as a marker indicating the stress a sample has experienced, the theory shows how to use core samples to determine the complete stress tensor. The advantages of the method are: (1) no expensive down-hole testing is required, (2) depth limitations are removed, (3) no model of the material properties is required, and (4) the complete stress tensor is determined, both magnitude and orientation.

Keywords: Rocks, Stress Analysis, Acoustic Emission, Theory

221. Transport Properties of Fractures

FY 1992
$129,000

DOE Contact: W. C. Luth, (301) 903-5822
SNL Contact: S. R. Brown, (505) 844-0774

Fluid flow in fractured rocks is of primary importance to oil recovery; this research focuses on the physics of transport of fluid and electric current through fractures in rock and the scale dependence of related properties. One inch diameter core samples from 23 natural rock joints have been collected for a study of the roughness and degree of mismatch of fracture surfaces. The resulting data have led to the development of a simple mathematical model of a rough-walled fracture, which contains only three free parameters. This model can be used to generate realistic computer models of rough fractures for use in the study of various physical properties including fluid permeability, elastic stiffnesses, and the frictional shear strength.

Keywords: Fluid Flow, Fracture in Rocks, Electrical Conductivity

222. Cation Diffusion Rates in Selected Silicate Minerals

FY 1992
$187,000

DOE Contact: W. C. Luth, (301) 903-5822
SNL Contact: R. T. Cygan, (505) 844-7354

The occurrence of disequilibrium behavior in geological is often attributed to the limited diffusion of chemical species through a silicate mineral. Evaluation and modeling of geochemical processes related to nuclear waste, energy, and materials problems will require the accurate determination of cation diffusion data in a variety of silicate minerals. In an effort to evaluate the relatively slow diffusion of Mg^{2+}, Mn^{2+}, and Ca^{2+} in phases such as garnet (where diffusion rates are on the order of 10-21 to 10-18 m^2/sec in the temperature range of 700R to 1000Rc) we have developed a new technique for the preparation of diffusion couples using thin film deposition. Depth profiles obtained using a Cameca 4f ion microprobe are reproducible and clearly define the oxide-garnet interface. Diffusional penetration depths for Mg2+ are on the order of 0.3 microns.

Keywords: Diffusion, Silicate Minerals, Ion Microprobe

223. Isotopic Tracer Studies of Diffusion in Silicates and Geological Transport Processes Using Actinide Elements

FY 1992
$345,000

DOE Contact: W. C. Luth, (301) 903-5822
California Institute of Technology Contact: G. J. Wasserburg, (818) 356-6439

We have measured Mg self-diffusion in spinel and coexisting melt, at bulk chemical equilibrium, using an isotopic tracer. The diffusion coefficients were calculated from the measured isotope profiles using a model that includes the complementary diffusion of ^{24}Mg, ^{25}Mg, and ^{26}Mg in both phases with the constraint that the Mg content of each phase is constant. The activation energy and pre-exponential factor of Mg in spinel are, respectively, 384 ± 7 kJ and 74.6 ± 1.1 cm^2/s. Diffusion coefficients for coexisting elements can be obtained in a single experiment. The measurement of slow diffusion rates can be determined from a coupled experiment with a phase where diffusion is faster. Diffusion coefficients of the order of 10^{-14} cm^2/s are now readily measurable. We have pursued resonance ionization mass spectrometry (RIMS) for elements which are hard to ionize by conventional techniques or whose analysis is seriously compromised by isobaric interferences.

Keywords: Diffusion, Spinel, Oxide Melts, Mass Spectrometry

224. Experimental Measurement of Thermal Conductivity in Silicate Liquids

FY 1992
$109,000

DOE Contact: W. C. Luth, (301) 903-5822
University of California, Berkeley, Contact: I.S.E. Carmichael, (510) 642-2577

The transient hot wire technique has been adapted for high temperature measurements of thermal conductivity in silicate liquids. The technique is based on the transient change in temperature before attainment of steady state. The apparatus is designed such that, over the time scale of the experiment, the cell wall plays no role during transient heading and hence the fluid can be treated as infinite.

Keywords: Thermal Conductivity, Silicate Melts

225. New Method for Determining Thermodynamic Properties of Carbonate Solid-Solution Minerals

FY 1992
$83,000

DOE Contact: W. C. Luth, (301) 903-5822
University of California, Davis, Contact: P. A. Rock, (916) 752-0940

Incorporation of metals into calcium carbonate minerals is an important pathway for elimination of potentially toxic metals from natural waters. The thermodynamic properties of the resulting solid solutions are, however, poorly known because of difficulties with the solubility measurements. This project uses a new method of measuring these properties which avoids some of these difficulties. The new method involves an electrochemical double cell including carbonate minerals and no liquid junctions. This cell is an advance over conventional solubility measurements because: (1) reversibility can be determined quickly, (2) the cell has a higher sensitivity and precision than other solubility determination methods; and (3) it is not sensitive to metal-carbonato speciation.

Keywords: Carbonate Minerals, Solubility, Electrochemical Cell

226. Development of Synchrotron X-Ray Microspectroscopic Techniques and Applications to Low Temperature Geochemistry

FY 1992
$105,000

DOE Contact: W. C. Luth, (301) 903-5822
University of Chicago Contact: J. V. Smith, (312) 702-8110

This proposal defines a research program to develop synchrotron X-ray microprobe techniques and instrumentation for X-ray fluorescence (XRF) and X-ray absorption spectroscopy (XAS) applications for low temperature geochemistry. Pilot studies are being carried out at beamlines X26-A & X-17 of the National Synchrotron Light Source at Brookhaven National Laboratory. Geochemical problems are being tackled currently are the chemical nature of hydrothermal fluid inclusions (resolution about 10 micrometers with aim of 1 micrometer at APS: sensitivity for trace elements varies but can approach the 10 part-per-million level); cation fixation mechanisms in phyllosilicates (redox state can be determined by XANES for certain transition elements at the 0.0n wt. % level, and the local atomic bonding by EXAFS at the n to 0.n wt. % level). The ultimate aims are to push the capabilities as close to the micrometer/micromolar level as possible at APS.

Keywords: X-ray, Microprobe, X-ray Absorption Spectroscopy, Geochemistry

227. High Resolution TEM/AEM and SEM Studies of Fluid Rock Interactions Using Cu, Ag, Se, Cr, and Cd-Bearing Solutions and Phyllosilicates

FY 1992
$134,000

DOE Contact: W. C. Luth, (301) 903-5822
Johns Hopkins University Contact: D. R. Veblen, (410) 516-8487

Oxidation-reduction reactions at the mineral-fluid interface can be responsible for attenuation or mobilization of multivalent cations in the near-surface environment. This research involves the investigation of the interaction of silver-, copper-, selenium-, and chromium-bearing solutions with ferrous phyllosilicates such as biotite, a potential reducing agent, under both oxic and anoxic conditions. The experimental work involves examination of the surfaces and interior portions of the reacted phyllosilicates with TEM, AEM, SEM, X-ray photoelectron spectroscopy.

Keywords: Oxidation-Reduction Reactions, Cations, Silicate Minerals

228. Thermodynamics of Minerals Stable Near the Earth's Surface — FY 1992 $130,000

DOE Contact: W. C. Luth, (301) 903-5822
Princeton University Contact: A. Navrotsky, (609) 258-4674

The purpose of this work is to expand our data base and understanding of the thermochemistry of minerals and related materials through a program of high temperature reaction calorimetric studies. The technique of oxide melt solution calorimetry (in molten $2PbO \cdot B_2O_3$) has been extended to volatile-bearing phases. Calorimetric conditions under which H_2O and CO_2 reach a well-defined final state upon reaction with molten lead borate have been perfected. These involve calorimetry under a flowing gas atmosphere, which purges all H_2O and CO_2 in the gas phase, leaving essentially none to interact with the solvent (R. Rapp and others).

Keywords: Thermochemistry, Reaction Calorimetry, Oxide Melts, Lead Borate

229. Poroelasticity of Rock — FY 1992 $244,000

DOE Contact: W. C. Luth, (301) 903-5822
University of Wisconsin Contact: H. F. Wang, (608) 262-5932
LLNL Contact: B. P. Bonner, (510) 422-7080

The coupled response of rock masses to stress and fluid flow is important in many geologic processes and energy technologies. This research program is an experimental study of static and dynamic poroelastic behavior of rocks. Measurements of Skempton's coefficient and undrained Poisson's ratio together with drained bulk modulus and shear modulus will provide a complete set of the four poroelastic moduli. Wave velocity and attenuation measurements from seismic to ultrasonic frequencies establish a phenomenological model of the effects of permeability, porosity and saturation for seismic exploration.

Keywords: Rock Masses, Stress Fluid Flow, Poroelastic Behavior, Sonic Velocity, Sonic Attenuation, Permeability, Porosity, Saturation

Division of Advanced Energy Projects

The Division of Advanced Energy Projects (AEP) provides support to explore the feasibility of novel, energy-related concepts that evolve from advances in basic research. These concepts are typically at an early stage of scientific definition and, therefore, beyond the scope of ongoing applied research or technology development programs. The AEP also supports high-risk, exploratory concepts that do not readily fit into a program area but could have applications that may span several disciplines or technical areas.

The Division provides a mechanism for converting basic research findings to applications that eventually could impact the Nation's energy economy. AEP does not support either ongoing, evolutionary research or large scale demonstration projects. Projects are supported for a finite period of time, which is typically three years. Technical topics include physical, chemical, materials, engineering, and biotechnologies. Projects can involve interdisciplinary approaches to solve energy-related problems. It is expected that, following AEP support, each concept will be sufficiently developed and promising to attract further funding from other sources in order to realize its full potential.

Materials Preparation, Synthesis, Deposition, Growth or Forming

230. Metallic Multilayer and Thin Film Fabrication — FY 1992 $337,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Lawrence Berkeley Laboratory Contact: Ian G. Brown, (415) 486-4174

The application of pulsed metal plasma gun techniques to the fabrication of metallic superlattices, multilayers and thin films will be investigated. Multilayer structures will be synthesized that are of relevance to X-ray optical devices, to magnetic and magneto-optical recording media, and to the fabrication of high temperature superconducting thin films. The quality and characteristics of the thin film structures formed in this way will be explored. This means of fabrication of metallic multilayer systems is new and has not yet been examined except in preliminary testing at this laboratory. At the same time, interest in artificial metallic superlattices from a fundamental scientific perspective, and in metallic multilayer structures from the standpoint of applied technology, is growing rapidly. It is important to explore and develop the application of this new technique to these fields. The proposed program will make immediate application of the method within the three fields mentioned above (X-ray optics, magneto-optics and superconducting thin films) via collaborations with materials science research groups at this laboratory.

Keywords: Thin Films, Plasma Gun

231. <u>Synthesis of New High Performance Lubricants and Solid Lubricants</u> FY 1992
$272,000

DOE Contact: Walter M. Polansky, (301) 903-5995
University of Texas Contact: Richard J. Lagow, (512) 471-1032

Work will be conducted on the synthesis and characterization of perfluoropolyethers, an extraordinary class of high performance lubricants, by a relatively new technique, direct fluorination, which is emerging as the best way to prepare perfluoropolyethers. Many new and important classes of perfluoropolyethers will be prepared with very significant potential as lubricants. Currently the highest obtainable molecular weight perfluoropolyether synthesized using conventional polymerization processes is 50,000. This fluid with a molecular weight of 50,000 has a viscous syrup-like consistency. High molecular weight solids with a perfluoropolyether backbone have not been attained using methods other than direct fluorination technology. There exists now the capability to synthesize perfluoropolyethers with molecular weights over 1,000,000. Thus solid perfluoropolyether lubricants are accessible for the first time. A feature of direct fluorination technology where hydrocarbon structures are converted to fluorocarbon structures is that the organic precursors are converted to fluorocarbon fluids and solids without substantial cross-linking and without increases or decreases in degree of polymerization. The synthesis of hydrocarbon polymers as starting materials has many other advantages and introduces great flexibility and capabilities not attainable using polymerization processes with various perfluorinated ethylene oxides. Work will be done on many generic classes of solid fluorocarbon lubricants. The capability to make perfluoropolyethers soluble (miscible) with less expensive hydrocarbon lubricants and poly alpha olefins has recently been developed. One of the most important and promising prospects of this research is the synthesis of chlorinated perfluoropolyether fluids which are very compatible and soluble in hydrocarbons offering potential as high performance lubrication additives.

Keywords: Lubricants, Fluorination

232. <u>Design of Materials with Photonic Band Gaps</u> FY 1992
$299,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Ames Laboratory Contact: Kai-Ming Ho, (515) 294-3481

This project is intended to design, fabricate and characterize a new class of composite materials which possess forbidden ranges of frequencies, in which electromagnetic waves cannot propagate in any direction. These materials are called "photonic crystals" and the forbidden frequencies are called "photonic gaps" and they can be regarded as photonic analogues of electronic semiconductors with electronic gaps. This class of material will exhibit many interesting physical properties, and will find important practical applications in lasers, mirrors, resonators, filters, and quantum optical devices. Theory will be directed at designing periodic dielectric structures that give the optimal frequency gap for various applications with special emphasis on the fabricability of these structures, especially in the sub-micron length scale where these materials will find applications in optical measurements. Experimental effort will apply theoretical results to fabricate structures in the micron and sub-micron length scales, using micro-fabrication patterning and etching techniques. The effect of disorder, defects and structural imperfections on the propagation of electromagnetic waves through these photonic crystals will be studied.

Keywords: Patterning, Plutonics, Optical Devices

233. High Flux, Large-Area Carbon-Cluster Beams for Thin-Film Deposition and Surface Modification — FY 1992 $445,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Argonne National Laboratory Contact: Dieter M. Gruen, (708) 252-3513

Fullerenes, such as C_{60} or buckminsterfullerene, are kinetically stable carbon cluster molecules, but are thermodynamically unstable with respect to diamond and graphite by ~5 kcal/mol C. The fact that C_{60} has a vapor pressure of $\sim 10^{-3}$ Torr at 500°C opens up the possibility of generating high-flux, high-energy carbon-cluster ion beams for thin-film deposition (including diamond films) and surface modification. A microwave-driven electron cyclotron resonance (ECR) plasma source will be used to generate the fullerene ion beams. The substrate impact energy will be controlled independently of the plasma parameters by biasing the substrate. End-Hall optics will be combined with the ECR plasma to provide additional control of beam characteristics. The ECR facility will be used to synthesize and characterize, by a variety of techniques, diamond films, diamond-like films, and carbon-implanted layers on large areas with high-deposition rates. The effort is directed toward producing high-quality films at low-substrate temperatures in a manner that can be adapted to industrial processes.

Keywords: Fullerenes, Ion Beams, Diamond, Diamond-like Carbon

234. Novel Composite Coatings for High Temperature Friction and Wear Control — FY 1992 $428,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Oak Ridge National Laboratory Contact: Theodore M. Besmann, (615) 574-6852

Chemical vapor deposition (CVD) techniques offer the opportunity to create very uniform self-lubricating composites which slowly wear away to expose pockets of lubricants which then spread across the surface. In CVD gaseous reactants are allowed to flow over a heated substrate where they react and deposit a solid coating. Solid lubricants have higher use temperatures and higher load-bearing capacities than do liquid lubricants. Consequently, they find use in applications where liquid lubricants prove inadequate. It has been noted that because buckminsterfullerene (C_{60}) is a spherical macromolecule and is thought to be very stable and slow to react with other substances, it should make an excellent lubricant. This project utilizes the controlled wear of a hard matrix to reveal the embedded high-temperature, solid lubricant. Such a composite coating would be produced by CVD, which has been demonstrated capable of producing multiphase coatings of controlled composition and microstructure. The C_{60} phase cannot be simultaneously formed during deposition, as can other of the proposed lubricants. The material can be incorporated into a coating, however, by entrainment in the coating gases.

Keywords: Chemical Vapor Deposition, Fullerenes, Composites

235. Synthesis of Advanced Composite Ceramic Precursor Powders by the Electric Dispersion Reactor

FY 1992
$179,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Oak Ridge National Laboratory Contact: Timothy C. Scott, (615) 574-5962

The use of high-intensity-pulsed electric fields for droplet size control in dispersed liquid systems is being investigated. This technology has been utilized in a device called the electric dispersion reactor (EDR) to carry out the synthesis of micron-sized particles for the production of precursor powders of advanced ceramic materials. In this approach, pulsed electric fields are employed to create dispersions of microscopic conducting (aqueous-based) drops in nonconducting (organic) liquids. Each of these droplets becomes a localized microreactor where reactants in the organic phase diffuse into the aqueous droplets in which precipitation and gelation occur, while water and reaction products diffuse into the organic phase. The particle morphology is altered by varying reactant compositions in the liquid-liquid system while achieving intraparticle stoichiometric consistency. This leads to the production of high-quality precursor powders which, in turn, yields dense, consistent green-body material. Furthermore, this method requires far less energy expenditures than conventional approaches which rely on such inefficient operations as solids blending, mixing, and grinding to accomplish the formation of mixed-oxide precursor material.

Keywords: Ceramics, Ceramic Precursors

236. New Ion Exchange Materials for Environmental Restoration and Waste Management

FY 1992
$430,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Argonne National Laboratory Contact: E. Philip Horwitz, (708) 972-3653

The objective of this program is to synthesize, characterize, and evaluate a new class of cation exchange resins. The new resins will contain the geminally substituted diphosphonic acid functional group. Ion exchange resins containing geminally substituted diphosphonic acid groups should have vastly superior properties compared to commercially available cation exchange resins and should find wide-scale applications in environmental restoration (e.g., groundwater cleanup) and in waste management (e.g., minimization of waste volume). Alkyl-1,1-diphosphonic acids are among the most powerful complexing agents for polyvalent metal ions in aqueous solution, particularly at pH<2. But, heretofore, it has not been possible to synthesize resins containing diphosphonic acid groups, because of the difficulty of introducing this group into a preformed polymer matrix. The synthesis of resins with the diphosphonic acid groups will be accomplished by the polymerization of vinylidene-1,1-diphosphonic acid (VDPA) or by the copolymerization of VDPA with suitable comonomers (e.g., acrylamide/bis-acrylamide or styrene/divinylbenzene). This approach represents a major departure from the traditional methods for preparing ion exchange materials whereby the exchangeable functional groups are introduced onto a preformed polymer matrix.

Keywords: Ion Exchange, Environmental Restoration

237. Development of an Ion Replacement Electrorefining Method

FY 1992
450,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Argonne National Laboratory Contact: Zygmunt Tomczuk, (708) 252-7294

The objective of this project is to investigate promising methods for carrying out a new metal separation and purification process called ion replacement electrorefining. The challenge and program focus lies in developing a counter electrode that can serve in a sequential and, if possible, reversible manner as a cathode during metal dissolution and an anode during metal separation/deposition. The key goal is to produce a clean separation between actinide and non-actinide elements, such as required for the separation of spent nuclear reactor fuel. One potential use for the ion replacement electrorefining method is the reprocessing of spent metal fuel from an Integral Fast Reactor (IFR), but it is also adaptable to the separation of transuranic elements from spent fuel and waste generated by the light water nuclear reactor (LWR) industry and the defense nuclear programs. In the case of the IFR, the proposed process offers a simplification of the conventional pyrometallurgical electrorefining process under development within the IFR program. For LWR and defense waste applications, the ion replacement electrorefining method could be used in conjunction with processes that incorporate reduction of actinide element compounds (usually oxides) to a metallic form.

Keywords: Separations, Actinides, Electrorefining

Materials Properties, Behavior, Characterization or Testing

238. A Study of Potential High Band-Gap Photovoltaic Materials for a Two Step Photon Intermediate Technique in Fission Energy Conversion

FY 1992
$294,000

DOE Contact: Walter M. Polansky, (301) 903-5995
University of Missouri Contact: Mark A. Prelas, (314) 882-3550

The efficiency of modern day power plants is limited by the steam cycle that they employ. Future power plants may be able to improve upon the efficiency of the steam cycle provided that other energy conversion techniques become available. One such energy conversion method is excimer channeling. Excimer channeling is a method of efficiently creating a narrow band photon spectrum directly from the products of nuclear reactions. This narrow band photon spectrum can be used in an energy conversion cycle based upon photovoltaic reactions. This project addresses the issue of photovoltaic materials that can interface with the narrow band photon spectrum in the excimer channeling energy conversion method. Photovoltaics are generally thought to be inefficient because of their association with solar cells. Solar cells are photovoltaic cells that convert the broad band photon spectrum of the sun directly into electricity at an efficiency of 10 percent to as much as 20 percent. Conversion of photons into electricity could be vary efficient (as high as 85 percent) if the photon spectrum were sufficiently narrow, such as that produced by excimer channeling, and matched to the bandgap of the photovoltaic material. Development of high bandgap photovoltaic materials that match the excimer channeling photon spectrum is the goal of this research program. This effort centers on materials with bandgaps exceeding 5 eV. High bandgap crystals will be synthesized, doped to form P-N junctions, characterized by various surface analysis methods, made into photovoltaic cells, the cells' characteristics tested, and the cells' tolerance to various types of electromagnetic radiation assessed.

Keywords: Photovoltaics, Nuclear Reactors

239. Nonlinear Optics in Doped Fibers FY 1992
$364,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Stanford University Contact: Richard H. Pantell, (415) 723-2564

The objective of this project is to develop a novel and simple technology for optical, all-fiber switches based on the third order nonlinear effect in doped, single-mode fibers. The principle is that when exciting a transition near resonance the electronic distribution changes and so does the contribution of this transition to the refractive index of the material. In this novel approach, a fiber doped with an appropriate impurity, is excited optically near an absorption resonance of the impurity to produce strongly enhanced nonlinear susceptibilities. Modeling shows that it is then possible to reduce the pump and length requirements by several orders of magnitude each, and to produce a π phase shift in centimeter lengths with milliwatts of pump power. A variety of impurities will be investigated. For picosecond response times, the reduction in the pump power-fiber length product is predicted to be 7-8 orders of magnitude over undoped silica. Erbium and neodymium doped fibers will also be studied. This investigation is anticipated to open the door to the first low-power, ultra-short switches and modulators made with single-mode optical fibers, operated with a low-power, long-lifetime laser diode. There are a variety of energy applications for the proposed research, including oil exploration, control of power substations, and management of consumer distribution systems.

Keywords: Optical Switch, Nonlinear Optic, Optical Fibers

Device or Component Fabrication, Behavior or Testing

240. Research on Magnet Replicas and the Very Incomplete Meissner Effect FY 1992
$341,000

DOE Contact: Walter M. Polansky, (301) 903-5995
University of Houston Contact: Roy Weinstein, (713) 749-4385

The aim of this project is to produce permanent magnets comprised of superconductors (SCs) which work at or near liquid nitrogen temperatures (77°K). These magnets show promise for applications in motors, generators, charged particle beam steering, industrial particle separators, MRI, and other constant field applications. One advantage of these magnets is that much higher fields are possible than for ferromagnets. A second is that the SC magnets can accurately copy the fields used to activate them, thus making possible magnet replicas. In addition, SC magnets are lighter than iron, cost less than an electromagnet, and consume negligible energy. The SC magnet replicas are made of bulk pieces of high temperature superconductor, and so do not require the availability of wire, which is difficult to produce from these materials. Both chemical and radiation methods are being used to produce materials capable of storing high field. Stored fields have been increased by factors of over 1,000 in the past two years. Presently, stored fields in 1 cm^3 samples are already three-ties stronger than samarium-cobalt magnets. Methods of improving stability have been found. A small motor has been run using a magnet replica.

Keywords: High Temperature Superconductors, Motor

241. Ultrafast Molecular Electronic Devices — FY 1992 $445,000

DOE Contact: Walter M. Polansky, (301) 903-5995
Argonne National Laboratory Contact: Michael R. Wasielewski, (708) 252-3538

The objective of this project is to apply the fundamental chemistry of ultra-fast photo-initiated electron transfer reactions to produce high speed, energy efficient molecular electronic devices. These molecules, designed around electron donor-acceptor molecules, will act as opto-electronic switches on a picosecond time scale. Photo-excitation of these molecules with visible light results in very efficient charge separation reactions that set the on or off state of the molecular switch. Two types of switches will be developed: (1) a bistable electron transfer switch that will use a light pulse of one color to store information in the solid state in the form of a long-lived charge separation, and a light pulse of a second color to recover it; and (2) a field effect switch that will use the electric field generated by one charge separated electron donor-acceptor pair to influence the on or off state of a second donor-acceptor pair. These electro-optic switch molecules will be assembled in ordered arrays on surfaces using self-assembled monolayer and liquid crystal polymer technology. Potential applications of this technology are optical computing, wavelength selective gates and switches, laser detectors, electro-optic devices, modulators, and memories.

Keywords: Opto-electronic Switches, Liquid Crystals, Photo-initiated Electron Transfer

Small Business Innovation Research Program

The Small Business Innovation Research (SBIR) program is mandated by the Small Business Innovation Research Program Reauthorization Act of 1992. The program is designed for implementation in a three-phase process, with Phase I determining, insofar as possible, the scientific or technical merit and feasibility of ideas proposed for investigation. The period of performance in this initial phase is about six months and awards have been limited to $50,000. Phase II is the principal research or research and development effort, and awards have been as high as $500,000 for work to be performed in periods of up to two years. Under Phase III, commercial applications of the research or research and development are to be pursued by small businesses with non-Federal capital or, alternatively, Phase III may involve follow-on non-SBIR Federal contracts for products or processes desired by the Government.

The materials-related projects, like all other projects in the DOE SBIR program, were selected using the specific evaluation criteria listed in the program solicitation. Conclusions were reached on the basis of detailed reports returned by reviewers drawn from DOE laboratories, universities, private industry, and government. In the case of Phase II, in some cases in which several proposals were judged to be of approximately equal technical merit, preference was given to those proposals that had demonstrated third phase, non-Federal capital commitments.

The work supported in this program represents high-risk research, but the potential benefits are also high if the objectives are met. Brief descriptions of all DOE SBIR projects (not just those of interest in materials research) are given in the following publications: Abstracts of Phase I Awards, 1992 (DOE/ER-0560), Abstracts of Phase II Awards, 1992 (DOE/ER-0561), and Abstracts of Phase II Awards, 1991 (DOE/ER-0506). Copies of these publications may be obtained by calling Mrs. Kay Etzler on (301) 903-5867.

Materials Preparation, Synthesis, Deposition, Growth or Forming

Phase I Projects:

On-Line Diagnostics for Fullerene Synthesis - DOE Contact Robert Marianelli, (301) 903-5804; Advanced Fuel Research, Inc. Contact Dr. Michael A. Serio, (203) 528-9806

Development of High-Selectivity, High-Permeability, Flexible Plasma-Polymer Membranes - DOE Contact Robert Marianelli, (301) 903-5804; Bend Research, Inc. Contact Dr. Dwayne T. Friesen, (503) 382-4100

Development of a Novel Reverse-Osmosis Membrane with High Rejections for Organic Compounds - DOE Contact Robert Marianelli, (301) 903-5804; Bend Research, Inc. Contact Dr. Scott B. McCray, (503) 382-4100

Selective Ceramic Membranes from Hybrid Templates - DOE Contact Robert Marianelli, (301) 903-5804; Cape Code Research, Inc. Contact Dr. Brian G. Dixon, (508) 540-4400

A New Class of Ionomer Membranes - DOE Contact Robert Marianelli, (301) 903-5804; EIC Laboratories, Inc. Contact Dr. Timothy L. Rose, (617) 769-9450

Stoichiometric Precursors for Preparation of Advanced Metal Oxide Solid Electrolytes - DOE Contact Robert Marianelli, (301) 903-5804; Eltron Research, Inc. Contact Dr. Anthony F. Sammells, (303) 440-8008

A Thin Solid Film Electrochemical Oxygen Partial Pressure Sensor for Control of Low Vacuum Inert Gas Processes - DOE Contact Robert Marianelli, (301) 903-5804; Jet Process Corporation Contact Dr. Bret L. Halpern, (203) 786-5130

Novel Durable Sol-Gel Fiber-Optic Sensors for Use in Elevated Temperature and/or Pressure Environments - DOE Contact Robert Marianelli, (301) 903-5804; Laser Photonics Technology, Inc. Contact Mr. Martin Casstevens, (716) 636-3626

Development of a Process to Synthesize Tubular Fullerenes - DOE Contact Robert Marianelli, (301) 903-5804; Materials and Electrochemical Research Corporation Contact Dr. J. C. Withers, (602) 574-1980

Molecular-Sieve-Modified Polymeric Membranes for Gas and Vapor Separation - DOE Contact Robert Marianelli, (301) 903-5804; Membrane Technology and Research, Inc. Contact Dr. Ingo Pinnau, (415) 328-2228

A Low Cost Process for Manufacture of Fullerenes - DOE Contact Robert Marianelli, (301) 903-5804; Pacific Fullerene Specialties Contact Mr. David Lane, (408) 293-0830

Continuous Production of Fullerenes from Hydrocarbon Precursors - DOE Contact Robert Marianelli, (301) 903-5804; TDA Research, Inc. Contact Mr. John D. Wright, (303) 422-7918

Preparation of Low-Density Microcellular Materials from Fullerenes - DOE Contact Robert Marianelli, (301) 903-5804; TDA Research, Inc. Contact Dr. William L. Bell, (303) 420-4329

A Novel Fabrication Method for Diamond Composites - DOE Contact Manfred Leiser, (301) 903-3426; Advanced Technology Materials, Inc. Contact Dr. Phil Chen, (203) 794-1100

In Situ Surface Elemental Analysis/Process Control at Millitorr Pressure During Superconductor Film Deposition - DOE Contact Manfred Leiser, (301) 903-3426; Ionwerks Contact Dr. J. Albert Schultz, (713) 522-9880

Control of Thin Film Microstructures by Gasdynamic Energy Deposition - DOE Contact Manfred Leiser, (301) 903-3426; Jet Process Corporation Contact Dr. Bret Halpern, (203) 786-5130

A Generic Chemical Processing Technology for the Production of Nanostructured Composite Materials - DOE Contact Manfred Leiser, (301) 903-3426; Nanodyne, Inc. Contact Dr. Larry E. McCandlish, (908) 249-8347

Fast-Pulse Hot Pressing with Fine-Scale Adiabatic Heating - DOE Contact Manfred Leiser, (301) 903-3426; Polaris Research Contact Mr. M. Dean Matthews, (408) 261-2803

Production of Controlled Microstructure Nanophase Ceramic Powders - DOE Contact Manfred Leiser, (301) 903-3426; PSI Technology Company Contact Dr. Joseph J. Helble, (508) 689-0003

Ductile High-Temperature Superconducting Alloys - DOE Contact Manfred Leiser, (301) 903-3426; Quantum Magnetics, Inc. Contact Dr. Ronald E. Sager, (619) 481-4015

New Gadolinium-Boron Compounds for Neutron Capture Therapy - DOE Contact Gerald Goldstein, (301) 903-3213; Boron Biologicals, Inc. Contact Dr. Bernard F. Spielvogel, (919) 832-2044

Lead Sulfate Scintillators for Use in Position Emission Tomography - DOE Contact Gerald Goldstein, (301) 903-3213; Radiation Monitoring Devices, Inc. Contact Mr. Kanai Shah, (617) 926-1167

Refractory Metal Coatings on Carbon/Carbon Composites for First Wall Applications - DOE Contact Marvin Cohen, (301) 903-4253; Applied Sciences, Inc. Contact Mr. Jyh-Ming Ting, (513) 766-2020

A Thermal Composite Plasma Facing Material - DOE Contact Marvin Cohen, (301) 903-4253; Energy Science Laboratories, Inc. Contact Dr. Timothy R. Knowles, (619) 552-2034

A Niobium-Tin Multifilamentary Composite Superconductor with Artificial Copper (Bronze) Inclusions - DOE Contact Marvin Cohen, (301) 903-4253; Supercon, Inc. Contact Dr. Dingan Yu, (508) 842-0174

A Porous Metal Heat Exchanger to Cool a Microwave Cavity - DOE Contact T. V. George, (301) 903-4957; Thermacore, Inc. Contact Mr. John H. Rosenfeld, (717) 569-6551

Development of Silicon Carbide Ceramic Composites for Fusion Reactor Applications - DOE Contact F. W. Wiffen, (301) 903-4963; Materials and Electrochemical Research Corporation Contact Dr. J. C. Withers, (602) 574-1980

Addition of Silicon to the Copper Between Niobium-Titanium Filaments to Reduce Interdiffusion - DOE Contact William Watson, (214) 708-2417; IGC Advanced Superconductors, Inc. Contact Dr. Eric Gregory, (203) 953-5215

Improved Critical Current Density at High Magnetic Flux in Niobium-titanium-tantalum Superconductors by Artificial Pinning Centers - DOE Contact William Watson, (214) 708-2417; IGC Advanced Superconductors, Inc. Contact Dr. L. R. Motowidlo, (203) 753-5215

Radiation Damage Resistant Silicon for Particle Physics Detectors - DOE Contact William Watson, (214) 708-2417; IntraSpec, Inc. Contact Mr. John Walter, (615) 483-1859

Epitaxial Wide Bandgap Semiconductor Films for Improved Detectors and Electronics - DOE Contact William Watson, (214) 708-2417; Lawrence Semiconductor Laboratories, Inc. Contact Dr. McDonald Robinson, (602) 438-2300

A Hydrogenated Amorphous Silicon Particle Detector - DOE Contact William Watson, (214) 708-2417; Materials Research Group, Inc. Contact Dr. Russell E. Hollingsworth, (303) 425-6688

Epitaxial Silicon Carbide Growth by Plasma Enhanced Chemical Vapor Deposition - DOE Contact William Watson, (214) 708-2417; Materials Research Group, Inc. Contact Dr. Russell E. Hollingsworth, (303) 425-6688

Development of High-Field Chevrel Phase Superconducting Wires Using Low Temperature Processing - DOE Contact Gerald Peters, (301) 903-5228; Supercon, Inc. Contact Dr. Dingan Yu, (508) 842-0174

Indium Iodide Photoconductive Detectors for Use in Scintillation Spectroscopy - DOE Contact Richard Rinkenberger, (301) 903-3613; Radiation Monitoring Devices, Inc. Contact Dr. Michael R. Squillante, (617) 926-1167

Casting of Metallic Nuclear Fuel Pins - DOE Contact Clint Bastin, (301) 903-5259; Creare, Inc. Contact Dr. Thomas Jasinski, (603) 643-3800

Enabling Materials Technology for Nuclear Propulsion - DOE Contact Chet Bigelow, (301) 903-4299; Ultramet Contact Dr. Robert H. Tuffias, (818) 899-0236

Jet Vapor Deposition of Corrosion Resistant Coatings on Critical Molten Carbonate Fuel Cell Components - DOE Contact Tom George, (304) 291-4825; Jet Process Corporation Contact Dr. Bret L. Halpern, (203) 786-5130

Lithium-Graphite Intercalation Anodes - DOE Contact Al Landgrebe, (202) 586-1483; EIC Laboratories, Inc. Contact Dr. Gerhard L. Holleck, (617) 769-9450

Phase II Projects: (First Year)

Graphite and Metal Oxide Catalyst Supports for Rechargeable Oxygen Electrodes - DOE Contact Ken Barber, (202) 586-2198; MATSI, Inc. Contact Mr. Ronald A. Putt, (404) 876-8009

Development of Hollow-Fiber Modules for the Purification of Natural Gas - DOE Contact Harold Shoemaker, (304) 291-4715; Bend Research, Inc. Contact Dr. Scott B. McCray, (503) 382-4100

Inexpensive Pathways for the Synthesis of p-Boronophenylalanine and New Boron Containing Agents - DOE Contact Donald W. Cole, (301) 903-3268; Boron Biologicals, Inc. Contact Dr. Bernard F. Spielvogel, (919) 832-2044

An Improved Method of Introducing Additional Alloying Elements into Nb_3Sn in Internal-Tin Processes - DOE Contact Warren Marton, (301) 903-4965; IGC Advanced Superconductors, Inc. Contact Dr. Eric Gregory, (203) 574-7988

Phase II Projects: (Second Year)

Single CrystalMolybdenum Mirrors for High Power Vacuum Ultraviolet and X-ray Radiation - DOE Contact Jerry Smith, (301) 903-4269; INRAD, Inc. Contact Dr. Warren Ruderman, (201) 767-1910

Ultrathin Metal Membranes - DOE Contact Robert Marianelli, (301) 903-5804; Membrane Technology and Research, Inc. Contact Dr. Amulya Athayde, (415) 328-2228

Changes in Niobium Tin Conductors Made by the Internal Tin Method to Improve Magnet Performance - DOE Contact T. V. George, (301) 903-4957; IGC Advanced Superconductors, Inc. Contact Mr. Gennardy M. Ozyeransky, (203) 753-5215

Alternative Fabrication Processes for Ultrafine Filament, Metal Matrix Microcomposites - DOE Contact T. V. George, (301) 903-4957; Supercon, Inc. Contact Mr. Charles Renaud, (508) 842-0174

A Novel Carbon First Wall Material - DOE Contact Robert Price, (301) 903-3565; Applied Sciences, Inc. Contact Mr. Max L. Lake, (513) 767-1477

Development of Radiation-Resistant Copper Matrix Composites - DOE Contact Robert Price, (301) 903-3565; Materials and Electrochemical Research Corporation Contact Dr. J. C. Withers, (602) 574-1980

Development of New Radiation Resistant Large Stokes Shifted Intermolecular Proton Transfer Fluorescent Composites - DOE Contact Gregory Haas, (214) 708-2510; Nanoptics, Inc. Contact Dr. J. Harmon, (904) 392-7105

More Economical Low Loss Niobium Tin for Pulsed Field Applications, Made by a Modified Internal Tin Process - DOE Contact Gerald Peters, (301) 903-5228; IGC Advanced Superconductors, Inc. Contact Dr. Eric Gregory, (203) 574-7988

A New Semiconductor Photosensor for Scintillation Spectroscopy - DOE Contact Stanley Whetstone, (301) 903-3613; Radiation Monitoring Devices, Inc. Contact Dr. Gerald Entine, (617) 926-1167

Magnetically Enhanced Plasma Deposition of Intrinsic Amorphous Hydrogenated Silicon Layers in Roll-to-Roll Systems - DOE Contact Alec Bulawka, (202) 586-5633; Iowa Thin Film Technologies, Inc. Contact Dr. Frank R. Jeffrey, (515) 294-7732

A Novel Precursor Yarn for Advanced Automotive Composite Structures - DOE Contact Stanley Wolf, (202) 586-9806; Pepin Associates, Inc. Contact Mr. John N. Pepin, (207) 883-8338

Materials Properties, Behavior, Characterization or Testing

Phase I Projects:

Pseudospectral Quantum Chemistry for Periodic Systems - DOE Contact Tom Kitchens, (301) 903-5800; Schrodinger, Inc. Contact Dr. Murco N. Ringalda, (818) 441-2830

Improved Scintillator Materials for Medical Applications - DOE Contact Gerald Goldstein, (301) 903-3213; ALEM Associates Contact Dr. A. Lempicki, (617) 236-1025

A Carbon-Carbon Repair System for the First Wall of the International Thermonuclear Experimental Reactor - DOE Contact Marvin Cohen, (301) 903-4253; Nuclear and Aerospace Materials Corporation Contact Mr. Glen B. Engle, (619) 487-0325

Aluminum-Wettable Coatings on Graphite for Improved Brazed Joints - DOE Contact Marvin Cohen, (301) 903-4253; Technical Research Associates, Inc. Contact Dr. Jared Lee Sommer, (801) 485-4994

Photon-Stimulated Desorption from Cold Surfaces - DOE Contact William Watson, (214) 708-2417; Comstock, Inc. Contact Dr. John E. Talmage, (615) 483-7690

Radiation Hard Diamond Transistors for the Superconducting Super Collider - DOE Contact William Watson, (214) 708-2417; Crystallume Contact Dr. Maurice Landstrass, (414) 324-9681

Large Area Distributed Electronics on Glass by Ion Implantation of Silicon-Germanium Alloys - DOE Contact William Watson, (214) 708-2417; Spire Corporation Contact Dr. Anton C. Greenwald, (617) 275-6000

Development of Methods to Substantially Reduce Photodesorption from Storage Ring Photo Absorbers - DOE Contact Gerald Peters, (301) 903-5228; Roop Scientific Research Contact Dr. Bobbi Roop, (509) 375-0938

Silicides for Thermoelectric Conversion - DOE Contact Chet Bigelow, (301) 903-4299; Hi-Z Technology, Inc. Contact Mr. Norbert B. Elsner, (619) 535-9343

Ceramic Matrix Composites for Magnetohydrodynamic Air Preheaters - DOE Contact Charles Thomas, (412) 892-5731; Ceramic Composites, Inc. Contact Mr. E. L. Ted. Paquette, (410) 987-3435

Novel Energy-Efficient Membrane System for the Recovery of Volatile Organic Contaminants from Industrial Process Gases - DOE Contact Dan Kung, (708) 252-2023; Bend Research, Inc. Contact Dr. Scott B. McCray, (503) 382-4100

Polymer Electrolytes for High Voltage Batteries - DOE Contact Al Landgrebe, (202) 586-1483; EIC Laboratories, Inc. Contact Dr. K. M. Abraham, (617) 769-9450

A Polymer Electrolyte for Lithium Batteries - DOE Contact Al Landgrebe, (202) 586-1483; TechnoChem Company Contact Dr. Shyam D. Argade, (919) 370-9440

Phase II Projects: (First Year)

Thermophotovoltaic Cogenerators for Advanced Integrated Appliances - DOE Contact Terry Statt, (202) 586-9169; Quantum Group, Inc. Contact Dr. Mark Goldstein, (619) 457-3048

Spontaneous Natural Gas Oxidative Dimerization Across Mixed Conducting Ceramic Membranes - DOE Contact Harold Shoemaker, (304) 291-4715; Eltron Research, Inc. Contact Dr. Anthony F. Sammells, (708) 898-1583

A Membrane Process for Hot-Gas Cleanup and Decomposition of H_2S to Elemental Sulfur - DOE Contact Thomas P. Dorchak, (304) 291-4305; Bend Research, Inc. Contact Dr. David J. Edlund, (503) 382-4100

In-Situ FT-IR Diagnostics for Coal Liquefaction Processes - DOE Contact Dr. Shelby Rogers, (412) 892-6132; Advanced Fuel Research, Inc. Contact Dr. Michael A. Serio, (203) 528-9806

Deep Hole Drilling in Refractory Metals Using Abrasive Waterjets - DOE Contact Clint Bastin, (301) 903-5259; Quest Integrated, Inc. Contact Dr. Mohamed Hashish, (206) 872-9500

Improved Properties in Nb 46.5 Weight % Ti Materials for the SSC by Reducing the Separation Between Filaments - DOE Contact Gregory Haas, (214) 708-2510; IGC Advanced Superconductors, Inc. Contact Dr. Eric Gregory, (203) 574-7988

Multifilamentary Nb_3Sn Superconducting Wire Using APC, Composite Filaments with Improved High Field Performance - DOE Contact Gerald Peters, (301) 903-5228; Supercon, Inc. Contact Mr. Kenneth DeMoranville, (508) 842-0174

A Novel Joining Method for Graphite and Carbon-Carbon Composites - DOE Contact Warren Marton, (301) 903-4965; Materials and Electrochemical Research Corporation Contact Dr. Sumit Guha, (602) 574-1980

Phase II Projects: (Second Year)

Utilization of Fluidized Bed Residuals for Producing Synthetic Aggregate by Vacuum Extrusion - DOE Contact Jerry L. Harness, METC, (304) 291-4835; Valley Forge Laboratories, Inc. Contact Mr. Robert J. Collins, (215) 688-8517

An Evaluation of Erosion Resistance in a Simulated Fluidized Bed Combustor Environment of Coatings Applied Using a Hypersonic Spray Process - DOE Contact Gary A. Nelken, METC, (304) 291-4216; Holtgren, Inc. Contact Dr. Edward R. Buchanan, (201) 686-2332

A Novel, Robust, High Repetition, High Brightness Cathode - DOE Contact Gerald Peters, (301) 903-5228; Integrated Applied Physics, Inc. Contact Mr. George Kirkman, (818) 821-0652

An All-Solid-State Titanium-Doped Sapphire Laser Source for Production of Polarized Electrons - DOE Contact Gerald Peters, (301) 903-5228; Lightwave Electronics Corporation Contact Dr. Thomas R. Steele, (415) 962-0755

Device or Component Fabrication, Behavior, or Testing

Phase I Projects:

Nondestructive Measurement of Residual Stresses in Power Plant Piping Welds Using Magnetoelastic Techniques - DOE Contact Oscar Manley, (301) 903-5822; Karta Technology, Inc. Contact Dr. Satish M. Nair, (512) 681-9102

Digital Processing Electronics for X-ray Detector Arrays - DOE Contact Manfred Leiser, (301) 903-3426; X-ray Instrumentation Associates Contact Dr. William K. Warburton, (415) 903-9980

A Cold/Thermal Beam Bender Using Capillary Optics to Increase the Number of End-Guide Instrument Positions - DOE Contact Manfred Leiser, (301) 903-3426; X-ray Optical Systems, Inc. Contact Dr. Qi-fan Xiao, (518) 442-5250

A Thomson-Scattering Plasma Diagnostic for Materials Testing - DOE Contact Charles Finfgeld, (301) 903-3423; Princeton Scientific Instruments, Inc. Contact Dr. Dirck L. Dimock, (908) 274-0774

Eddy-Current Nondestructive Testing Methods for On-Line Detection of Cable Manufacturing Defects - DOE Contact William Watson, (214) 708-2417; SE Systems, Inc. Contact Dr. Duane P. Johnson, (510) 293-3000

Low-Cost Microstrip Detectors on Conductivity-Modified Kapton - DOE Contact William Watson, (214) 708-2417; Spire Corporation Contact Dr. Anton C. Greenwald, (617) 275-6000

Development of a Low Cost Method for the Fabrication of Fine Filament Niobium-Titanium Conductor - DOE Contact William Watson, (214) 708-2417; Supercon, Inc. Contact Dr. James Wong, (508) 842-0172

A Gallium Arsenide Heterojunction Bipolar Transistor for High-Speed Electronic Instrumentation - DOE Contact Robert Woods, (301) 903-3367; Shen Associates Contact Mr. Ahmadreza Rofougaran, (714) 509-9107

High Strength Mono- and Multifilament High Temperature Superconductors for High Field Applications - DOE Contact Gerald Peters, (301) 903-3367; Shen Associates Contact Mr. Leszek R. Motowidlo, (203) 753-5215

Fabrication of Niobium-Aluminum Superconducting Strands Using Mechanical Alloying - DOE Contact Gerald Peters, (301) 903-5228; IGC Advanced Superconductors, Inc. Contact Mr. G. M. Ozeryansky, (203) 753-5215

A Novel Detector for Neutron Spectroscopy - DOE Contact Richard Rinkenberger, (301) 903-3745; Analysis Consultants Contact Dr. B. G. Martin, (714) 830-1033

Acoustic Sensor Development for Use in Robotics - DOE Contact Harry Alter, (301) 903-3745; Analysis Consultants Contact Dr. B. G. Martin, (714) 830-1033

A Uranium Carbide/Rhenium Thermionic Fuel Element - DOE Contact Chet Bigelow, (301) 903-4299; Space Exploration Associates Contact Mr. Elliot B. Kennel, (513) 766-2020

Durable, Low Cost Ceramic Materials for Use in Hot Gas Filtration Equipment - DOE Contact Norman Holcombe, (304) 291-4829; Industrial Filter and Pump Manufacturing Company Contact Mr. Paul Eggerstedt, (708) 656-7800

An Innovative Device to Enhance Jet Pulse Cleaning of Ceramic Candle Filters - DOE Contact Felixa Eskey, (412) 892-4769; Industrial Filter and Pump Manufacturing Company Contact Mr. Paul Eggerstedt, (708) 656-7800

Microwave Joining of Silicon Carbide for Fabrication of Ceramic Heat Exchanger Assemblies - DOE Contact Paul Micheli, (304) 291-4625; Technology Assessment and Transfer, Inc. Contact Dr. Richard Silberglitt, (301) 261-8373

A Torsional Rheometer for Measuring the Properties of Coal and Coal Slurries - DOE Contact Sean Plasynski, (412) 892-4867; Viscoustech Contact Dr. Chandrika Rajagopal, (412) 486-0730

High Cation Mobility Lithium Polymer Batteries - DOE Contact Al Landgrebe, (202) 586-1483; Covalent Associates, Inc. Contact Dr. Larry A. Dominey, (617) 938-1140

A Real-Time X-ray Detector - DOE Contact Stan Sobczynski, (202) 586-1878; Advanced Technology Materials, Inc. Contact Mr. David Kurtz, (203) 794-1100

Multiplexed Thermopile-Based Infrared Imaging Arrays - DOE Contact Stan Sobczynski, (202) 586-1878; Dexter Research Center, Inc. Contact Dr. Robert S. Toth, (313) 427-3921

Novel Ultrasonic Thermometers with Non-Metal Sensing Elements for Hostile Environments - DOE Contact Stan Sobczynski, (202) 586-1878; Research and Manufacturing Company, Inc. Contact Mr. David W. Varela, (602) 889-7900

Phase II Projects: (First Year)

Metal Hydride Refrigerators - DOE Contact Terry Statt, (202) 586-9169; Thermal Electric Devices, Inc. Contact Dr. E. M. Redding, (505) 345-8668

A Catalytic Membrane Reactor for Facilitating the Water-Gas Shift Reaction at High Temperature - DOE Contact James R. Longanbach, (304) 291-4414; Bend Research, Inc. Contact Dr. David J. Edlund, (503) 382-4100

A Single Manifold, Radial Flow, Solid Oxide Fuel Cell - DOE Contact William Huber, (304) 291-4663; Technology Management, Inc. Contact Mr. Michael Petrik, (216) 541-1000

Ceramic/Metal Elements for MHD Sidewalls - DOE Contact Charles A. Thomas, (412) 892-5731; Busek Company, Inc. Contact Dr. V. Hruby, (617) 449-3929

Fabrication of Tungsten and Tungsten-Molybdenum Alloy Tubing - DOE Contact Clint Bastin, (301) 903-5259; Creare, Inc. Contact Dr. Thomas J. Jasinski, (603) 643-3800

A Distributed Fiber Optic Sensor for Reversible Detection of Atmospheric CO_2 - DOE Contact Robert Marianelli, (301) 903-5804; Eltron Research, Inc. Contact Dr. Ronald L. Cook, (708) 898-1583

An Electron Bombarded Semiconductor Device - DOE Contact Gerald Peters, (301) 903-5228; Advanced Technology Materials, Inc. Contact Dr. Charles P. Beetz, Jr., (203) 794-1100

Silicon Junction Diode Absolute Radiometers for Plasma Diagnostics in the Soft X-ray and Vacuum Ultraviolet Spectral Region - DOE Contact Charles Finfgeld, (301) 903-3423; International Radiation Detectors Contact Dr. Raj Korde, (213) 542-0041

Phase II Projects: (Second Year)

A Ceramic Filter for Removal of Particulates from Flue Gas - DOE Contact Thomas D. Brown, (412) 892-4691; CeraMem Corporation Contact Dr. Robert L. Goldsmith, (617) 899-0467

A Ceramic Filter for Removal of Particulates from Hot Gas Streams - DOE Contact Curtis V. Nakaiski, (304) 291-4275; CeraMem Corporation Contact Dr. Robert L. Goldsmith; (617) 899-0467

Development of a Flue Gas Desulfurization Process Using a Novel Regenerative Hydrogen Bromide System - DOE Contact Felixa Eskey, (412) 892-4769; Giner, Inc. Contact Dr. John A. Kosek, (617) 899-7270

Perovskite Solid Electrolytes for Intermediate Temperature Fuel Cells - DOE Contact William Huber, METC, (304) 291-4663; Eltron Research, Inc. Contact Dr. Anthony F. Sammells, (708) 898-1583

Development of Flexible Super-Lattice Artificial Crystals for Use in Fixed-Source Johann X-ray Spectrometers with Application to Analytical Transmission Electron Microscopy - DOE Contact Jerry Smith, (301) 903-4269; Multilayer Optics and X-ray Technology, Inc. Contact Dr. Mark W. Lund, (801) 378-3972

An Efficient X-ray Wavelength Spectrometer for Improved Elemental Analysis on the Analytical Electron Microscope - DOE Contact Jerry Smith, (301) 903-4269; Peak Instruments, Inc. Contact Dr. Nicholas C. Barbi, (609) 737-8133

A Fiber Optic Long Counter - DOE Contact Stanley Whetstone, (301) 903-3613; Fibertek, Inc. Contact Dr. Garry Spector, (703) 471-7671

Advanced High-Concentration High-Efficiency Tandem Photovoltaic Assemblies - DOE Contact Alec Bulawka, (202) 586-5633; Kopin Corporation Contact Dr. Mark B. Spitzer, (508) 824-6696

OFFICE OF ENVIRONMENTAL RESTORATION AND WASTE MANAGEMENT

	FY 1992
Office of Environmental Restoration and Waste Management - Grand Total	$20,015,000
Office of Waste Management Projects	$13,951,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 1,271,000
Technical Support West Valley Demonstration Project	1,271,000
Materials Properties, Behavior, Characterization or Testing	$12,680,000
Materials Characterization Center Testing of West Valley Formulation Glass	459,000
Development of Test Methods and Testing of West Valley Reference Formulation Glass	360,000
Process and Product Quality Optimization for the West Valley Waste Form	250,000
Waste Form Qualification	5,000,000
Immobilization, Volume Reduction, and In-Place Stabilization	6,611,000
Office of Technology Development	$ 6,154,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 5,554,000
Polymer Solidification	200,000
Polymer Solidification Development for Rocky Flats	160,000
Polymer Solidification Development	775,000
Pyrochemical Treatment of ICPP HLW Calcine	370,000
Minimum Additive Waste Stabilization Program	3,349,000
Spray Casting	500,000
Laser Decontamination and Recycle of Metals	200,000
Materials Properties, Behavior, Characterization or Testing	$ 600,000
Interim Subsurface Barrier for Underground Storage Tanks	150,000
Polyethylene Encapsulation of Low-level Single Shell Tank Wastes	350,000
Pu Oxyhydroxide Volatility	100,000

OFFICE OF ENVIRONMENTAL RESTORATION AND WASTE MANAGEMENT

The Office of Environmental Restoration and Waste Management (EM) was established in November 1989 to effectively coordinate and manage the Department's activities to remediate the DOE Defense Complex and to properly manage waste generated by current operations. This new office combines nuclear waste management and the environmental clean-up elements that were spread across four offices. EM's goal is to insure that risks to human health and safety and to the environment from past, present, and future operations are either eliminated or reduced to prescribed, safe levels by the year 2019.

Office of Waste Operations

Division of Waste Management Projects

The objective of the Office of Waste Management Projects is to conduct waste management activities for ending interim storage of high-level waste and achieving permanent disposal of high-level waste. Defense wastes were generated by atomic energy defense activities at three Departmental operating locations: the Savannah River Site in South Carolina, the Hanford Site in Washington, and the Idaho National Engineering Laboratory in Idaho. Additionally, Congress directed the Department in 1980 to demonstrate the solidification of liquid high-level waste at West Valley (New York) which originated at the nation's only commercial plant to reprocess spent nuclear fuel. At each of the four sites a program is in place to immobilize the high-level waste in preparation for geologic disposal. All four projects are managed by this Office.

At Savannah River and West Valley, high-level waste will be immobilized in a borosilicate glass prepared in a liquid-fed ceramic joule-heated melter. Hanford is planning to use the same waste form and process, but the project is in the detailed design (Title II) and site preparation stage. At Idaho, the merits of several alternative waste management strategies are being evaluated. A strategy will be selected in compliance with the National Environmental Policy Act (NEPA) process for disposal of Idaho high-level waste.

The Defense Waste Processing Facility at Savannah River is beginning nonradioactive operations in preparation for radioactive operation. West Valley is constructing the vitrification cell. For these two projects, materials research focuses on verifying the product consistency of the waste form based on a reference formulation chosen some time ago. The Hanford Waste Vitrification Plant project is studying pre-treatment options to identify the most effective methods of partitioning the several chemically-varied types of waste into a high-activity stream which will be vitrified and a low-activity stream which will be immobilized in a grout or cement waste form.

Materials Preparation, Synthesis, Deposition, Growth or Forming

242. Technical Support to West Valley Demonstration Project FY 1992 $1,271,000

DOE Contact: W. S. Ketola, (716) 942-4314
PNL Contact: W. A. Ross, (509) 376-3644

Pacific Northwest Laboratory (PNL) provides technical assistance to the West Valley demonstration project in characterizing high-level waste samples taken from the West Valley tanks; characterizing operating conditions for ion exchange processes that remove cesium and plutonium from the high level supernate; developing an empirical model which relates borosilicate glass composition to the chemical durability of the final waste form (including both preparation and testing of materials and the statistical analysis of the results to allow modeling); and characterizing individual process operations to show overall control of the vitrification process and the final waste form.

Keywords: Ion Exchange, Borosilicate Glass, Process Control, Radioactive Waste Host

Materials Properties, Behavior, Characterization or Testing

243. Materials Characterization Center Testing of West Valley Formulation Glass FY 1992 $459,000

DOE Contact: W. S. Ketola, (716) 942-4314
PNL Contact: S. C. Marschman, (509) 376-3569

Materials Characterization Center (MCC) is evaluating the chemical durability of glasses whose compositions are within the expected range of composition of the West Valley Demonstration Project borosilicate glass waste form. These include nonradioactive glass containing surrogate elements for radionuclides and radioactive glass doped with appropriate radionuclides. The MCC also began testing of a small sample of glass containing actual West Valley high-level waste. MCC continues to provide assistance to West Valley relative to enhancing the quality of their analytical data.

Keywords: Radioactive Waste Host

244. Development of Test Methods and Testing of West Valley Reference Formulation Glass

FY 1992
$360,000

DOE Contact: W. S. Ketola, (716) 942-3414
CUA Contact: P. B. Macedo, (202) 635-5327

Vitreous State Laboratory (VSL) of the Catholic University of America(CUA) continues to develop test methods for nonradioactive and radioactive borosilicate glass waste forms for the West Valley Demonstration Project and is studying means to maximize the region of acceptable quality around the point of optimal durability for the borosilicate waste form.

Keywords: Radioactive Waste Host

245. Process and Product Quality Optimization for the West Valley Waste Form

FY 1992
$250,000

DOE Contact: W. S. Ketola, (716) 942-4314
AU Contact: L. D. Pye, (607) 871-2432

Alfred University(AU) is studying properties and crystallization behavior of the West Valley borosilicate glass reference composition in anticipation of providing methods for control of product quality during routine manufacture of the West Valley Demonstration Project waste form.

Keywords: Radioactive Waste Host, Borosilicate Glass

246. Waste Form Qualification

FY 1992
$5,000,000

DOE Contact: W. Deerson, (803) 557-1066
WSRC Contact: M. J. Plondinec, (803) 725-2170

These studies provide fundamental data for start-up of the Defense Waste Process Facility, for waste compliance activities, and for acceptance of borosilicate glass at a repository. Site specific testing is included.

Keywords: Waste, Waste Form, Borosilicate Glass, Waste Acceptance Specifications

247. Immobilization, Volume Reduction, and In-Place Stabilization FY 1992
$6,611,000

DOE Contact: S. Burnum, (509) 376-8409
WHC Contact: G. A. Meyer, (509) 372-0415

These studies provide information about the process flowsheets for treatment of high-level waste at the Hanford site and Idaho National Engineering Laboratory. The focus is on reduction of volume of immobilized waste. These studies include waste characterization, retrieval technology, and waste processing. Technology for in-place stabilization is being investigated where appropriate in order to ensure that disposal strategies include all technically feasible options.

Keywords: High-level Waste, Volume Reduction, In-Place Stabilization

Office of Technology Development

The Office of Technology Development (TD) has the mission to facilitate EM's 30-year goal by developing and implementing new technologies which assist DOE in achieving compliance with all applicable statues and regulations. The TD program is designed to make new, innovative, and effective technology available for use and transfer it to users.

Certain areas of TD's program focus on materials research in order to provide better, faster, safer and less expensive approaches to identify, characterize and clean up DOE's waste problem. In the area of soil and ground water remediation, TD is investigating various types of grouting and cement to be used for stabilization and containment of wastes. The applicability of these substances, as well as synthetic materials, is being demonstrated, tested, and evaluated for implementation at specific sites. Research into glasses is also being pursued to better understand in situ and ex situ vitrification technologies, useful in containment of contaminated soils. New fibers are being tested for efficiency and reuse in HEPA filters that DOE uses for air cleaning in a wide variety of operations involving radioactive particulates. TD will continue to fund these materials research projects, as well as others, to provide the basis for other applied research in the TD program.

Materials Preparation, Synthesis, Deposition, Growth or Forming

248. Polymer Solidification — FY 1992 $200,000

DOE Contact: Joel Haugen, (702) 252-2093
BNL Contact: Peter Colombo, (516) 282-3045

This effort, in parallel with "Polymer Solidification Development for Rocky Flats" (CH321204), will provide near-term technical support to Rocky Flats Plant (RFP) for polymer solidification of nitrate salts and other wastes. Several technical issues remain prior to implementation of polyethylene encapsulation of nitrate salt waste at RFP. The focus of this project is determining concentration limits for toxic metals in the waste form that will satisfy EPA leachability criteria and evaluation of potential process modifications to minimize hazardous constituent leachability.

Keywords: Polyethylene, Encapsulation, Leachability

249. Polymer Solidification Development For Rocky Flats — FY 1992 $160,000

DOE Contact: Joel Haugen, (702) 252-2093
BNL Contact: Peter Colombo, (516) 282-3045

This effort, in parallel with "Polymer Solidification" (CH321202), will provide near-term technical support to Rocky Flats Plant (RFP) for polymer solidification of nitrate salts and other wastes. Several technical issues remain prior to implementation of polyethylene encapsulation of nitrate salt waste at RFP. The focus of this project will be selection and testing of a suitable dryer technology for pretreatment of waste prior to polymer processing and characterization of waste particle size for optimal processing.

Keywords: Polyethylene, Encapsulation, Drying Technology

250. Polymer Solidification Development — FY 1992 $775,000

DOE Contact: Reginald W. Tyler, FTS 345-5927
EG&G Rocky Flats Contact: Kirk A. Lindahl, FTS 345-7872

The objective of this task is to demonstrate polyethylene extrusion as a method to solidify nitrate salts generated by the Waste Stream Evaporator treatment process. The development's success will establish if this process can replace the current and planned cementation processes. Polymer solidification developed at Brookhaven National Laboratory has potential for treating HEPA and other filter wastes to meet all disposal requirements. Several streams must be analyzed to decide what encapsulation process is appropriate, and which polymer is most compatible. Particular attention must be given to the up-front processing required to derive a waste form treatable by polymer solidification. Several kinds of lab, bench, and pilot scale equipment will be tested as it is anticipated that one method of encapsulation will not be successful with all waste streams.

Keywords: Encapsulation, Polymer Solidification, Polyethylene

251. Pyrochemical Treatment of ICPP HLW Calcine — FY 1992 $370,000

DOE Contact: W. N. Fitch, (208) 526-4983
WINCO Contact: Dieter Krecht, (208) 526-3627

This program aims to establish the feasibility of a pyrochemical treatment process for Idaho Chemical Process Plant high-level waste calcine. Pyrochemical treatment consists of a high-temperature selective slagging removal of inert materials and electrolytic separation of actinides and fission products in a molten metal/salt environment.

Keywords: Pyrochemical, Calcine, Slag

252. Minimum Additive Waste Stabilization Program — FY 1992 $3,349,000

DOE Contact: Joel Haugen, (708) 252-2093
FEMP/WEMCO Contact: Rod Gimbel, FTS 774-6112

The Minimum Additive Waste Stabilization (MAWS) concept investigates the chemistry of various wastes requiring treatment and disposal (including secondary waste streams during processing) and identifies/combines individual treatment technologies into a treatment system to produce one single final waste form requiring disposal. The focus is on minimizing addition of additives for the vitrification process and minimization of secondary waste streams by combining soil washing and water treatment processes. By blending individual waste streams, additives such as sodium fluxes and silicon dioxide (sand) can be eliminated or greatly reduced thereby producing significant reductions in both cost and waste volume.

Keywords: Vitrification, Glass Melting

253. Spray Casting

FY 1992
$500,000

DOE Contact: W. N. Fitch, FTS 583-4983
MSE Inc. Contact: R. A. Carrington, FTS 587-7416

Spray casting technology is being jointly developed by the Office of Technology Development and the U.S. Air Force. The process uses a controlled aspiration process to spray liquid metal on a substrate as a coating or onto a mold for near net shape forming applications. The coating portion of this project will be demonstrated by the Air Force. The near-net shape portion of this project is sponsored by OTD to support waste minimization in the fabrication of special nuclear material to near-net shape components. Spray forming greatly reduces the amount of waste compared to current fabrication technology. Final demonstration for this portion of the project will be at the Y-12 Plant or other locations involved in the fabrication of special nuclear metal and alloy components.

Keywords: Spray Casting, Near-Net Shape

254. Laser Decontamination and Recycle of Metals

FY 1992
$200,000

DOE Contact: Joel Haugen, (708) 252-2093
Ames Contact: J. P. Corones, (515) 294-4987

This task investigates the use of lasers to decontaminate metals in a manner that requires no solvents, avoids exposure of expensive instrumentation to radionuclides, and can be remotely performed for maximum worker protection. Technology for surface decontamination is based on rastering a powerful laser across the contaminated metal surface. This effectively removes contaminants within 25 microns of the metal surface. Bulk decontamination employs selective laser ionization to remove impurity atoms from molten metal samples.

Keyword: Laser Processing, Decontamination, Metal Recycling

Materials Properties, Behavior, Characterization or Testing

255. Interim Subsurface Barrier for Underground Storage Tanks

FY 1992
$150,000

DOE Contact: Joel Haugen, (708) 252-2093
Brookhaven National Laboratory Contact: P. Colombo, (516) 282-3045

In this program, potential improved barrier materials for interim subsurface confinement for underground storage tanks will be screened and investigated. These candidate barrier materials and processes include advanced, new or non-traditional grouts developed and used in chemical processing and other non-fuel cycle industries with demonstrated ability to withstand aggressive chemicals, high radiation doses and temperature. For selected barrier materials, further work is anticipated to: (1) obtain physicochemical data in laboratory studies; (2) ascertain the effects of placement methodology at pilot plant studies; (3) evaluate placement technologies under full-scale demonstration; and (4) determine relevant properties of full-scale grout specimens relative to conditions anticipated.

Keywords: Grout, Barrier Materials

256. Polyethylene Encapsulation of Low-level Single Shell Tank Wastes — FY 1992 $350,000

DOE Contact: Joel Haugen, (708) 252-2093
BNL Contact: P. Colombo, (516) 282-3045

Applicability of polyethylene encapsulation to single shell tank (SST) wastes will be assessed. The effects of elevated thermal conditions and radiation dose on several key waste for performance parameters such as mechanical strength and leachability will be determined. Standardized compressive strength testing conducted for polyethylene waste forms under ambient conditions will be repeated following a period of extended conditioning at temperatures anticipated under actual disposal conditions. In addition, waste from creep properties will be examined under several temperature regimes to determine impacts on performance. The effects of elevated temperature on containment leachability will be determined by an accelerated leach test that has been developed at Brookhaven National Laboratory (BNL).

Keywords: Polyethylene, Waste Form, Creep, Leachability

257. Pu Oxyhydroxide Volatility — FY 1992 $100,000

DOE Contact: R Scott, (510) 273-7878
LLNL Contact: Oscar Krikorian, (510) 422-8076

This project is an experimental study of PuO_2 volatility (as an oxyhydroxide) in the presence of steam and oxygen at incinerator temperatures. These data are required in order to corroborate predictions of Pu volatility under these conditions.

Keywords: Plutonium, Volatility, Plutonium Oxyhydroxide

OFFICE OF NUCLEAR ENERGY

	FY 1992
Office of Nuclear Energy - Grand Total	$156,262,000
Office of Uranium Enrichment	$ 20,362,000
Gaseous Diffusion	$ 5,062,000
Barrier Quality	2,110,000
Materials and Chemistry Support	2,952,000
Uranium-Atomic Vapor Laser Isotope Separation (U-AVLIS)	$ 15,300,000
Separator Materials	5,000,000
Uranium Processing	10,300,000
Office of Civilian Reactor Development	$ 28,945,000
Office of Advanced Reactor Programs	$ 3,345,000
Division of High Temperature Gas-Cooled Reactors	$ 3,345,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 190,000
Fuel Process Development	190,000
Materials Properties, Behavior, Characterization or Testing	$ 3,155,000
Fuel Materials Development	535,000
Fuel Development and Testing	2,545,000
Graphite Development	40,000
Metals Technology Development	35,000

OFFICE OF NUCLEAR ENERGY (Continued)

	FY 1992
Office of Technology Support Programs (LMRs)	$ 25,600,000
Fuels and Core Materials	$ 25,600,000
Materials Properties, Behavior, Characterization or Testing	$ 25,600,000
Fuel Performance Demonstration	5,100,000
Pyroprocess Development	5,500,000
Fuel Safety Experiments and Analysis	4,500,000
Core Design Studies	3,500,000
Fuel Cycle Demonstration	5,600,000
ALMR Technology R&D	1,000,000
Program Management	400,000
Office of Isotope Systems	$ 1,955,000
Office of Special Applications	$1,955,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 1,605,000
Development of Improved Thermoelectric Materials for Space Nuclear Power Systems	300,000
Development of an Improved Process for the Manufacture of DOP-26 Tridium Alloy Blanks and Exploratory Alloy Improvement Studies	1,075,000
Carbon Bonded Carbon Fiber Insulation Manufacturing Process Development and Product Characterization	230,000
Materials Properties, Behavior, Characterization or Testing	$ 350,000
Characterization of State-of-the-Art Improved Silicon-Germanium Thermoelectric Device/Materials	50,000
Development of an Improved Carbon-Carbon Composite Graphite Impact Shell Replacement Material	300,000
Office of Naval Reactors	$105,000,000*

*Approximate

OFFICE OF NUCLEAR ENERGY

The Office of Nuclear Energy conducts materials research and development through the Office of Uranium Enrichment, the Office of Civilian Reactor Development, the Office of Space and Defense Power Systems, and the Office of Naval Reactors. Summarized below are the areas of research in which the Department is currently engaged.

Office of Uranium Enrichment

The Department of Energy is authorized by the Atomic Energy Act of 1954, as amended, to provide toll uranium enrichment services. Customers deliver natural uranium containing about 0.7 percent uranium 235 to one of DOE's plants and, for a fee, DOE returns material enriched to 2-5 percent in the isotope uranium 235 for use in nuclear power reactors. The goal of the Uranium Enrichment program is to maintain this activity as a strong viable enterprise retaining a market share that preserves a long term competitive position. It is intended that these services be done for worldwide commercial and United States defense customers to help ensure national and energy security in an economical, reliable, safe, secure, and environmentally acceptable manner and also to assure a reasonable recovery of the Government's investment.

Revenues received by DOE for the enrichment of uranium are retained and used for the specific purposes of offsetting costs incurred by the Department in providing uranium enrichment service activities as authorized by Section 201 of Public Law 95-238, not withstanding the provisions of Section 3617 of the Revised Statutes (31 USC 484). The sum appropriated is reduced as uranium enrichment revenues are received during a fiscal year so as to result in no net fiscal year appropriations. Total obligations for all uranium enrichment activities in FY 1991 was approximately $1.3 billion.

Presently, uranium is enriched in gaseous diffusion plants that force uranium hexafluoride (UF_6) gas through porous barriers. These plants are located at Portsmouth, Ohio, and Paducah, Kentucky. A diffusion plant at Oak Ridge, Tennessee, used since World War II, was placed in standby in 1985 and shut down in 1987. In 1985, the DOE determined that of all the new and competing processes the Uranium-Atomic Vapor Laser Isotope Separation (U-AVLIS) process had, and still has, the best potential for providing the lowest cost uranium enrichment in the future.

The U-AVLIS process is based on utilizing differences in the electronic spectra of the atoms of uranium isotopes to induce the selective absorption required for isotopic separation. The process utilizes the controlled vaporization of uranium atoms from metal feed followed by selective excitation and ionization of uranium 235 using tunable lasers in the visible regions of the spectrum. The uranium 235 ions can then be removed from the vapor in a separator using electromagnetic methods. Collection of the metal product is as a liquid that is allowed to solidify upon withdrawal.

The specific statutory authority which established the Department of Energy's role in the enrichment of uranium is the Atomic Energy Act of 1954, as amended. The goal of the Uranium Enrichment Program is to maintain this activity as a strong viable enterprise retaining a market share that preserves a long term competitive position. It is intended that these services be done for worldwide commercial and United States defense customers in an economical, reliable, safe, secure, and environmentally acceptable manner that will ensure a reasonable return on the Government's investment.

Materials R&D activities within the Office of Uranium Enrichment are varied and, for the most part, classified Restricted Data. In FY 1992, approximately $20 million was used in these endeavors. Paragraph summaries of these activities are presented. The DOE contact is A. P. Litman, (301) 903-5777.

Gaseous Diffusion

Uranium as found in nature contains about 0.7 percent uranium 235 and the remainder is essentially non-fissionable uranium 238. The fissionable characteristics of uranium 235 are necessary and useful for power reactor fuel. To date, most nuclear reactors designed for producing electrical power require uranium 235 concentrations between 2 and 5 percent. Presently, uranium is enriched to the desired uranium 235 assay levels in gaseous diffusion plants. These plants operate on the principle that lighter weight gaseous isotopes have slightly higher average velocities and thus can be made to diffuse through a porous barrier more rapidly than heavier species. Two streams can be created, one enriched in the lighter isotope and one depleted. Because enrichment for a single cycle, or stage, is very small, a cascade of stages is required. For example, a plant constructed for producing 4 percent assay U-235 would contain about 1200 stages. Although many other methods for enrichment are still being investigated and another production technique is being used in parts of Europe, diffusion plants today still provide approximately 90 percent of the world's enrichment services. The United States gaseous diffusion plants are located at Portsmouth, Ohio, and Paducah, Kentucky. A diffusion plant at Oak Ridge, Tennessee, used since World War II, was placed in standby in 1985 and shut down in 1987.

258. Barrier Quality

FY 1992
$2,110,000

These studies include evaluation of the short- and long-term changes in the separative capability of the gaseous diffusion barrier and methods to recover and maintain barrier quality in the production plants. This activity is a long-term undertaking and will be maintained at appropriate levels of effort in the future.

Keywords: Nuclear Fuel Isotopic Separations, Gaseous Diffusion, Barrier, Uranium

259. Materials and Chemistry Support

FY 1992
$2,952,000

This activity involves the routine materials and chemistry support for the diffusion plants. It includes the characterization of contaminant-process gas cascade reactions, the physical and chemical properties of UF_6 substances. Also, the work incorporates studies of the corrosion of materials, failure analyses, improving trapping technology, and alternative materials replacement on a continuing basis.

Keywords: Nuclear Fuel Isotopic Separations, Uranium, Gaseous Diffusion

Uranium-Atomic Vapor Laser Isotope Separation (U-AVLIS)

The U-AVLIS process is based on utilizing the differences in the electronic spectra of uranium isotopes to induce the selective absorption required for isotopic separation. The process utilizes the controlled vaporization of uranium atoms from metal feed followed by selective excitation and ionization of uranium 235 using tunable lasers in the visible regions of the spectrum. The uranium 235 ions can then be removed from the vapor in a separator using electromagnetic methods. Collection of the product is as a liquid metal that is allowed to solidify upon withdrawal.

In June 1985, DOE selected U-AVLIS for further development and possible future deployment into the uranium enrichment enterprise. The primary emphasis for the U-AVLIS program in FY 1992 was to conduct demonstrations, including a large-scale demonstration of the entire U-AVLIS enrichment process in full size equipment in 1992. Available resources in FY 1992 were focused on these demonstration goals. As shown below, the U-AVLIS mataerials activities in FY 1992 were in support of the separator system and the uranium processing activities. The latter technologies will have strong economic leverage for a U-AVLIS production plant. The overall goal of uranium processing is to develop and demonstrate low-cost paths for integrating the U-AVLIS metal feed and product into the existing uranium oxide/fluoride nuclear fuel cycle.

260. Separator Materials

FY 1992
$5,000,000

This activity includes the selection and testing of alternative candidate structural and component materials and coatings for the U-AVLIS separator system. It also supports the fabrication of full size separator components and subsystems for verification tests, plus the off line operation of a full size separator.

Keywords: Enrichment, Uranium, Laser Isotope Separation, Uranium-Atomic Vapor Laser Isotope Separation (U-AVLIS)

261. Uranium Processing

FY 1992
$10,300,000

This year experiments and design studies concentrated on alternatives for preparation of U-AVLIS feed from uranium ore. To support the back end of the fuel cycle, the design of a demonstration system for U-AVLIS metal product conversion to precursor oxide for light water power reactor fuel was continued. Interfacing continued with the private sector converters, metal makers, and fuel fabricators to lay the groundwork for efficient integration of U-AVLIS requirements into the nuclear fuel cycle, the overall goal of this effort.

Keywords: Enrichment, Uranium, Laser Isotope Separation, Uranium-Atomic Vapor Laser Isotope Separation (U-AVLIS)

Office of Civilian Reactor Development

Office of Advanced Reactor Programs

Division of High Temperature Gas-Cooled Reactors

The objective of this division is to develop the base technology, systems concepts, and reactor designs which will permit the Government, in cooperation with utilities and private industry, to commercialize the High Temperature Gas-Cooled Reactor (HTGR). The materials interests of this division include those required for the development of coated particles fuels, graphite moderator and reflector blocks, graphite core support blocks and posts, and heat exchanger tubing, tube sheets and vessels. The DOE contact for these projects is P. Williams, (301) 903-2022.

Materials Preparation, Synthesis, Deposition, Growth or Forming

262. Fuel Process Development | FY 1992 $190,000

DOE Contact: P. Williams, (301) 903-3985
General Atomics Contact: R. F. Turner, (619) 455-2306

This work includes establishing, characterizing, and qualifying fabrication processes and equipment for the preparation of microsphere fuel particles of uranium-oxycarbide (UCO) coated with layers of pyrolytic carbon (2) and silicon carbide (1). Major processing operations include solution mixing, kernel forming, drying, calcining, and sintering. Coatings are applied in a fluidized-bed furnace at temperatures up to 1600°C. The objective is to develop kernel fabrication and coating specifications, which yield very low fractions of defective particles. This work also includes development of the process for fabricating the fuel compacts (short rods).

Keywords: Fuel, Ceramics, Sintering, Coatings, Chemical Vapor Deposition

Materials Properties, Behavior, Characterization or Testing

263. Fuel Materials Development | FY 1992 $535,000

DOE Contact: P. Williams, (301) 903-3985
General Atomics Contact: R. F. Turner, (619) 455-2306

This work includes development of the technology base required to design, qualify, and license the fuel systems for near-term steam cycle and advanced process heat HTGRs. These efforts are focused on the low enriched uranium-oxycarbide/thorium-oxide fuel system. Major elements of the work include the preparation and evaluation of irradiation specimens, development and verification of fuel performance models, and preparation and updating of fuel specifications and a design data manual.

Keywords: Fuel, Ceramics, Coatings, Microstructure, Radiation Effects, Diffusion, High Temperature Service

264. Fuel Development and Testing | FY 1992 $3,545,000

DOE Contact: P. Williams, (301) 903-3985
ORNL Contact: M. J. Kania, (615) 576-4856

This work supports development of the technology base required to design, qualify, and license the fuels systems for steam cycle HTGRs. These efforts are focused on the low-enriched uranium-oxycarbide/thorium-oxide fuel system. Major elements of the work include services associated with the design, assembly, and irradiation of fuel capsules, and post-irradiation examination work in support of qualification and licensing of the reference fuel system. This work also includes support of International Cooperatives with West Germany and Japan, and a fuel test program in the French CEA COMEDIE Test Facility.

Keywords: Fuel, Ceramics, Coatings, Microstructure, Radiation Effects, Diffusion, High Temperature Service

265. Graphite Development FY 1992
$40,000

DOE Contact: P. Williams, (301) 903-3985
General Atomics Contact: R. Vollman, (619) 455-3310

This work supports the evaluation and qualification of graphite materials for applications in HTGRs. Major goals of this work are to develop high strength graphites with sufficient stability under irradiation to be qualified for core components, and with sufficient oxidation resistance to be qualified for core support components. The major element of this work is the development of graphite materials test specifications and failure criteria required for reliable design analyses.

Keywords: Graphite, Ceramics, Irradiation Effects, Strength, Corrosion, High Temperature Service

266. Metals Technology Development FY 1992
$35,000

DOE Contact: P. Williams, (301) 903-3985
General Atomics Contact: R. F. Turner, (619) 455-2306

This work includes activities to characterize and qualify the metallic materials selected for applications in the HTGR system. Tasks involve work to establish the database required for design validations and code qualifications. Principal alloy is Alloy 800H.

Keywords: Alloys, Strength, Corrosion, Joining, Microstructure, High Temperature Service

Office of Technology Support Programs (LMRs)

The applied research and development technology activities, conducted at several national laboratories, industrial organizations, universities, and through bilateral and trilateral technology programs and exchanges with foreign nations, relate to current and advanced reactor systems. The scope of these activities include the following areas: fuel cycles; design and performance of high quality core components for fuels, blanket, and control systems; development of the structural materials used in these components and systems; development and demonstration of equipment, processes, and procedures for fabricating, processing, handling, and producing binary and ternary metal fuels, mixed oxide bearing fuels materials, and components; sodium technology; standards and quality assurance; assuring a reliable high quality economical fuel supply for LMRs; destructive and nondestructive testing, examination, and evaluation of core components and the facilities and capabilities for conducting such examinations; responsibility for engineering and supporting facilities; associated safety, safeguards, and nonproliferation; maintaining competent capabilities in the several contractor organizations that conduct the pertinent R&D activities and programs. These activities are responsive to the administration's policies and goals, the National Energy Strategy and, to the DOE programs that support them.

In-reactor and out-of-reactor property evaluations are being conducted on core materials, clad/ducts, fuels and absorber materials. Through irradiation testing in FFTF and EBR-II, the Technology Support Programs are developing, qualifying, and verifying the use of reference, improved and advanced metal fuels, mixed oxide fuels, and boron carbide absorbers, including full-size driver and blanket fuels, and absorber pins and assemblies. Fabrication development, evaluation, qualification, and verification (raw material processing, melting, hot working, cold working, and finishing) are conducted on reference, improved, and advanced alloys including in-reactor qualification of pins, ducts, and assemblies. Improved insulation is being developed for advanced electromagnetic pumps which can operate without cooling. Improved and advanced materials are being tested for use in future cores. The testing for these programs is primarily conducted at government laboratories: Argonne National Laboratory at Chicago, Illinois and Idaho Falls, Idaho; Oak Ridge National Laboratory at Oak Ridge, Tennessee; Westinghouse Hanford Company at Richland, Washington; and universities.

Fuels and Core Materials

Materials Properties, Behavior, Characterization or Testing

267. Fuel Performance Demonstration

FY 1992
$5,100,000

DOE Contact: Andrew Van Echo, (301) 903-3930
ANL Contact: Leon C. Walters, (208) 533-7384

Establish U-Pu-Zr fuel fabrication process, irradiation performance characteristics and high burn-up capability. EBR-II lead test achieved 17.1 and 18.4 a/o burn-up and were removed for PIE. Program plans to complete initial off-normal testing in EBR-II, including RBCB and fabrication variable tests.

Keywords: Breeder Reactor, Actinides, Fuel, Burn-up

268. Pyroprocess Development FY 1992
$5,500,000

DOE Contact: Eli I. Goodman, (301) 903-2966
ANL Contact: James J. Laidler, (708) 252-4479

Establish technical feasibility of the proposed pyroprocesses including electrorefining, waste treatment, and waste form production processes. Program will complete selection of optimum cathode configuration for electrorefining process, conduct engineering scale (10 kg) demonstration of electrorefining with uranium, and run laboratory-scale demonstration of waste treatment and waste form production processes. Process optimization and development of a commercially applicable flow sheet will be carried out.

Keywords: Waste Treatment, Electrorefining, Pyroprocesses

269. Fuel Safety Experiments and Analyses FY 1992
$4,500,000

DOE Contact: Harry Alter, (301) 903-3766
ANL Contact: John F. Marchaterre, (708) 252-4570

Conduct analyses and experiments required for the demonstration of the safety performance of metallic fuel in fast reactor systems. Include transient fuel behavior, validated models and codes which describe fuel behavior, and safety mechanisms which contribute to inherent safety. Program will initiate analysis of high plutonium fuel to expand the data base pertaining to safety margins, failure thresholds, and transient behavior.

Keywords: Reactor Safety, Actinides, Fuel, Inherent Safety, Transient Behavior

270. Core Design Studies FY 1992
$3,500,000

DOE Contact: Philip B. Hemmig, (301) 903-3579
ANL Contact: David C. Wade, (708) 252-4858

Provide direct support in developing optimized metallic core designs for ALMR and establish a validated design and safety analysis methodology suitable for initiation of detailed design and for licensing interactions. Conduct studies of fuel management strategies for the closed fuel cycle, including physics and economic impacts of self-sufficient uranium start-up versus maximized ratio start-up of sequential plant modules. Emphasize actinide self-consumption in the IFR metal fuel cycle to support the overall IFR waste management strategy.

Keywords: Actinides, Reactor Design, Metal Fuel

271. Fuel Cycle Demonstration FY 1992
$5,600,000

DOE Contact: Sol Rosen, (301) 903-3218
ANL Contact: M. J. Lineberry, (208) 533-7434

Provide for start-up of fuel cycle comprehensive remote operations demonstration including completion of equipment in FCF and start-up with irradiated fuel. Proceed to fabrication of first recycled U-Zr fuel assembly. Quantify the ultimate fuel cycle economics through development of commercial fuel cycle facility design and cost estimates.

Program will continue equipment development activities for remotized in-cell application, including reusable mold concept for injection-casting furnace, semi-automated pin processor, engineering-scale pyroprocessing equipment, etc. Refine and update commercial-scale fuel cycle facility design and cost estimates, including sensitivities to throughput requirements and develop initial set of prototype equipment systems.

Keywords: Fuels, Injection-Casting, Pyroprocessing, Metal Fuel Cycle, Remotized

272. ALMR Technology R&D FY 1992
$1,000,000

DOE Contacts: Philip B. Hemmig, (301) 903-3579
ANL Contact: Yoon I. Chang, (708) 252-4856

Comprehensive inherent safety testing with EBR-II, including incorporation of advanced reactor control technology and diagnostics systems, to demonstrate the passive safety aspects of the ALMR. Test and analysis support for ALMR mechanical components such as electromagnetic sodium pumps, auxiliary cooling systems, etc.

Keywords: Inherent Safety, Control Technology, Passive Safety, Sodium Pump

273. Program Management FY 1992
$400,000

DOE Contacts: Philip B. Hemmig, (301) 903-3579
ANL Contact: Yoon I. Chang, (708) 252-4856

Provide management for the ANL-LMR programs.

Keywords: Management

Office of Isotope Power Systems

Office of Special Applications

The Office of Isotope Power Systems, Office of Special Applications responsibilities include the development, system safety and production of radioisotope powered thermoelectric generators (RTG) and dynamic power systems for NASA and DOD space and terrestrial applications and advancing base technologies for these power systems. Thus, applied materials research programs are supported in the areas of thermoelectric materials and devices, high temperature heat source materials, materials systems compatibility and safety related materials characterization and testing.

Materials Preparation, Synthesis, Deposition, Growth or Forming

274. Development of Improved Thermoelectric Materials for Space Nuclear Power Systems — FY 1992 $300,000

DOE Contact: W. Barnett, (301) 903-3097
Iowa State University, Ames Laboratory Contact: B. Cook, (515) 294-9673

The prime objective of this program is to apply and exploit the capabilities of the mechanical alloying process for the development of improved performance Si-Ge type thermoelectric materials. The goal or target properties are average Figure of Merits, Z of 0.8 and 1.2 x 10^{-3}/°C over the temperature range 300 to 1000°C for "P" and "N" type materials, respectively.

During 1992 major emphasis was continued to be in place on the optimization of mechanical alloying process parameters. Techniques were continued to be developed to produce relatively low oxygen product. Methods were developed to produce both "N" and "P" type Si-Ge material exhibiting thermoelectric properties equal or slightly superior to that of standard Si-Ge materials. Techniques for adding nano-size stable second phase particulates continue to be explored. Mechanical alloying continues to show excellent promise.

Keywords: Mechanical Alloying, Consolidation of Powder, Powder Synthesis, Semiconductors, Thermoelectrics

275. Development of an Improved Process for the Manufacture of DOP-26 Iridium Alloy Blanks and Exploratory Alloy Improvement Studies FY 1992
$1,075,000

DOE Contact: W. Barnett, (301) 903-3097
RNL Contact: E. P. George, (615) 574-5085

An iridium alloy, DOP-26 (i.e., Ir-0.3 wt.% W with Th and Al dopant additions), serves as the fuel clad or capsule material for isotope heat sources employed in recent and contemporary space power systems for NASA deep space missions. This program is aimed at the development of an improved process route for the production of DOP-26 iridium alloy sheet, namely a consumable vacuum arc cast/extrusion/"warm" rolling route. Thermomechanical process parameters shall be optimized with respect to uniformity of product grain morphology, high strain rate, high temperature ductility and formability. The product must meet or exceed existing DOP-26 iridium alloy specifications.

The new sheet process has proven successful and has been accepted for DOP-26 iridium alloy production. Goal improvement in yield has been demonstrated. New process sheet exhibits properties equivalent or somewhat superior to the prior process, namely grain growth, formability, high temperature high strain rate ductility.

Studies of cerium and boron as alternate iridium alloy doping agent were continued.

Keywords: Consumable Arc Melt, Extrusion, Noble Metal

276. Carbon Bonded Carbon Fiber Insulation Manufacturing Process Development and Product Characterization FY 1992
$230,000

DOE Contact: W. Barnett, (301) 903-3097
ORNL Contact: C. E. Weaver, (615) 574-9978

Carbon-bonded carbon fiber (CBCF) type thermal insulation material is employed in Isotopic General Purpose Heat Source (GPHS) Module assemblies for use in current GPHS-RTG (radioisotope thermoelectric generator). The GPHS RTGs power the spacecraft for the NASA Galileo and NASA/ESA Ulysses missions and will be employed for the 1995-1997 NASA Cassini missions. This CBCF process development program is intended to accommodate a replacement carbon fiber (present specified fiber is no longer available), improve process controls, and optimize process parameters. The product shall meet prior flight quality CBCF specification. Product characterization shall include chemical purity, density, compressive strength, and thermal conductivity. Characterization studies were focused ono density, uniformity and thermal diffusivity measurements over the range, 25°C to 2000°C.

Keywords: Insulators/Thermal, High Temperature Service, Fibers

Materials Properties, Behavior, Characterization or Testing

277. Characterization of State-of-the-Art Improved Silicon-Germanium Thermoelectric Device/Materials

FY 1992
$50,000

DOE Contact: W. Barnett, (301) 903-3097
Iowa State University Contact: B. Cook, (515) 294-9673

This program is concerned with the evaluation and characterization of state-of-the-art Si-Ge/GaP and other "improved" silicon-germanium type thermoelectric materials. Also, the compatibility of materials, both current and candidates, employed in the manufacture of the multicouple (i.e., glass bonded close packed arrays of silicon-germanium couples) device are to be studied. Advanced "N" and "P" materials produced by General Electric (now Martin Marietta) were characterized.

Keywords: Semiconductor, Thermoelectrics

278. Development of an Improved Carbon-Carbon Composite Graphite Impact Shell Replacement Material

FY 1992
$300,000

DOE Contact: W. Barnett, (301) 903-3097
Oak Ridge National Laboratory Contact: G. R. Romanowski, (616) 574-4838

The Graphite Impact Shell (GIS), a component of the General Purpose Heat Source isotopic heat source module is a closed end/capped tubular shape machined from AVCO 3D-CC fine weave pierced fabric material. It is anticipated that a change in the fiber reinforcement architecture from the current orthogonal structure to a cylindrical type structure will enhance energy absorption in high velocity impact. The current program is a feasibility study of commercially available materials.

During 1991 two different reinforcement architectures were selected, procurement of test materials completed and evaluation was initiated.

Keywords: Composites, Carbon-Carbon

Office of Naval Reactors

The Materials Research and Development Program is in the Reactor Materials Division under the Deputy Assistant Secretary for Naval Reactors. The program supports the development and operation of improved and longer life reactors and pressurized water reactor plants for naval nuclear propulsion.

The objectives of the materials program is to develop and apply in operating service materials capable of use in the high power density and long life required of naval ship propulsion systems. This work includes irradiation testing of reactor fuel, poison, and cladding materials in the Advanced Test Reactor at the Idaho National Engineering Laboratory. This testing and associated examination and design analysis demonstrates the performance characteristics of existing materials as well as defining the operating limits for new materials.

Corrosion, mechanical property, and wear testing is also conducted on reactor plant structural materials under both primary reactor and secondary steam plant conditions to confirm the acceptability of these materials for the ship life. This testing is conducted primarily at two Government laboratories—Bettis Atomic Power Laboratory in Pittsburgh and Knolls Atomic Power Laboratory in Schenectady, New York.

One result of the work on reactor plant structural material is the issuance of specifications defining the processing and final product requirements for materials used in naval propulsion plants. These specifications also cover the areas of welding and nondestructive testing.

Funding for this materials program is incorporated in naval projects jointly funded by the Department of Defense and the Department of Energy. This funding amounts to approximately $105 million in FY 1992 including approximately $58 million as the cost for irradiation testing in the Advanced Test Reactor. The Naval Reactors contact is Robert H. Steele, (703) 603-5565.

OFFICE OF CIVILIAN RADIOACTIVE WASTE MANAGEMENT

Office of Civilian Radioactive Waste Management - Grand Total	$700,000
Materials Properties, Behavior, Characterization or Testing	$600,000
Waste Packages	$600,000
Device or Component Fabrication, Behavior or Testing	$100,000
Defense Waste Canisters	$100,000

OFFICE OF CIVILIAN RADIOACTIVE WASTE MANAGEMENT

Materials research is ongoing in the Office of Civilian Radioactive Waste Management in two areas: the development of canisters for containing the glass waste from the Defense Waste Processing Facility at the Savannah River Laboratory and the design of waste packages for eventual geologic disposal.

Materials Properties, Behavior, Characterization or Testing

279. Waste Packages — FY 1992 $600,000

DOE Contact: Diane Harrison, (702) 794-7275
Babcock and Wilcox Contact: Hugh Benton, (702) 794-1891

A modest effort is underway on the design of the waste packages to be emplaced on the geologic repository. Materials testing of candidate metal barriers include planned tests at 50-250°C in humid environments. A survey of degradation modes for ferrous materials is also underway. Waste package design and the fabrication/closure development studies are now in progress.

Keywords: Waste Packages, Ferrous Metals, Geologic Repository

Device or Component Fabrication, Behavior or Testing

280. Defense Waste Canisters — FY 1992 $100,000

DOE Contact: M. Scott Higgenbottom, (803) 557-1094
Westinghouse Contact: John Harbour, (803) 725-8725

Canisters for containing the glass waste from the Defense Waste Processing Facility at the Savannah River Laboratory (SRL) are presently being produced by a conventional rolled and welded fabrication process for the cylindrical center section of the canister. The canister is completed by welding a top head and a bottom piece to the ends of the center section and a nozzle to the top head. Alternative fabrication processes are being investigated, including a deep-drawn canister and a spin-forged canister.

Two deep-drawn prototype canisters fabricated by Norris Division of NI industries will be delivered to SRL shortly. The fabrication process begins with a forged cup of A314-88 304L stainless steel (0.75% max Si) that is deep-drawn to obtain canister dimensions of 118 inch length by 2 feet OD with a wall thickness of 3/8 inch (1/2 inch on the bottom). A nozzle section is welded to the top. Testing of these canisters will involve filling with glass (at 500°C) followed by dimensional measurements, drop testing (bottom and angled top drops), metallographic examination and mechanical tests.

Two spin-forged canisters are about to be ordered by SRL from Spin Forge Inc. The process begins with a forged and machined preformed cup using A336 304L stainless steel, and has the capability of producing a totally seamless canister to the same dimensions as above. Tests similar to those described above are planned for these canisters. The FY92 work under this area was confined to planning activities.

Keywords: Waste Canisters, Glass Waste, Testing

OFFICE OF DEFENSE PROGRAMS

	FY 1992
Office of Defense Programs - Grand Total	$78,597,000
The Weapons Research, Development and Test Program	$78,817,000
Sandia National Laboratories	$45,696,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 5,758,000
Adhesion and Characterization of Diamond Films on Tungsten	211,000
Ceramic-Filled-Glass Composite Densification	344,000
Diamond-Like Carbon Films for Protected Volumes	443,000
Dielectric and Ferroelectric Properties of PZT Thin Films	442,000
Dopant and Firing Effects on Decoupling Capacitor Performance	144,000
Nucleation and Growth of Metastable Ceramic Films	175,000
Sputtering of Varistor Films	20,000
Surface Chemistry of Silicon Carbide Relevant to CVD	581,000
Development and Characterization of Aluminum and Aluminum-Composite Coatings for Use as Laser-Driven Flyer Plates	378,000
Fundamental Electrochemical Studies of Al-Based Intermetallic Precipitate Phases and Their Relevancy to Localized Corrosion of Al-Alloys	102,000
High Energy and Large Flux Ion Beams for Surface Modification	58,000
Removal of Copper from Aluminum Alloy Surfaces by Chemical Dissolution	484,000
Synthesis of Palladium Powder	295,000
Chemical Modification of Buckminsterfullerene, C_{60}	80,000
Development of Environmentally-Sound Removable Encapsulants	47,000
MDA-Free Epoxy Encapsulant Formulations	285,000
New Environmentally Conscious Encapsulant	38,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Sandia National Laboratories (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Nanoscale Engineering of Smart Membranes	102,000
Nonlinear Optical Chromophores: Design and Synthesis	316,000
Tailoring Interfaces with Block Copolymers	213,000
Advanced Dielectrics for Passivation of InSb	300,000
Chemical Vapor Deposition of Copper	150,000
New Group IV Semiconductors: $Si_{1-y}C_y$ and $Si_{1-x-y}Ge_xC_y$ Materials	130,000
Quantum Confinement and Light Emission in Silicon Nanostructures	220,000
The Growth of InSb Using Tris-Dimethyl Aminoantimony or Tertiarybutyldimethylantimony and Trimethyl Indium	100,000
Visible Light Emission from Porous Silicon Nanostructure	100,000
Materials Structure and Composition	$1,902,000
Silicon Nitride Defect Centers	118,000
Adsorbate Electronic Structure	130,000
Nanocluster Electronic Structure	250,000
Numerical Methods for Determining Interstitial Oxygen in Silicon	200,000
Silica/Silicon Interface Structures	120,000
Simulation of 2-D RF-Driven Plasmas	542,000
Theory and Simulation of RF-Driven Plasmas	542,000
Materials Properties, Behavior, Characterization or Testing	$12,962,000
Optical Absorption and Electrical Properties of Ferroelectric Ceramics	73,000
Radiation and Diffusion Effects in Glass	1,755,000
The Effects of Hydrogen and Radiation Exposure on the Fracture Structure of Glasses	581,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Sandia National Laboratories (continued)	
Materials Properties, Behavior, Characterization or Testing (continued)	
Environmental Effects on the Thermomechanical Fatigue of Solders for Electronic Applications	102,000
Intermetallic Compound Growth (IMC) in Solder Joints	45,000
Liquid Metal Surface Studies	269,000
Metal Alloys for Hydrogen Storage and Handling	153,000
Metallurgical Evaluation of A356 Castings	200,000
Post Irradiation Examination of New Production Reactor Fuels	34,000
Solid Film Lubricants for WR Applications	121,000
Stockpile Support Capabilities	3,135,000
Studies on Fe-29Ni-27Co (Kovar) Alloy	566,000
Wetting and Mechanical Behavior of Interfacial Intermetallics in Solder Joints	102,000
Development of Improved Methods for Predicting and Validating Polymer Lifetimes in Weapon Environments	536,000
Development of Solid Polymer Electrolytes for Lithium Rechargeable Batteries	155,000
Electromechanical Gels	133,000
Electronic Properties of Organic Materials	626,000
Electrorheological Fluids	241,000
Electrostatics of Electrorheological Fluids	97,000
Environmental O-Ring Seals—Properties, Aging and the Argon Method	854,000
Field Enhancements Associated with Conductive Protrusions	117,000
Moisture Absorption by Molded Desiccant	1,959,000
Resist Characterization for Soft X-ray Projection Lithography	91,000
Tensile Properties of Epoxy Encapsulants	992,000
Viscoelasticity of Crosslinked Polymers	25,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Sandia National Laboratories (continued)	
Device or Component Fabrication, Behavior or Testing	$12,772,000
Failure Analysis of the SA3694 Leaded Chip Thermistor	115,000
Characterization of Quartz Resonators Having Multiple Nonpiezoelectric Layers	158,000
Decoupling Capacitors Using Ferroelectric Films	4,000,000
Diamond Substrates for Hybrid Circuits and Multichip	120,000
Evaluation of Hermetic Packaging in Optoelectronic Devices	213,000
Advanced Exclusion Barriers Study	1,213,000
Brazing of Ferralium 255 Safing Wheels	365,000
Development of Diffusion Bonding Processes	68,000
FastCast	1,106,000
Fusion Welding of Advanced Borated Stainless Steels	131,000
Fusion Welding of Fe-29Ni-17Co Alloys	116,000
Inertial Confinement Fusion (ICF) Target Fabrication	242,000
Laser Beam Welding Calorimetry Studies	300,000
Palladium Cleaning	345,000
Plasmatron Improvement Program Materials Selection Activities	57,000
Solder Joint Assessment of Resonator Packages	15,000
Solvent Substitution for Heather Parts	25,000
Thin Film Solder Conductor as Weak Link Device	100,000
Conductive Adhesives	993,000
Design and Fabrication of ICF, X-ray Laser, and Hohlraum Targets	241,000
MDA-Free Printed Wiring Boards	993,000
Materials Substitution for Transparent Armor	20,000
Patterned Metallization of Teflon	100,000
Plasma Cleaning	100,000
Polymeric Optical Waveguide Sensors Integrated with Active GaAs Devices	296,000
Rocket Propellant Removal and Size Reduction by Cryocycling	449,000
Smart Materials and Structures	652,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Sandia National Laboratories (continued)	
Device or Component Fabrication, Behavior or Testing (continued)	
Stockpile Shipping Container Evaluation	31,000
Very Fine Line TaN_2 Resistors for Circuit Applications	205,000
Instrumentation and Facilities	$12,302,000
Analytical Capabilities	4,701,000
Chemometric Methods and Software	52,000
Combustion Efficiency Measurements at the Combustion Research Facility	804,000
Compatibilitiy Studies with the Energetic Liquid Components of PEX	1,119,000
Continuous Monitoring of Metal-Aerosol Emissions	422,000
Diagnostics for Steelmaking	20,000
Electron Beam Treatment of Oil Spills	42,000
Focusing X-rays Using Capillary Optics	65,000
Hazardous Materials Characterization for Weapons Dismantlement	238,000
Hydration and Optical Absorption in Implanted Alumina	120,000
Interfacial Force Microscope Development	220,000
K-Map Characterization of InGaSb Buffer Layers and InAsSb/InSb Superlattices Grown on InSb	150,000
Materials Testing for Use in Super Critical Water Oxidation of Mixed Waste	1,548,000
NMR Studies of Helium Bubbles in Aged Palladium Tritide	279,000
Non-Destructive Characterization of Porous Si by X-ray Reflectivity	220,000
Phase Identification by Electron Backscattered Kikuchi Patterns	135,000
Screened Rutherford Backscattering Cross Sections for Heavy Ion and Low Energy Backscattering Analysis	50,000
Sorting of Waste Plastics by Spectroscopy and Neural Networks	27,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Sandia National Laboratories (continued)	
Instrumentation and Facilities (continued)	
Use of Parallel Beam Optics for X-ray Diffraction Studies of Thin Films	255,000
Volatile Organic Monitor	25,000
XRD Studies of PZT Thin Films	91,000
Fundamental Studies of Electronically-Stimulated Surface Processes	150,000
Effects of Excitation Symmetry on Parallel-Plate RF Discharges	542,000
Elimination of Chamber-to-Chamber Differences in Industrial Plasma Reactors	342,000
Femtosecond Spectroscopy of Optical Materials	365,000
Particle Formation, Transport and Detection in Plasmas	320,000
Lawrence Livermore National Laboratory	$21,784,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 4,769,000
Inorganic Aerogels	200,000
Organic Aerogels	325,000
Nanostructure Laminates	264,000
Sol Gel High Reflectors	325,000
Modified Phosphate Laser Glass Composition	400,000
KDP Growth Development	500,000
Advanced Finishing Development	700,000
Synthesis of Energetic Molecules	500,000
Doped Polymers for ICF	500,000
Plasma Polymer Coating Development	900,000
Polymer Films by RF Sputtering	125,000
Excimer Laser Micromachining	30,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Lawrence Livermore National Laboratory (continued)	
Materials Structure and Composition	$ 617,000
Electronic Structure and Dynamic Properties in Superconducting Oxides	500,000
Electronic Structure Study of the Thermodynamic and Mechanical Properties of Al-Li Alloys	117,000
Materials Properties, Behavior, Characterization or Testing	$ 7,078,000
Synchrotron Radiation-Based Materials Science	700,000
Structural Transformation and Precursor Phenomena	230,000
Underwater Explosive Energetics	50,000
Composite Explosive Energetics	150,000
Low Vulnerability Composites Explosives (LOVEX)	200,000
Fundamentals of Explosive Vulnerability	1,000,000
Very High Energy Density Materials	100,000
Liquid Actinide Embrittlement of Refractory Metals	120,000
The Formation of Bands of Ultrafine Particles During Rapid Solidification	67,000
Failure Characterization of Composite Materials	330,000
Modeling Superplastic Materials	260,000
Interfaces, Adhesion and Bonding	500,000
Laser Damage: Modeling and Characterization	500,000
KDP Characterization	300,000
Damage Testing	600,000
Transferable Insensitive High Explosives	1,500,000
Energy Transfer Dynamics in Energetic Materials	300,000
Processing-Structure-Property Correlations in Laminated Metal Composites	171,000

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Lawrence Livermore National Laboratory (continued)	
Instrumentation and Facilities	$ 9,320,000
Scanning Tunneling Microscopy (STM) and Atomic Force Microscope (AFM) as a Detector	320,000
Decontamination and Waste Treatment Facility (DWTF)	(9,400,000)*
Pyrochemical Plutonium Process Development	9,000,000
Los Alamos National Laboratory	$11,117,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$ 4,575,000
Actinide Processing Development	1,350,000
Plutonium Oxide Reduction	150,000
Low-Density, Microcellular Plastic Foams	200,000
Physical Vapor Deposition and Surface Analysis	300,000
Chemical Vapor Deposition (CVD) Coatings	150,000
Polymers and Adhesives	430,000
Tritiated Materials	175,000
Salt Fabrication	750,000
Slip Casting of Ceramics	90,000
Plasma-Flame Spraying Technology	300,000
Rapid Solidification Technology	500,000
Bulk Ceramic Processing	150,000
Synthesis of Ceramic Coatings	30,000

*Line-item construction project. It is not included in subtotal or total.

OFFICE OF DEFENSE PROGRAMS (Continued)

	FY 1992
Los Alamos National Laboratory (continued)	
Materials Structure or Composition	$ 1,237,000
Actinide Surface Properties	700,000
Neutron Diffraction of Pu and Pu Alloys and Other Actinides	237,000
Surface, Material and Analytical Studies	300,000
Materials Properties, Behavior, Characterization or Testing	$ 2,375,000
Mechanical Properties of Plutonium and Its Alloys	450,000
Phase Transformation in Pu and Pu Alloys	450,000
Plutonium Shock Deformation	350,000
Non-Destructive Evaluation	550,000
Powder Characterization	50,000
Shock Deformation in Actinide Materials	200,000
Dynamic Mechanical Properties of Weapons Materials	225,000
Dynamic Testing of Materials for Hyper-Velocity Projectiles	100,000
Device or Component Fabrication, Behavior or Testing	$2,930,000
Radiochemistry Detector Coatings	200,000
Target Fabrication	1,500,000
Filament Winder	30,000
High Energy Density Welding in Hazardous Environments	800,000
Uranium Scrap Conversion and Recovery	400,000

OFFICE OF DEFENSE PROGRAMS

Summaries of materials activities which were selected to present the diversity of materials research, development and application projects conducted for the Office of the Assistant Secretary for Defense Programs are included in this section. Activities are organized in groupings that indicate the Defense Program Laboratory at which the specific project was performed. Funds for FY92 materials activities within Defense Programs were provided by the Weapons Research, Development and Test program including the Core Research and Development program and the Technology Transfer Initiative program and by the Inertial Confinement Fusion program, the Production and Surveillance program, and Laboratory Research and Development program. Projects with proprietary, patentable, or classified information were not reported.

The Weapons Research, Development and Test Program $78,817,000

Sandia National Laboratories

Sandia's materials program is coordinated through the Materials Science and Technology Council. This council develops the strategies and business plans to assure the ability of the Laboratories to fulfill its missions for the Department of Energy.

The mission of the Engineered Materials and Processes program at Sandia is to deliver reliable, comprehensive, integrated solutions to meet the materials and process needs of our customers. Teaming within Sandia and partnering with industry, other labs, and academia, we perform research development and applications engineering for DOE, other government agencies, and U.S. industry.

Materials Preparation, Synthesis, Deposition, Growth or Forming

281. Adhesion and Characterization of Diamond Films on Tungsten FY 1992
$211,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Diane E. Peebles, (505) 845-8087

Hot-filament CVD diamond films deposited on tungsten have been studied to determine the correlation between processing parameters and the quality and adhesion of the diamond films. Diamond and diamond-like-carbon (DLC) display unique combinations of properties that are potentially useful for a wide variety of applications in the fields of friction and wear, thermodynamics and optics. In practice, problems are encountered which impede efforts to make useful, commercially successful products employing diamond and DLC coatings. Potential problems include: surface roughness, poor adhesion, high intrinsic stresses, slow deposition rates, high substrate temperatures, nonuniformity, etc. We have been exploring the relationship between the properties of diamond coatings and the deposition parameters, contributing to the basic understanding of the parameters affecting coating characteristics. In particular, those parameters which influence the coating adhesion to the substrate and the quality of the deposited diamond films are being examined. Diamond films have been deposited on tungsten substrates by a hot filament-assisted CVD process.

Keywords: Ceramics, Diamond, Surface, Coating, CVD

282. Ceramic-Filled-Glass Composite Densification FY 1992
$344,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Kevin G. Ewsuk, (505) 845-8492

Sintering models have been developed that can provide useful design and process guidelines for manufacturing advanced ceramic composites. Ceramic composites are becoming increasingly important advanced materials as they provide the materials engineer a new class of materials with unique properties that can be tailored to specific applications. One of the major obstacles to the commercialization of ceramic composites is that composite densification is difficult to control. A better understanding of the fundamentals of composite sintering is required reproducibly to manufacture reliable composites. Through a combination of modelling and experimentation, it has been determined that low-temperature-sintering (i.e., ≤1000°C), ceramic-filled-glass (CFG) composites densify by a combination of glass redistribution, grain rearrangement, and viscous flow in a three-stage process described as non-reactive, liquid-phase sintering (NLPS). For reproducible densification, the final stage of sintering is the most critical. Under ideal conditions, CFG composite densification during final-stage NLPS is controlled by a combination of pore size, CFG composite dispersion viscosity, and glass surface tension, and can be modeled using modified viscous sintering theory.

Keywords: Ceramics, Composite, Modeling

283. Diamond-Like Carbon Films for Protected Volumes FY 1992
$443,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Edward McKelvey, (510) 294-2326

Diamond-like carbon films have been deposited on plastics, polycarbonates, and aluminum. The films are stable to air exposure and are relatively uniform. Because of its unique properties, diamond-like carbon (DLC) films have been identified as good candidates for use in protected volumes of weapons systems. Our research includes identifying the problem areas, identifying customers, and performing initial proof-of-principle tests. At the start of the project a study was done to determine if there is a good match between DLC technology and the needs of protected volumes. Among the many interesting properties of DLC, the study identified low chemical reactivity, high mechanical hardness, the ability to coat large areas as the most noteworthy. Furthermore, the study also pointed out the ability to deposit at near-room temperature to be a key advantage over the process for depositing crystalline diamond. Several proof-of-principle tests were conducted in which DLC was coated on polycarbonates, polystyrene, aluminum, and anodized aluminum. With this knowledge in hand the team was able to attract the interest of several protected volume customers who are willing to participate in the research and provide design guidance.

Keywords: Ceramics, Diamond, Membrane, Film, Coating

284. Dielectric and Ferroelectric Properties of PZT Thin Films — FY 1992 $442,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Robert W. Schwartz, (505) 844-3532

PZT thin films with varied PZ-PT ratios were fabricated by solution deposition. Excellent dielectric and ferroelectric properties were obtained for a range of compositions. PZT thin films are under investigation at Sandia for applications ranging from decoupling capacitors to nonvolatile memories and optical storage media. Most investigations, both at Sandia and externally, have focused on compositions near the morphotropic phase boundary (PZT 53:47). PZT thin films of other compositions are potentially required for other applications or may be more suitable for the applications presently under consideration. The purpose of this study was to prepare PZT thin films with lead zirconate to lead titanate ratios varying from 30:70 to 70:30 and to compare the dielectric and ferroelectric properties of these films to the reported results for bulk PZT ceramics. The PZT films displayed both interesting differences and similarities to results reported for bulk PZT ceramics. As with the bulk materials, the dielectric constants of the thin films were greatest, and coercive fields were lowest, for PZT compositions near the morphotropic phase boundary. Also as with bulk PZT ceramics, ferroelectric hysteresis loops became more square for compositions with larger amounts of lead titanate.

Keywords: Ceramics, Ferroelectric, Sol Gel, Film, Dielectric

285. Dopant and Firing Effects on Decoupling Capacitor Performance — FY 1992 $144,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Robert W. Schwartz, (505) 844-3532

Donor dopant incorporation and novel heat-treatment conditions yield PZT thin films with improved high temperature leakage characteristics. PZT thin films are of great interest to Sandia for on-chip applications such as decoupling capacitors. One critical performance criterion that must be met by our thin films for this application is the high temperature (T ~ 125°C) leakage characteristics of the films. Films that display < 1 nA current flow at these temperatures are desired. The films that we are currently fabricating are acceptable in this regard, but further improvements in leakage current characteristics and long term capacitor stability are still desirable. We have investigated two approaches to minimizing the leakage currents of our solution deposited PZT thin films. Since leakage behavior is potentially related to the point defect nature of the films, we have studied the effects of incorporating small additions of donor dopants, e.g., Nb or La, in the films. These dopants should impact the defect nature of the film, and therefore, potentially, film leakage characteristics. Dopant additions may impact film microstructure, which may also serve to define leakage behavior.

Keywords: Ceramics, Dielectric, Sol Gel, Film, Ferroelectric

286. Nucleation and Growth of Metastable Ceramic Films — FY 1992 $175,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Thomas M. Mayer, (505) 844-0770

Oriented growth of h-BN crystalline films on Ni (111) and (100) substrates has been demonstrated by thermal and plasma assisted CVD of borazine. We are investigating growth of crystalline ceramic thin films by low temperature CVD processes. The long term goal of the project is to control the nucleation and growth of metastable phases of various nitride, carbide, and other refractory ceramic films. Boron nitride (BN) was chosen as an initial target material because of the attractive properties of the metastable cubic phase. We have investigated a wide range of processing parameters using thermal and microwave ECR plasma assisted CVD techniques. By analogy to diamond film deposition processes, we have emphasized concepts of hydrogen passivation of the growth surface, and promotion of cubic phase formation by ion bombardment To date we have not seen evidence of cubic phase formation for any of the process variations studied. This appears due to rapid chemical etching of deposited material by H, and inefficient momentum transfer from the largely H containing ions in the plasma. Most deposited films are amorphous in nature with sp^2 bonding.

Keywords: Ceramics, Nitride, Spectroscopy, Film, CVD

287. Sputtering of Varistor Films — FY 1992 $20,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Terry J. Garino, (505) 845-8762

Thin film ZnO-based varistors are being fabricated by RF sputtering for a classified application. Sputtered thin film varistors are being developed for devices that have switching voltages around 10 volts, are able to withstand heating to moderate temperatures, 500-1000°C, without significant performance degradation, and are able to repeatedly withstand voltages above the switching voltage, again without serious degradation. RF sputtering is being used to fabricate these devices on metal electrodes. Initially, bi-layer devices were made by sputtering a bismuth oxide layer on top of a semiconducting zinc oxide layer. These devices exhibited varistor-like electrical behavior without any post-sputtering heat treatment. The switching voltage could be controlled by the sputtering conditions (Ar pressure and time) used during Bi_2O_3 sputtering and was in the desired range. However, both mild heating and repeated high voltages greatly increased the leakage current of these devices. Both of these effects are related to the destruction of the space charge present due to defects in the Bi_2O_3 layer. Because of these problems, a second approach is being pursued; this involves sputtering a multicomponent film from a single target which has a composition typical of a bulk varistor.

Keywords: Ceramics, Varistor, Film

288. Surface Chemistry of Silicon Carbide Relevant to CVD — FY 1992 $581,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Duane Outka, (510) 294-1311

The reactivity of a silicon carbide surface toward gases used in CVD deposition was examined to support CVD modeling and predict rate-limiting reactions in CVD. Silicon carbide (SiC) is of interest for a variety of applications including as a structural ceramic, as a high temperature wear and oxidation resistant coating, and as a wide band-gap semiconductor. SiC is typically deposited by CVD from a gas phase reaction mixture onto a heated substrate. Computational models of the CVD process are in development to aid the scale-up and optimization of SiC deposition. These models require detailed information about the gas-phase and surface kinetics of the varies species in the CVD reactor. While several studies have examined the issue of gas-phase chemistry of SiC precursors, little previous work has been reported on the surface chemistry of SiC. The global reaction for depositing SiC of interest to this work is

$$CH_3SiCl_3 \xrightarrow{H_2} SiC + HCl$$

This study explores the surface reactivity of methyltrichlorosilane (MTS) and hydrogen chloride (HCl) on SiC.

Keywords: Ceramics, CVD, Modeling, Coating, Carbide

289. Development and Characterization of Aluminum and Aluminum-Composite Coatings for Use as Laser-Driven Flyer Plates

FY 1992
$378,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Dale C. McIntyre, (505) 272-7621

A process has been developed (with collaborators) to deposit aluminum and multilayer aluminum/aluminum oxide films. The microstructure of the films has been characterized using SEM, TEM, and XTEM. Laser-driven aluminum and aluminum-composite flyer plates are being utilized in the Direct Optical Ignition (DOI) program at Sandia National Laboratories in New Mexico. The flyer plate assemblies are constructed by attaching (using epoxy) an optical fiber to a stainless steel ferrule and to the rear body of an optical fiber connector. The assemblies are mechanically polished. Vacuum evaporation is used to coat the polished end of the fibers and ferrules with an aluminum film or aluminum/ aluminum oxide multilayer film. Optimum performance of the flyer plates can only be achieved if the coatings have excellent adherence to the flyer plate assemblies. Early in FY92, the adherence of the coatings was poor and flyer performance was inadequate. In FY92, we made a detailed evaluation of the baseline process for producing and coating flyer plate assemblies. We made specific recommendations for improvements in assembly cleaning and handling and provided analytical support to help evaluate the condition of the assemblies at different stages in the production process.

Keywords: Metals, Coating, Microstructure, Microscopy

290. Fundamental Electrochemical Studies of Al-Based Intermetallic Precipitate Phases and Their Relevancy to Localized Corrosion of Al-Alloys

FY 1992
$102,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Rudolph G. Buchheit Jr., (505) 844-6904

We have synthesized a variety of Al-based intermetallic compounds in bulk form for study by conventional electrochemical and surface analytical techniques. Results obtained have been used to understand the role of finely dispersed precipitate particles. The solid solubility of most metals in aluminum is low; usually less than 1.0 wt.%. As a result, Al-alloys typically contain a variety of submicrometer intermetallic particles. These particles can serve as initiation sites for corrosion during aqueous processing and cleaning procedures, or during exposure to service environments. Although it is widely known that these particles induce local attack, the exact role of these particles (e.g., selective dissolution or galvanic attack) in the process is not well understood. Thorough understanding of the electrochemical behavior of intermetallic compounds will enable the development of methods for mitigating localized corrosion. In this project, we seek to define the electrochemical behavior of several *intermetallic compounds and apply this information to the development of corrosion mitigation* schemes for several important engineering applications.

Keywords: Metals, Corrosion, Surface, Film, Cleaning, Alloy

291. High Energy and Large Flux Ion Beams for Surface Modification — FY 1992 $58,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Dale C. McIntyre, (505) 272-7621

A surface modification technique that uses Repetitive High Energy Pulsed Power (RHEPP) technology is being developed. This technique can be used to enhance the wear resistance and corrosion resistance of materials. We are developing a source capable of modifying the near-surface regions of materials by exposing them to pulses (60 ns duration) of high energy (≈ MeV) ions in ion-beams with energy fluxes of approximately 10 J/cm^2. Energy pulses of this magnitude are sufficient to cause rapid melting and rapid cooling (rates of 10^{10} K/sec) in the top several microns of most materials. The process of rapid melting and cooling of the surfaces of materials can be used to produce amorphous surface layers or to provide a mechanism for surface alloying. These materials modifications may in turn increase the wear and corrosion resistance of many materials. Personnel in 1200 are working cooperatively with personnel from Cornell University to develop a Repetitive High Energy Pulsed Power (RHEPP) source capable of producing high energy/large flux ion beam bursts at rates of 120 Hz. These sources will be capable of treating materials at 1/100th of the cost of competitive technologies for surface melting (laser glazing, electron beam melting, etc.).

Keywords: Metals, Surface, Microscopy, Diffraction

292. Removal of Copper from Aluminum Alloy Surfaces by Chemical Dissolution — FY 1992 $484,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: LeRoy L. Whinnery, (510) 294-1215

This project is directed at removal of copper from the surface of aluminum alloys using chelating agents prior to applying a conversion coating. It is believed this will improve the performance of coatings being considered to replace chromium coatings. Chromate conversion coatings applied to aluminum alloys are widely used in the plating community within the DOE complex and industry. Conversion coatings serve several purposes: (1) corrosion resistance; (2) slight conductivity; (3) easily touched-up in the field; (4) colored; and (5) self-healing properties. Several kinds of coatings are currently being pursued at Sandia to replace these chromium (a carcinogen) containing coatings. A common dilemma with these new replacement coatings is their lack of corrosion resistance on copper containing aluminum alloys, for example 2024 and 7075. Copper is present in aluminum alloys to provide additional strength. It is generally believed that copper precipitates on the surface of aluminum result in localized galvanic corrosion.

Keywords: Metals, Corrosion, Surface, Coating, Alloy

293. Synthesis of Palladium Powder — FY 1992 $295,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Don Meeker, (510) 294-2817

Basic parametric studies of the conditions necessary to produce palladium powders of desired characteristics have been conducted. These studies have led to an easily scalable precipitation procedure. We are interested in precipitated palladium powder for use as a catalyst and as a medium for solid storage of hydrogen. In these applications, the material properties and purity of the palladium powder are critical, and must be reproducible from one batch to another. We are conducting research to understand the fundamental processes of precipitating palladium, and to acquire the knowledge to scale up production of these materials. Over the past six months, we have produced palladium powders in laboratory-scale batches that meet or exceed the surface area and purity requirements. These experiments have provided insight into the kinetic and mass transport aspects of the precipitation reaction which, in turn, have enabled powders with highly uniform microstructure to be synthesized. This precipation technique differs from others in the literature in that it is easily scalable. This will allow much larger batch sizes to be made reproducibly without additional development. A bench-scale semi-continuous process is being designed based on the acquired knowledge.

Keywords: Metals, Precipitation

294. Chemical Modification of Buckminsterfullerene, C_{60} — FY 1992 $80,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Paul A. Cahill, (505) 844-5754

Evidence for the synthesis of $C_{60}H_2$, the simplest derivative of C_{60}, has been obtained via the reaction of borane with C_{60} followed by hydrolysis. The chemistry of C_{60}, Buckminsterfullerene which is a revolutionary new form of carbon, has scarcely been explored. As part of an LDRD project which combines theoretical and experimental investigations of C_{60}, we have begun to investigate the chemistry of this highly reactive molecule. Our research is directed toward the development of high yield syntheses of characterizable derivatives for use in the development of new materials with useful electrical, optical, or tribological properties. One of our first targets has been $C_{60}H_2$. Semi-empirical computations which we performed indicated that at most two of the 23 possible inequivalent isomers of C_{60} are likely to be present at thermal equilibrium and has spurred our interest in this molecule by suggesting that product mixtures will be characterizable. We have recently isolated a material with UV and NMR data consistent with $C_{60}H_2$ through the protonolysis of an intermediate proposed to be $C_{60}(BH2)$ H.

Keywords: Organic, Fullerene, Synthesis

295. Development of Environmentally-Sound Removable Encapsulants — FY 1992 $47,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Charles Arnold Jr., (505) 844-8728

Electronic assemblies such as firesets and radar units utilize foam encapsulants (polyurethanes and polystyrene) for mechanical support and environmental protection. Currently, recovery of these electronic assemblies requires the use of aggressive and/or toxic solvents which are harmful to the environment and can incur damage to the electronic parts. There is a need for foam encapsulants that can be easily removed with relatively non-aggressive and environmentally acceptable solvents. There is also a need to find substitutes for the hydrocarbon and chlorofluorocarbon blowing agents which are either being phased out (the chlorofluorocarbons) or stringently regulated (the hydrocarbons). These foams must also meet or exceed the property, performance and processing requirements of the current foams. Major accomplishments to date include the following: (1) demonstrated the feasibility of replacing pentane with carbon dioxide in polystyrene foam encapsulants; (2) successfully molded strong foam parts of polystyrene particles of various sizes (0.033 to 0.127 in) using carbon dioxide as the blowing agent; (3) demonstrated that polar polymers that are soluble in non-toxic solvents such as water or ethanol could be foamed using carbon dioxide.

Keywords: Organic, Encapsulant, Foam

296. MDA-Free Epoxy Encapsulant Formulations — FY 1992 $285,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Linda A. Domeier, (510) 294-2350

Neutron generators and other DOE components use epoxy encapsulants cured with MDA (methylene dianiline), an OSHA regulated carcinogen. Epoxy encapsulant formulations cured with aliphatic amines are being developed to replace the current materials. Both processing and performance advantages have been obtained. Alternative epoxy formulations using less hazardous curing agents while still meeting or improving on other requirements are needed and have been the target of an on-going multi-site program. The particular formulations developed and reported here have utilized combinations of aliphatic amine curing agents to obtain the desired balance of toughness, Tg and other processing and performance characteristics.

Keywords: Organic, Encapsulant, Adhesive

297. New Environmentally Conscious Encapsulant — FY 1992, $38,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Kenneth B. Wischmann, (505) 845-9755

A need exists to remove toxic and carcinogenic materials from the workplace. A newly developed epoxy/anhydride encapsulant is being characterized and would find application in neutron generators, magnetic devices and other electronic components. New regulations from OSHA have identified and are regulating the use of materials which contain suspect human carcinogens such as methylene dianiline (MDA) in U.S. industry and the DOE complex. A number of polymer formulations use MDA either as a curative or as part of the base resin system. It is the goal of this program to identify, characterize, qualify and introduce an epoxy encapsulant material which does not contain MDA and publicize this to industry and the DOE production agencies. An epoxy/anhydride encapsulant has been formulated which consists of 50 pbw EPON 828-CTBN, 40 pbw hexahydrophthalic anhydride, 15 pbw of a polyol flexibilizer, ARCOL 1025 and 2 pbw of EMI 24, an imidazole catalyst. To this resin mixture, the following respective fillers have been incorporated: 30 pbw glass microballoons, 300 pbw Al_2O_3, 100 pbw MICA. These three filled encapsulants have been characterized with respect to their mechanical, electrical, thermal and adhesion properties.

Keywords: Organic, Encapsulant, Polymer

298. Nanoscale Engineering of Smart Membranes — FY 1992, $102,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: C. Jeffrey Brinker, (505) 845-8930

Organic molecules are used as templates to create inorganic films with precise control of pore size and shape for applications in separations, sensors, and catalysts. The purpose of this project is the development of chemically robust inorganic membranes that mimic active biological membranes, i.e., that exhibit molecular recognition capabilities, for applications in gas or liquid separations, sensors, catalysts, selective absorbents, etc. Our approach is to embed designed organic templates in dense sol-gel thin film matrices. Removal of the templates creates within the inorganic matrix a microporous channel system that exhibits molecular recognition or molecular sieving capabilities. This project combines Sandia's expertise in sol-gel derived thin films with computer-aided molecular design and molecular recognition and catalysis capabilities. Using sol-gel processing, we synthesized and characterized a model silica matrix prepared as a thin film on both porous and solid supports. Films on porous supports were characterized by gas permeability, cross-sectional TEM, and x-ray micro-fluorescence spectroscopy. Films on solid supports were characterized by gas sorption using surface acoustic wave (SAW) techniques and ellipsometry.

Keywords: Organic, Membrane, Microporous, Biometric

299. Nonlinear Optical Chromophores: Design and Synthesis FY 1992
$316,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Paul A. Cahill, (505) 844-5754

New chromophores have been synthesized as thermally stable dyes for electro-optical applications. Second order nonlinear optical materials are of increasing interest to the microelectronics industry as a means of further increasing desktop and mainframe computer processing speeds. Such materials are the basis of high speed electrooptic switches in which an electric field is used to modulate, via a change in refractive index, waveguided light. In this reporting period, we have concentrated on the synthesis of NLO chromophores with thermal stabilities adequate to survive semi-conductor processing steps (up to and possibly beyond 300°C) and to function indefinitely at 80-100°C. Our initial approach was to investigate fluorination of known NLO chromophores as a method of increasing thermal stabilities; however, this was ineffective as a general method. We have now synthesized several donor-acceptor substituted molecules based on known and highly thermally and photochemically stable scintillator dyes. Related azole derivatives have recently been reported to have excellent thermal stabilities; we anticipate that our derivatives will have similar stabilities with high NLO figures of merit.

Keywords: Organic, Nonlinear, Polymer, Optical, Electrooptic

300. Tailoring Interfaces with Block Copolymers FY 1992
$213,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Michael Kent, (505) 845-8178

The detailed structure of block copolymers at polymer-polymer, solid-polymer, and liquid-air interfaces is being investigated with neutron reflectivity. Detailed concentration profiles have been obtained at the liquid-air interface. We are studying the behavior of block copolymers at the liquid-air interface. The liquid-air interface is a model interface which allows the crucial variable, the surface coverage, to be varied continuously. In addition, the essential physical characteristics of the layer, the polymer segment concentration profile and the free energy of interaction, can be probed in a straightforward manner by neutron reflectivity and surface tension measurements. Information obtained regarding the behavior of block copolymers at this model interface will be applicable to important industrial problems such as the use of block copolymers to a) enhance adhesion between a polymeric matrix and a solid surface, b) control the domain size in incompatible polymer blends, and c) stabilize colloidal dispersions.

Keywords: Organic, Surface, Polymer, Adhesive, Surface

301. Advanced Dielectrics for Passivation of InSb — FY 1992 $300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: J. Charles Barbour, (505) 844-5517

Novel growth processes for low-temperature synthesis of high-quality silicon oxynitrides (SiO_xN_y) and sulfidized layers are important for surface passivation of narrow-gap III-V semiconductors in order to enhance the U.S. competitiveness in the utilization of devices such as InSb infra-red detectors. A combination of electrochemical, and Electron Cyclotron Resonance (ECR) growth has yielded new processes to synthesize dielectric surface passivation layers on InSb. Composition analysis has been done using Rutherford backscattering spectrometry (RBS) and elastic recoil detection (ERD). In addition, the type of bonding within the sulfidized layers and at the InSb-sulfide interface has been examined using X-ray photoelectron spectroscopy (XPS). The quality of the passivation has been studied using 1 kHz to 1 MHz capacitance-voltage (C-V) measurements on metal-insulator-semiconductor structures. Sulfidized layers, SiO_xN_y on InSb, and sulfidized layers capped with SiO_xN_y all exhibited good electrical properties, and the passivation of side-walls in diode structures is currently being tested with each of these thin-film dielectrics. This work is the first time high-quality silicon oxynitride and sulfidized layers passivated an InSb surface, thereby meeting the major goal for this period of the project.

Keywords: Semiconductor, Compound, Plasma, Electrical Properties, Electronic Structure, Ion Beam, Chemical

302. Chemical Vapor Deposition of Copper — FY 1992 $150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: John E. Parmeter, (505) 844-8594

We have observed striking differences in the chemistry of copper precursors reacting on Pt(111) surfaces relative to copper surfaces. Due to the low resistivity and high electromigration resistance of copper, there has recently been much interest in the chemical vapor deposition (CVD) of copper for microelectronics applications. Two of the most promising families of copper CVD precursors are the Cu(I) and Cu(II) beta-diketonates, which are capable of depositing copper films of high purity under reasonably mild reaction conditions.

Investigations of the surface chemistry of these precursors are of importance in order to identify surface intermediates that may be involved in CVD reaction mechanisms, to identify possible pathways of impurity incorporation into growing copper films, and to understand the differences in reactivity of the precursors show towards different substrates. We are investigating the chemistry of the precursor molecules Cu(hfac)2 and (hfac)Cu(VTMS), and of the related hfacH molecule, on the Pt(111) surface.

Keywords: Semiconductor, Metallization, CVD

303. New Group IV Semiconductors: $Si_{1-y}C_y$ and $Si_{1-x-y}Ge_xC_y$ Materials — FY 1992 $130,000

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New Group IV semiconductor $Si_{1-y}C_y$ binary and $Si_{1-x-y}Ge_xC_y$ ternary compounds produced by ion implantation are under investigation. These binary and ternary compounds are being explored for possible applications in Si-based microelectronics and infrared detectors. We report here on $Si_{1-y}C_y$ material quality and substitutionality of C in layers regrown by solid phase epitaxy (SPE) at 700°C. To produce SPE Si, a layer of Si is made amorphous by self-ion implantation, and then implanted with 0.5 to 2 atomic percent of C before the 700°C SPE step. Ion channeling and x-ray diffraction data show excellent quality in the SPE layer for $C_y < 1.5$ atomic percent. The lattice parameter change produced by the C is in good agreement with that expected from Vegard's law and extrapolation to B silicon carbide. A vibrational mode for C detected by infrared absorption indicates the C is in substitutional sites in the SPE layers even though the C concentration is more than three orders of magnitude higher than the equilibrium solid solubility. We find this metastability is maintained through a 30 min 850°C annealing period. Carbon is lost slowly from substitutional sites at 900°C, and absorption due to silicon carbide in addition to substitutional C is observed after heating for 30 min at 950°C.

Keywords: Semiconductor, Ion Implantation, Silicon

304. Quantum Confinement and Light Emission in Silicon Nanostructures — FY 1992 $220,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Silicon nanostructures have been produced by electrochemical anodization and by He ion implantation. Changes in photoluminescence spectra have been correlated with changes in processing parameters to tailor nanostructure growth. Electrochemically formed porous silicon was reported (1991) to exhibit visible photoluminescence and thus might allow optical devices to be formed in Si-based microelectronic circuits. We have performed experiments aimed at identifying the mechanism responsible for light emission. Parallel work has involved tailoring the microstructure and chemistry of the Si to obtain photoemission that is usable for devices. We have used He ion implantation and subsequent annealing to produce microstructures (2 nm) in Si and have characterized them with cross section TEM for a range of ion fluences and annealing conditions. The structures are passivated with H to remove unwanted recombination centers. We have added photon counting capability to our optical laboratory to examine these samples for possible light emission due to quantum confinement and/or hydride related mechanisms. To date, these structures have not exhibited photoluminescence as seen for electrochemically produced porous silicon.

Keywords: Semiconductor, Silicon

305. The Growth of InSb Using Tris-Dimethyl Aminoantimony or Tertiarybutyldimethylantimony and Trimethyl Indium

FY 1992
$100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Novel organometallic sources of Sb are being investigated to improve the properties of InSb grown by metal-organic chemical vapor deposition. Alternate organometallic Sb sources are being investigated to obtain improved InSb characteristics compared to those of InSb grown using TMIn and TMSb. InSb was grown using TMIn and tris-dimethylaminoantimony (TDMASb) at temperatures of 475-515°C and V/III ratios of 1 to 25. This yielded similar quality InSb to that grown using TMIn and TMSb as indicated by Hall measurements. Lower temperature and pressure experiments using TDMASb are underway. The growth of InSb using tertiarybutyldimethylantimony (TBDMSb) and TMIn was investigated over a temperature range of 350-475°C, 5/3 ratios of 0.8 to 24, and pressures of 200 to 660 torr. Among the Sb sources investigated so far, which include TESb, TMSb, TIPSb, TDMASb and TBDMSb, the highest mobility, and smoothest surface InSb was grown using TBDMSb at T 425°C. The growth of n-type InSb using TBDMSb or TIPSb and TMIn for T 425°C supports the explanation of an intrinsic defect as the cause of the p-type carrier concentrations for InSb grown above 425°C.

Keywords: Semiconductor, CVD, Compound

306. Visible Light Emission from Porous Silicon Nanostructure | FY 1992 $100,000

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Porous silicon nanostructures formed by anodization yield visible light in the red to green wavelengths. We are correlating composition with light emission. Recent evidence that visible light emission from silicon could be achieved using porous silicon (PS) nanostructures has brought about renewed interest in the properties of this material. We are currently using electrochemical, surface analytical, and microscopic techniques to examine the properties of porous silicon and their effect on the resulting photoluminescence. For example, by varying the formation conditions of porous silicon, e.g., the current density, silicon doping type and level, and electrolyte composition, we produce samples that emit light in wavelengths corresponding to red, orange, yellow, and green. We also find that as the current density increases, all other formation conditions being equal, there is a blue shift in the emitted light. It is well known that increasing current density increases porosity and increases the average pore size; in one set of our experiments, a porosity increase from 81 to 96% produced a shift in emitted light from orange to green. We are also using *in situ* Raman spectroscopy to characterize porous silicon formation.

Keywords: Semiconductor, Etching, Silicon, Spectroscopy, Porosity, Microstructure

Materials Structure and Composition

307. Silicon Nitride Defect Centers | FY 1992 $118,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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It has been shown that the dominant deep electron and hole traps in silicon nitride thin films are the positively $(^{+}\mathrm{Si}\int\mathrm{N})$ charged and negatively charged $(^{-}\mathrm{Si}\int\mathrm{N}_3)$ Si sites, respectively. Over the past decade or two a lot of effort has gone into understanding the nature of charge trapping centers in amorphous hydrogenated silicon nitride (a-SiN_x:H) thin films. The nature of these charge trapping sites is important in predicting the memory characteristics of polysilicon-nitride-oxide-silicon devices, as well as to help eliminate the charging effects observed in a-SiN_x:H thin film transistors. We have employed electron paramagnetic resonance spectroscopy, electrical measurements, charge injection, optical excitation studies, theoretical considerations, and a wide range of processing conditions to provide strong evidence that the traps are charged, compensated, diamagnetic Si centers, and that these Si centers are negative U candidates. The negative U model simply states that the charged diamagnetic Si sites are more stable than the neutral paramagnetic Si site. It was found that a double change in charge state of pre-existing positively and negatively charged Si centers is responsible for the trapping phenomenon observed, and characteristic of, these thin film dielectrics.

Keywords: Ceramics, Dielectric, Silicon, Nitride, Electronic

308. Adsorbate Electronic Structure FY 1992
$130,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A general theory of chemisorbed molecules has been developed. It explains several previously mysterious aspects of both spectroscopy and dynamical processes such as photodesorption and photochemistry. The theory recognizes that a great simplification can be achieved if a substantial separation in timescales exists between the charge fluctuation time of the adsorbate (driven by the covalent interaction with the substrate) and the relaxation time of the substrate. If this separation is valid, adsorbates can be modeled by a semi-empirical configuration interaction model, which can also be transformed into a Hubbard-like model. Recently, we derived realistic parameters for the strength of the covalent interaction (~0.5 eV) and for the fractional charge transfer (1/4-1/2 electron) in the NO:Pt(111) system. We were then able to show that the above mentioned timescale separation existed for all choice of model parameters. This established the basic validity of the model for adsorbates with relatively weak covalent bonds—a case which includes most chemisorbed molecules on metal surfaces. (The theory does not apply to strongly chemisorbed species such as H, O, or S atoms.)

Keywords: Metals, Surface, Adsorbates

309. Nanocluster Electronic Structure FY 1992
$250,000

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Work has begun to theoretically understand the properties of metal clusters of from ~1-200 atoms. While work by others has dealt with simple-metal clusters such as alkali's, because of breakthroughs in manufacture and the possibility of revolutionary catalytic properties, our work centers on transition metal and Group IB ("coinage metal") clusters. Local density functional (LDF) calculations using a plane wave basis and pseudopotentials have been found to converge on Ag clusters but not on Pd clusters. However, the Gaussian basis function LDF code of Peter Feibelman (1114) has shown convergence on small Pd clusters. Pending the porting of Feibelman's code to the hypercube computer, work being done by Mark Sears (1422), we will use the plane-wave code to study ever-larger Ag clusters with and without adsorbates. A problem which immediately arises is the large number of geometries which are possible with the large clusters. Pending the advent of molecular dynamics LDF calculations, in which a hot cluster can be cooled to find the preferred geometry, it is necessary to explore cheaply the many possible geometries before testing likely ones with more detailed and time consuming calculations.

Keywords: Metals, Cluster, Surface, Catalyst

310. Numerical Methods for Determining Interstitial Oxygen in Silicon — FY 1992 $200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Joel O. Stevenson, (505) 845-8652

The concentration of interstitial oxygen in Czochralski silicon is an important parameter for device manufacture because oxygen impurities affect the mechanical, electrical, and gettering properties of silicon. The oxygen concentration is monitored by FTIR spectroscopy of the antisymmetric stretch vibration of the interstitial oxygen species. Numerical methods which report an ability to determine interstitial oxygen in silicon include ASTM standard test F1188-88, Short Baseline (SBL), and Curved Baseline (CBL). FORTRAN software was written that implements these three independent techniques in a single integrated package. A number of equations in the literature were shown to be very unstable over particular data regimes. A correction for multiple internal reflections was the most glaring numerical instability. Precision was also being lost in many instances. Using well-known mathematical relationships, these ill-behaved equations were reformulated to minimize numerical instabilities and to preserve accuracy that might otherwise be lost. Routine oxygen measurements are desirable over a wide range of wafer resistivities, but there has been confusion concerning which of the three methods is most suitable for the high resistivity portion of this continuum.

Keywords: Semiconductor, Silicon

311. Silica/Silicon Interface Structures — FY 1992 $120,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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We are evaluating the structure and morphology of SiO_2/Si interface structure as a result of standard IC fabrication procedures. The effects of surface morphology (roughness) on electrical properties of devices fabricated using these procedures will be evaluated. X-ray reflectivity has been developed as a suitable technique for evaluation of surface morphology. Si surface subjected to a variety of etching and cleaning procedures commonly used in IC fabrication have been examined for their effects on morphology of the Si surface. Prior reports have indicated a correlation between Si/SiO_2 interfacial roughness and the quality of electronic devices (MOS structures) fabricated on these surfaces. Roughness on the scale of <1 nm is thought to be deleterious to interface state density and carrier lifetime. We have examined a number of standard chemical cleaning procedures using x-ray reflectivity to measure the surface roughness produced. Different variations of the RCA cleaning process have shown little variation in surface roughness, with values of about 0.4 nm typical. Other more aggressive cleaning procedures are being investigated to confirm our ability to quantify and observe the effects of roughness.

Keywords: Semiconductor, Silicon, Etching, X-ray, Microstructure

312. Simulation of 2-D RF-Driven Plasmas | FY 1992 $542,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Merle E. Riley, (505) 844-3141

Most simulations of the RF-driven plasmas appropriate for processing applications are done in 1-D because of the intensive computational effort required to resolve the disparate time scales of the physical processes. In our work in 1-D, we have developed techniques that allow the time scales to be separated, with numerical solutions done iteratively for various processes. The electrons are driven by the RF field, and they create electronic excitation and ionization of the atoms. we solve this fast part of the dynamics either by a Boltzmann equation or by a particle simulation by Monte Carlo methods. The rf-cycle averaged rates are computed and used in the slower time-scale evolution. Heavy particle diffusion is one of the slower processes to equilibrate in the plasma. We solve this by using the cycle-averaged rates and an ambipolar-like diffusion for the ions in a corrected, cycle-averaged electric field. The final, periodic steady state of the plasma is found by repeating the sequence of fast scale and slow scale calculations.

Keywords: Semiconductor, Plasma, Etching

313. Theory and Simulation of RF-Driven Plasmas | FY 1992 $542,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Merle E. Riley, (505) 844-3141

Computer simulations are being developed and used to better our understanding of glow discharges used in semiconductor processing. Plasma processing is widely used in microcircuit manufacture. We have begun a quantitative research program (GEC Reference Cell) to understand the basic physics of these radio frequency (RF) driven plasmas. The system is a partially ionized, nonequilibrium, time -dependent, multicomponent, reactive plasma with interacting surfaces and applied RF. The theory consists of a 1-D Boltzmann description of the electrons, a five level model of He, a fluid description of ions and neutrals, and many numerical and physical approximations to overcome the six-order-of-magnitude variation in time scales of the important dynamical processes. We have done extensive exploratory calculations of the plasma typical of GEC Cell operating conditions. One of the most interesting of the experimental results was the relative populations of the singlet and triplet metastable He levels, which can be loosely interpreted as a thermometer for the electron temperature (which is highly non-Maxwellian). The results suggested that the mean electron energy was quite low, i.e. sub eV, at the higher plasma densities.

Keywords: Semiconductor, Plasma, Etching

Materials Properties, Behavior, Characterization or Testing

314. Optical Absorption and Electrical Properties of Ferroelectric Ceramics FY 1992
$73,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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We are continuing electrical and optical measurements to help us understand the mechanisms for optical writing and electrical fatigue in bulk lead lanthanum zirconate-titanate (PLZT) and thin film PZT electrical ceramics. Sandia Labs is committed to thin film lead zirconate-titanate (PZT) ferroelectric devices for the next generation of non-volatile memories, and is exploring bulk PLZT devices for optical memory applications. The mechanisms whereby the electrically switched memories exhibit reduced polarization after being cycled through many hysteresis loops is not understood and presents severe limitations on device life. Likewise the ability of UV light to assist switching in bulk PLZT plates has previously been modeled with a simple phenomenological picture which clearly fails to predict some of the observed behavior. In this research period we have continued measurements to understand the nature of the UV-induced trapped charge in bulk PLZT ferroelectrics. By the use of phase sensitive photothermal deflection spectroscopy (PDS) and measurements where the probe laser beam is switched between the UV-exposed and non-exposed side of these samples, we have accurately determined the spatial location of the induced absorption.

Keywords: Ceramics, Piezoelectric, Electrooptic, Electronic

315. Radiation and Diffusion Effects in Glass FY 1992
$1,755,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A decay-limited diffusion model which includes trapping has been developed to predict concentration and compaction profiles in aged glasses. Since the structural integrity of aged glasses may be affected by radiation damage caused by electron energy deposition from tritium decay, it is desirable to have the capability to predict the tritium concentration profile both as a function of depth and of time. In the present work, a diffusion theory model is used to describe the concentration of tritium molecules in glass. The model is based on the radial form of the decay-limited diffusion equation and is used to solve for the tritium concentration in a hollow cylinder. In addition to the dissolved molecules, two terms are included in the model to represent trap sites arising from differing sources. One source of traps is based on an intrinsic population of trapping centers; the other results from the formation of radiation-induced hydroxyls and hydrides (Si-OT, Si-T). Although the exact nature of the former type of traps is unknown, it has been pointed out that during production various glasses can contain vastly different amounts of hydroxyl (for example, ranging from 0.0005 mol% to 0.4 mol%) which could serve as plausible "exchange" centers for tritium.

Keywords: Ceramics, Glass, Seals, Modeling, Fracture

316. The Effects of Hydrogen and Radiation Exposure on the Fracture Structure of Glasses

FY 1992
$581,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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The microscopic topograhics of fracture glass surfaces were studied using atomic force microscopy. Clear modifications in the structure resulted from hydrogen and radiation exposure. The primary goal of our work was directed towards understanding the fracture properties of different glasses and the effects of hydrogen and radiation exposure using atomic force microscopy. The motivation for these studies is the durability of glass materials in the storage of nuclear waste in which they would be exposed to an enviroment of hydrogen and gamma radiation. It is known from fracture measurements that exposure to hydrogen and radiation does increase the toughness of many glasses. However, the structure of the fracture surfaces have not been investigated. These surfaces display mirror-like finishes to the naked eye and even at scanning electron microscopy magnifications, no significant variation to the surface smoothness can be detected. To investigate the microscopic aspects of these surfaces, we have employed atomic force microscopy which provides topographical resolution in the nano-meter range. Our studies were performed on two types of glasses, fused silica and pyrex 7740.

Keywords: Ceramics, Glass, Microstructure, Fracture

317. Environmental Effects on the Thermomechanical Fatigue of Solders for Electronic Applications

FY 1992
$102,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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This study evaluates the response of solder joints under a variety of environmental conditions. Conditions of thermomechanical fatigue arise in solder joints in electronic packaging due to the combination of solder joints mechanically joining materials of different coefficients of thermal expansion and temperature fluctuations. The solder joints can fail under these conditions. It is important to understand and fully characterize the mechanics of solder joints under conditions of thermomechanical fatigue in order to predict the lifetime of the solder using fundamental principles. The characterization of solders under these conditions must be performed experimentally. Thermomechanical fatigue tests are performed on solder joints to examine the effect of cyclic strain and temperature on solder. Under a controlled setting, it is possible to determine metallurgical mechanisms and make lifetime predictions for solder joints. Thermal cycles are typically on the order of a few cycles a day for up to 30 years of life. To perform these experiments in a timely fashion, the testing rates must be accelerated and all factors that can influence solder joint behavior must also be accelerated including environmental effects.

Keywords: Metals, Solder, Microstructure, Chemical, Corrosion

318. Intermetallic Compound Growth (IMC) in Solder Joints

FY 1992
$45,000

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Diffusion modeling and empirical analysis are being used to predict IMC growth in solder joints for long-term reliability prediction. Intermetallic compound (IMC) growth occurs in solidified solder joints at the solder/substrate interface through solid state diffusion processes. Reaction kinetics cause appreciable thickening of the IMC layer(s) at elevated service temperature and long time periods. Excessive IMC growth can jeopardize solder joint reliability or interfere with follow-up processing steps. The accurate modeling of solder/substrate reactions will permit the prediction of product reliability beyond the limited laboratory test periods. Analytical diffusion models are being developed to model the growth of IMC layers characteristic of the base metal and solder chemistries. Software development is being based upon the copper-tin IMC architecture found in traditional solder joints on electronic assemblies. The basic analysis will then be extended to alternative solder (e.g., lead-free alloys now under evaluation) to similarly predict the growth and composition of the reaction layers. The techniques developed in this study will be applicable to evaluating the behavior of brazed joints used in high temperature, advanced metal alloys and ceramics.

Keywords: Metals, Joining, Solder

319. Liquid Metal Surface Studies FY 1992 $269,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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We have established a collaborative effort to investigate, using UHV techniques of Surface Science, the surface chemistry of liquid metals, an area with important technological implications, but limited scientific foundation. Soldering with liquid metals is central to technologies as diverse as electronics and plumbing and has been in use for millennia. But the surface chemistry of liquid metals is only poorly understood, if at all. This is especially surprising since liquid surface chemistry has been an established science for at least 100 years and there has been an explosion in the surface science of metals over the past 30 years. Thus, a combination of the established knowledge and techniques from these two diverse areas can be expected to have important impact on basic science as well as on important technological areas. We have established a collaborative program between Sandia and Case-Western Reserve University which combines expertise in Surface Science, Liquid Surface Chemistry, and Soldering Science. The central feature of this collaboration is a study of the surface chemistry of liquid metals in an UHV environment where the whole array of Surface Science techniques can be applied to characterize the liquid surface.

Keywords: Metals, Solder, Surface, Alloy

320. Metal Alloys for Hydrogen Storage and Handling FY 1992 $153,000

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Pure metal hydrides provide a safe and convenient method for storing hydrogen gas in an easily retrievable form. We are exploring new metal alloy combinations that will decrease material cost, increase the specific hydrogen capacity and improve the overall kinetics of the hydriding process. From a material standpoint, the formation of a metal hydride resembles a very heavy "cold working" process and therefore exacts a heavy toll on the metal media in terms of defect generation and potential phase separation of the alloy. This rigorous process is further complicated in a tritium environment by the effects of the radioactive decay by-products. Therefore, the eventual separation of all these effects into causal relationships requires a careful preliminary microscopic characterization of the candidate alloys. During this past year, we have completed such a characterization on an alloy system for which we have complementary tritium data. This characterization included chemical analysis, scanning electron microscopy, X-ray microprobe and equilibrium thermodynamic measurements. This study has already identified potential problems related to certain manufacturing methods and elemental segregation effects due to the hydriding process alone.

Keywords: Metals, Alloy, Microstructure

321. Metallurgical Evaluation of A356 Castings — FY 1992 $200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Brian K. Damkroger, (505) 845-3105

The base plate for the MC4369/MC4073 programmer used in the SRAM II/SRAM A/ PRESS A program is a casting made of an aluminum alloy, A-356, heat treated to the T6 condition. Because the mechanical properties of castings are widely thought to be inferior to those of wrought material, some concern arose over the use of a casting in this application. A investigation was undertaken to evaluate the properties of the supplied castings.Several production castings were used this investigation. It was first determined that these castings were representative of current production. The castings were evaluated using non-destructive inspection techniques and found to be of a very high quality, equivalent to at least a Grade B and perhaps a Grade A level under the applicable specification scheme. Several of the castings were sectioned and metallographically examined. The microstructure was found to be consistent with what was expected for this material in the as-cast plus heat treated condition. A literature survey was conducted and used to compile a representative mechanical property base for castings of the quality supplied. The specification actually called out for castings of a far lower quality, Grade C, which is associated with a more significant internal defect structure.

Keywords: Metals, Failure, X-ray, Microstructure, Fracture

322. Post Irradiation Examination of New Production Reactor Fuels — FY 1992 $34,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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We have been examining in detail radioactive fuel samples taken from ACRR experiments intended to simulate accident conditions in the formerly proposed New Production Reactor (NPR). Weight, size, density measurements, and optical and scanning electron microscopy (with EDAX and WDX analysis) are being performed. This experimental program is aimed at modeling accident behavior conditions in order to assure reactor safety. Two experiments have been conducted to date, one with fresh fuel and the other with irradiated fuel. The metallurgical examination of the post-irradiated fuel specimens is aimed at understanding the changes in the fuel material and its relocation behavior during fuel melt. In particular we are examining the interaction between the Al-U fuel and an essentially pure Al cladding, the production of defects (primarily radiation by-product induced voiding and swelling), and whether the molten fuel/clad droplets will interact with Al-Li target structures which surround the fuel. Results to date have showed that extensive dissolution of the cladding occurred in the first experiment, and that grain boundary penetration also occurred. Voiding was not observed.

Keywords: Metals, Melting, Porosity, Microstructure, Alloy

323. Solid Film Lubricants for WR Applications — FY 1992 $121,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A working group of Sandia and Allied Signal KCD personnel has continued to identify and demonstrate feasibility of alternate ES&H-Safe lubricants for weapons. The use of dry film lubricants in strong-link components and other weapons-related electromechanical mechanisms is widespread. Desired properties are reproducible friction and wear characteristics, long-term chemical stability and good adhesion upon exposure to weapon environments. These requirements place severe demands on commercial dry film lubricants. It is unclear whether new formulations expected from commercial lubricant vendors will fulfill these requirements. Poor performance and more stringent ES&H requirements prompt the development of alternatives to lubricants presently in use. The goal of this work is to identify alternative lubrication technologies to those presently in use that meet the performance and ES&H requirements of future weapons applications, and increase the reliability of present and future surety components through consistent lubrication.

Keywords: Metals, Tribology, Treatments, Film, Coating

324. Stockpile Support Capabilities — FY 1992 $3,135,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Robert J. Eagan, (505) 845-8943

Stockpile Surveillance programs require tests, analysis, materials and process certification activities. This project supports those critical capabilities. To fulfill Sandia's mission in stockpile surveillance, the materials organizations provide expert support in materials and process analysis, test interpretation, and production support. This project assures that critical capabilities, staff, and equipment meet the needs of this program.

Keywords: Metals, Capabilities

325. Studies on Fe-29Ni-27Co (Kovar) Alloy

FY 1992
$566,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: John J. Stephens, Jr., (505) 845-9209

A study of grain growth in Kovar™ leads has developed predictive relations for grain size as a function of time and temperature. The Fe-29Ni-17Co (Kovar™) alloy is widely used for a number of SNL-designed DP components which involve metal-to-ceramic brazing. For components such as neutron tubes, it has been known for a number of years that the composition of oxide-forming elements in this alloy must be tightly controlled to avoid generating an undesirable oxide haze on the surface of this alloy following dry hydrogen firing. More recently, problems with the wettability of Fe-29Ni-17Co alloy used for certain brazed switch tube subassemblies (which use a *wet hydrogen* atmosphere for brazing) suggested that oxide forming elements in Fe-29Ni-17Co alloy needed to be tightly controlled for this application as well. Other physical metallurgy aspects of this alloy, such as the grain growth kinetics in small diameter leadwires, were not well characterized. These items suggested that a significant amount of work needed to done on a material that is often considered to be "mature technology."

Keywords: Metals, Alloy, Seal, Modeling, Braze

326. Wetting and Mechanical Behavior of Interfacial Intermetallics in Solder Joints

FY 1992
$102,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Darrel R. Frear, (505) 845-9023

A study of intermetallic formation, aging characteristics and mechanical behavior of interfacial intermetallics in solder joints and also development of a general methodology for the characterization of interfacial intermetallics is underway. Intermetallics form at the interface between the solder substrate surface. The presence of an intermetallic is often an indicator for good wetting. However, after aging the increased thickness of intermetallics have also been cited as a possible cause for poor subsequent solderability. Furthermore, intermetallics tend to be brittle phases and their presence in solder joints that must withstand deformation may be a detriment to joint reliability. The principal problems with, and the lack of fundamental understanding of, interfacial intermetallics are: (1) How is wetting influenced by intermetallic formation? and (2) What influence do intermetallics have on the properties of solder joints? This work is a cohesive study of a variety of solder systems to develop a fundamental understanding of the role of intermetallics on solder joint properties and behavior.

Keywords: Metals, Solder, Microstructure, Packaging, Joining

327. Development of Improved Methods for Predicting and Validating Polymer Lifetimes in Weapon Environments.

FY 1992
$536,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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This project is attempting to rigorously validate/understand the often-used Arrhenius extrapolation technique for predicting polymer lifetimes in weapon environments and to develop improved methods for such predictions. The so-called Arrhenius methodology has been used for many years as a means of predicting thermal lifetimes of weapon materials and components. This approach, which is found to work well in many cases, but to fail in other cases, assumes a linear relationship between the log of the time to a certain amount of degradation and the inverse absolute temperature. If such behavior is confirmed under accelerated, high temperature conditions, the results are linearly extrapolated to lower temperatures in order to make long-term predictions. The goals of our program are to rigorously assess the applicability of the Arrhenius approach. In particular, we are trying to determine (1) when and why Arrhenius behavior is followed under accelerated conditions, and (2) whether methods can be developed to better assure that the linearity assumed in extrapolated predictions can be experimentally confirmed. The materials being studied include several elastomers, including two which are frequently used as weapon environmental seals.

Keywords: Organic, Aging, Seal, Polymer, O-ring, Modeling

328. Development of Solid Polymer Electrolytes for Lithium Rechargeable Batteries

FY 1992
$155,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Polymeric gels based on acrylonitrile polymers and low Tg block copolymers are being prepared and evaluated for use as solid polymer electrolytes in lithium rechargeable batteries. The U.S. battery industry wants to develop rechargeable lithium batteries having higher energy density and less environmental impact than the current commercial batteries such as nickel-cadmium and lead-acid batteries. Lithium rechargeable batteries can be made from liquid electrolytes or solid polymer electrolytes (SPE). A solid polymer electrolyte (SPE) is preferred over a liquid because of safety problems associated with leakage and the possibility of achieving higher battery performance at lower cost. To achieve higher performance, improvements in ambient ionic conductivity of present SPEs without sacrifice of mechanical properties is required. Specific property goals for the SPEs that we make are ambient ionic conductivities of 10^{-3}/ohm cm and a shear modulus of $\sim 10^7$ dynes/cm^2. Our approach is to prepare and evaluate ion-doped polymeric gels and low T_g block copolymers.

Keywords: Organic, Polymer, Electrolyte

329. Electromechanical Gels — FY 1992 $133,000

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The mechanism for gel deformation in electric field and dependence of shear modules on gel properties was determined. Measurements of osmotic pressure are beginning. An electromechanical (EM) gel is a low-modulus polyelectrolyte network swollen with water, but when an electric field is applied, the gel collapses as the water is squeezed out. In many respects, EM gels mimic the action of muscles. For this reason there are many exciting applications such as fine-motion robotic "fingers," large-deformation actuators, controlled selective membranes, and drug delivery systems. The major limitations of these materials involve the speed of the transition and the retractive force. Only one EM gel at present exhibits strengths approaching muscle tissue, most being an order of magnitude less. More serious, perhaps, is the lethargy of the transition, measured in hours rather than seconds. The goals of our program are two-fold: (1) to develop a better understanding of structure/property relationships in polyelectrolyte gels, and in particular, of their response to an applied field and (2) to develop applications for EM gels as "smart materials"-materials whose response to programmed external factors can be utilized as a controlling device.

Keywords: Organic, Polymer

330. Electronic Properties of Organic Materials — FY 1992 $626,000

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The electronic states and properties of delta- conjugated polymers pertinent to applications as light emitting diodes have been obtained. The electronic properties of organic materials pertinent to a variety of applications are being determined with the present emphasis on conjugated polymers and on those properties pertinent to polymeric light emitting diodes. Recent emphasis has been on the electronic states of s-conjugated polymers, which exhibit high fluorescence quantum efficiencies and are thus potential light emitting diode materials. We have used absorption, fluorescence, electroabsorption, two-photon and photoconductivity spectroscopies to locate and characterize the various states pertinent to possible applications in nonlinear optics and light emitting diodes and the s-conjugated polymers have served as model compounds to test and develop appropriate theoretical models.

Keywords: Organic, Electronic, Nonlinear, Fluorescent

331. Electrorheological Fluids — FY 1992 $241,000

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We investigated the polarization mechanisms of collodial particulates in an electric field, the shear rate dependence of the field dependent viscosity, and the evolution of structure as the field is applied. An electrorheological (ER) fluid is normally a low-viscosity colloidal suspension, but when an electric field is applied, the fluid undergoes a reversible transition to a solid, being able to support considerable stress without yield. The commercial possibilities for such fluids are enormous, including clutches, brakes, valves, shock absorbers, and stepper motors. However, performance of current fluids is inadequate for many proposed applications. Our goal is *to engineer improved fluids* by investigating the key technical issues underlying the solid-phase yield stress and the liquid to solid switching time. Our studies focused on the field-induced interactions between colloidal particles that lead to solidification, the relationship between fluid structure and performance (viscosity, yield stress), and the time evolution of structure in the fluid as the field is switched on or off.

Keywords: Organic, Electrorheological

332. Electrostatics of Electrorheological Fluids — FY 1992 $97,000

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Equipotential distributions associated with chains of particles in electrorheological fluids have been calculated, and from this the interparticle forces and effective dielectric constants of the fluid are obtained. Electrorheological (ER) fluids are colloidal suspensions that undergo reversible changes in viscosity in response to an applied electric field. Numerous applications for this effect have been suggested, and most of these would benefit from a greater contrast between high and low viscosity states than found in available ER fluids. Directorate 1800 has been studying a new class of fluids with highly polarizable particulates, which show a promisingly high yield stress at kV/mm applied fields. A detailed understanding of the interparticle interactions in such fluids is lacking, due in part to the relatively large multipole and multi-particle effects that arise from the high contrast between particle and carrier fluid polarizabilities. As a first step in elucidating the electrostatics of this class of ER fluids the equipotential distributions associated with isolated chains of spherical particles have been calculated, and the interparticle forces and effective dielectric constants of the mixture have been determined from these distributions for comparison with experimental data.

Keywords: Organic, Electrorheological, Modeling, Dielectric

333. Environmental O-Ring Seals—Properties, Aging and the Argon Method FY 1992
$854,000

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Our program goal is to enhance the reliability of weapon environmental O-ring seals by deriving better knowledge of properties degradation rate and methods for monitoring field aging. Environmental O-ring seals are used in most weapons to protect the interior from the ingress of potentially harmful quantities of such substances as water vapor and oxygen. Two components contribute to the ingress. The first, permeation through the o-rings, requires accurate knowledge of the O-ring's permeability coefficient. The second, which involves leakage between the O-ring and its metal-mating surface, is much more difficult to estimate, since it will depend both on the long-term aging characteristics of the seal and on other factors hard to predict (e.g., leaks may occur during low temperature exposures). Our O-ring program is attempting to address these issues by studying the aging (from surveillance units and from accelerated aging studies) of important O-ring materials used in weapons, as well as by generating the required permeation data. In addition, we have suggested and are developing an easy, inexpensive method for estimating the lifetime ingress of oxygen and water vapor into a weapon based on the analysis of the argon gas content in the weapon's interior.

Keywords: Organic, O-ring, Aging, Seal, Polymer, Modeling

334. Field Enhancements Associated with Conductive Protrusions FY 1992
$117,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A novel method for calculating field enhancements associated with axially symmetric conductive protrusions on planar electrodes has been developed. Results for a variety of practial shapes have been obtained. High-voltage components typically rely on dielectric encapsulation to prevent electrical breakdown during operation. The safety margin between the presumed dielectric strength of the encapsulant and normal operating stress is intentionally large; nevertheless, breakdowns are a recurrent problem. It is usually assumed that breakdowns are initiated at defects that cause localized field enhancement in the encapsulant, and that once initiated, a breakdown propagates through the remainder of the dielectric thickness. Knowledge of the amount of field enhancement associated with various defect geometries would be of considerable value in failure analysis as well as in studies characterizing defect initiated bulk breakdown. Enhancement factors are known analytically for a few special cases (for example, prolate semi-ellipsoids of revolution protruding normal to a conductive plane), but real-world defects are rarely found to conform to the well understood shapes.

Keywords: Organic, Encapsulant, Modeling, Bulk Breakdown, Dielectric

335. Moisture Absorption by Molded Desiccant FY 1992
$1,959,000

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The equilibrium and kinetics of moisture absorption by a composite of polyurethane and molecular sieve were determined experimentally and related by modeling. Maintaining a low dew-point in compartments containing electronics and other moisture-sensitive materials is essential for long-term reliability of most warheads. Desiccants are available in a variety of forms, including a composite material consisting of a molecular sieve powder dispersed in a polyurethane matrix. This material can be molded to fit into available space in the warhead and can also serve as a structural element. The moisture absorption capacity and permeability of this molded desiccant were measured gravimetrically for a material consisting of 40 wt.% Type 13X zeolite molecular sieve powder dispersed in a rigid polyurethane matrix. Moisture absorption properties were determined over a range of temperature and relative humidity and related to the properties of the individual constituents.

Keywords: Organic, Composite, Modeling, Encapsulant

336. Resist Characterization for Soft X-ray Projection Lithography FY 1992
$91,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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We are characterizing resist materials for use in soft x-ray projection lithography (SXPL). SXPL uses 14 nm radiation and reflective imaging systems to project high-resolution, sub-tenth micron demagnified images onto a photoresist. SXPL using a laser plasma source illumination is being developed at Sandia with AT&T for the ltihographic definition of 0.15 micron features required for 1 Gbit DRAM. Due to the extremely high photoabsorption cross sections of all elements at 14 nm, traditional photoresists do not yield adequate resolution in SXPL. Additionally, precise experimental values of absorptance at 14 nm, required for predictive modeling of resist performance, have not been available for any resist. We are investigating the sensitivity, lithographic performance, absorptance, and photodesorption of new resists in an effort to develop a sensitive, high-resolution resist which does not liberate highly absorbing contaminants onto the reflective imaging optics.

Keywords: Organic, Lithography, Polymer, Modeling

337. Tensile Properties of Epoxy Encapsulants — FY 1992 $992,000

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Tensile properties were measured for nineteen different formulations of epoxy encapsulants. Formulations were combinations of two neat resins, three fillers and four hardeners. The design of electronic assemblies is often critically dependent upon the appropriate use of polymeric encapsulating materials. Encapsulation can provide sensitive electrical assemblies with protection against shock, vibration, and atmospheric environments. However, due to substantial differences in thermal expansion between electronic assemblies and the encapsulation materials, detrimental stresses and deformations can be developed during the encapsulation process and subsequent thermal excursions. To assist in design and evaluation of encapsulated assemblies, accurate predictions of the states of stress and strain are needed. Finite element computations can yield the desired results provided there are proven material models and accompanying thermo-mechanical material properties to input into the models. In this program the tensile properties of epoxy encapsulants were measured to provide material properties for finite element computations.

Keywords: Organic, Encapsulant, Polymer

338. Viscoelasticity of Crosslinked Polymers — FY 1992 $25,000

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Fully-cured networks show the same viscoelastic response as critical gels. Computer simulation determined this is due to remnant percolative disorder in the fully-cured networks. The gel point demarks the continuous transition from liquid- to solid-like behavior. The viscoelastic response of critical gels (networks at the gel point) is a peculiar bridge between these disparate states. We have shown that the relaxation times for condensation-type critical gels are distributed in the power-law fashion. Since the sol-gel transition is analogous to a second-order phase transition, this power-law should also apply to gels cured somewhat past the gel point for times less than a characteristic time, t_z. The time, t_z, which decreases with increasing extent of reaction, corresponds to a length scale, x, below which the network retains a memory of the structure of the critical gel. This remnant percolative disorder can be observed in a region of extent of reaction called the *critical regime*. Although the critical regime is expected to be small, we have experimentally shown that the critical regime is actually extremely broad, extending to the fully-cured network for condensation-type crosslinked polymers.

Keywords: Organic, Encapsulant

Device or Component Fabrication, Behavior or Testing

339. Failure Analysis of the SA3694 Leaded Chip Thermistor — FY 1992 $115,000

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This project covers failure analysis activities led by Dept. 1845 on the SA3694 Leaded Chip Thermistor, which exceeded resistance change limits during thermal shock tests, required for qualification. During thermal cycling tests of the SA3694 Thermistor (W89) failure of one of the Buyer Thermal Shock test samples occurred. A resistance change (DR) of ~5% was measured, exceeding the 2% limit specified in the Qualification Requirements of PS412090. R. Baron, 2553 needed to know whether the failure was mechanical in nature, and whether the dual-layer epoxy encapsulation of the ceramic contributed to the failure. Of further interest was whether some test could be used to identify bad parts. We were unable to obtain useful information from the failed part because it had been altered during previous examinations. We obtained parts from the same testing lot which had exhibited significant positive DR's. Ultrasonic examination and careful epoxy removal revealed that each part contained cracks in the ceramic. Cracks were observed in the ceramic both perpendicular and parallel to the ceramic/electrode interface.

Keywords: Analysis, Fractography, Encapsulant, Joining, Modeling, Electronic

340. Characterization of Quartz Resonators Having Multiple Nonpiezoelectric Layers — FY 1992 $158,000

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The electrochemical quartz crystal microbalance (EQCM) is a device that combines the mass measurement capabilities of a QCM with the charge measurement capability of an electrochemical system. The EQCM and QCM have several applications, including chemical sensing and film characterization, wherein a polymer-coated quartz crystal is in contact with a liquid. In order to understand the electrical response of this composite resonator, a model has been developed to calculate the electrical admittance vs. frequency for an AT-cut quartz crystal in contact with any number of nonpiezoelectric layers. The goal of this work is to be able to distinguish between frequency changes induced by mass uptake in the polymer and frequency changes caused by polymer softening, effects that can occur simultaneously if the polymer film is acoustically thick. A paper is being prepared on the characterization of a quartz resonator with multiple nonpiezoelectric layers. The theory developed in the paper is more general than any published to date and reduces properly to known limiting cases, such as a QCM with simultaneous mass and liquid loading or a QCM with a single polymer film.

Keywords: Ceramics, Piezoelectric, Polymer, Resonance, Modeling

341. Decoupling Capacitors Using Ferroelectric Films — FY 1992 $4,000,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Ferroelectric thin films have been investigated for use in fabricating decoupling capacitors which can be integrated into semiconductor circuits and ceramic packages. Significant improvements in the high-speed performance of semiconductor devices can be achieved by replacing discrete capacitors with integratable thin-film capacitors. The improved performance would come primarily from a substantial reduction in interconnect length. The lead zirconate titanate (PZT) films being investigated are fabricated by sol-gel techniques. The materials have dielectric constants greater than 1000, which are ideal for 50-100 nF capacitors. The associated reduction in capacitor volume (1000:1) may also be critical for many advanced packages. For decoupling applications, the critical issues are the high-frequency impedance response, the leakage currents, and the long-term stability and reliability. Preliminary high-frequency dielectric measurements have demonstrated that the impedance response of thin-film capacitors are comparable to standard ceramic chip capacitors up to 1 GHz. Therefore, ferroelectric film capacitors have the potential to replace multilayer ceramic capacitors for some applications.

Keywords: Ceramics, Dielectric, Sol Gel, Packaging, Film, Ferroelectric

342. Diamond Substrates for Hybrid Circuits and Multichip — FY 1992 $120,000

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Thin film process development and characterization of integral thin film components to allow circuit fabrication on diamond substrates. Resistor-conductor networks have been made which meet requirements of circuits on alumina substrates. As applications for hybrid circuits and multichip modules create demand for higher density circuits and higher power components, new substrate materials are required to deal with the heat generated on the circuit. Sandia Laboratories is developing diamond substrate technology to meet the requirements of high thermal conductivity. Thin film processes were developed and characterized to delineate conductor-resistor networks on free standing diamond substrates having fine line gold conductors and low and high sheet resistivity resistors. Thin film hybrid circuit technology was developed on CVD processed polycrystaline diamond substrates having as-deposited surface finishes as well as those with polished surfaces. Conductors were defined by pattern plating gold and resistors were processed from sputtered tantalum nitride films which were deposited to sheet resistivities of 5 and/or 100 ohms per square.

Keywords: Ceramics, Diamond, Packaging, Metallization, Coating

343. Evaluation of Hermetic Packaging in Optoelectronic Devices — FY 1992 $213,000

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Three optoelectronic devices used in the W-89 fireset require hermetic packaging. Recommendations for changes in design and manufacturing processes were made to preserve the integrity of glass-to-metal seals. The W-89/PRESS-A utilizes two optoelectronic assemblies, MC4161 and MC4162, to couple signals in the firing set. The optoelectronic devices in these assemblies are the SA3678 Photodiode, the SA3679 Light Emitting Diode and the SA3680 Laser Diode. These devices are hermetically packaged in Kovar cases with glass insulated Kovar feedthroughs; hermeticity is required to assure that device performance is not compromised during stockpile lifetime. Processes during manufacture of the devices were found to degrade the glass-to-metal seals and cause loss of hermeticity of the package. Generic processing and handling modifications were recommended to glass seal suppliers, SA device suppliers and MC assembly manufacturers to preserve the integrity of the glass seals. A glass seal inspection requirement, SS396251, was defined and incorporated into the drawing package. Recommendations were made to have device sensitivity to water determined once hermetic packaging limitations were established.

Keywords: Ceramics, Packaging, Seals, Modeling, Glass, Fracture

344. Advanced Exclusion Barriers Study — FY 1992 $1,213,000

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Finite element analyses are used to quantify the mechanical response of specific barrier geometries. Results will be translated into the Barrier Design/Assessment Guide whose contents will be determined by a "house of quality" approach. The exclusion barrier is critical to the nuclear safety theme of modern weapons and must remain intact during all abnormal and accident conditions. For example, the barrier must be able to withstand high loadings at extremes in temperature and loading rate. The microstructural behavior of metallic materials during large scale deformations has not been well understood, and the ability of stress analysts to accurately model large scale deformations and predict cracking or tearing has been limited. This program is providing a better understanding of the roles of materials and geometry in barrier design. The first objective of this project has been to improve materials modeling and the ability to accurately predict large scale deformation behavior through finite element modeling. The second objective is to translate those results into a tool that can be easily applied by designers and nuclear safety analysts.

Keywords: Metals, Modeling, Failure

345. Brazing of Ferralium 255 Safing Wheels — FY 1992 $365,000

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Standard brazing procedures for safing wheels result in embrittlement of Ferralium 255. A new brazing procedure has been developed which does not degrade the mechanical properties of the alloy. Brazing of Ferralium 255 stainless steel MC4063 Safing Wheels in the 800-850°C temperature range is known to result in embrittlement of the alloy. In order to circumvent this problem, past efforts have been directed at development of alternative low-temperature braze materials and procedures. Although these efforts were successful in producing satisfactory brazes while maintaining base metal ductility, concerns were expressed by production vendors with regard to their ability to implement the low temperature brazing process. A study was therefore initiated to determine the feasibility of brazing at temperatures above those previously used (i.e., above 850°C). Specifically, the intent of this work was to determine if brazing thermal cycles, modeled after the solution annealing heat treatment for Ferralium 255, would result in degradation of the mechanical properties of the alloy.

Keywords: Metals, Embrittlement, Microstructure, Microscopy, Joining, Braze, Alloy

346. Development of Diffusion Bonding Processes — FY 1992 $68,000

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Diffusion bonding processes utilizing thin films are being developed as alternatives for fusion metal joining processes in weapon components. The diffusion bonding process is of interest for the vacuum seal and the formation of internal assemblies because it can significantly reduce the number of particles in comparison to high energy welding methods. The program also involves the development of a unique non-destruction ultrasonic testing method that utilizes color imaging to graphically represent the integrity of bonded interfaces. A bonding process was developed that successfully bonded a two-mil-thick screen with a fine wire mesh to a six-mil-thick screen having a coarse wire mesh. Each screen was coated with a thin film of silver (Ag), 10 μm, and diffusion bonded with a hot press at 450°C and 15,000 psi for 1 hr in a forming gas environment. The composite screen was subsequently formed to a test shape with no degradation, as indicated by non-destructive Ultrasonic Tests of the Ag-Ag diffusion bonds. The temperature and pressure were determined from a statistically designed factorial experiment. Materials incompatibilities with respect to adhesion between Ag and Mo were resolved by using a palladium interlayer.

Keywords: Metals, Weld, Joining, Bond

347. FastCast — FY 1992 — $1,106,000

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The FASTCAST program has continued to mature into specific theme areas being addressed by a multi-center team. The goal of FASTCAST is to integrate the theme areas of feature-based solid modeling, computational mechanics for casting simulation, and a rule-based toolkit for gating design into investment casting. In the area of feature-based solid modeling, several SGI workstations have been installed at each of the locations where processing of patterns, ceramic molds, and metal occurs. Each workstation runs the ProEngineer™ solid modeling software. With this capability, optimization of parts for casting, and placement of gates and runners can be readily achieved based on input from all three technology areas. In the area of rapid enmeshment, release of the alpha version of the CUBIT software allows for automated mesh generation for 2-D surfaces and 2\|F(1,2) -D with extrusions, sweeps, and swings. The simulation of casting has been expanded to address fluid filling with the commercial code ProCast™. In concert with in-house codes for thermal and structural analysis (COYOTE and JAC-3D), these codes are being applied to simplified casting simulation on complex parts. Generation of data for the casting toolkit is continuing with experimental validation of the filling of a center gated flat plate.

Keywords: Metals, Fastcast, Modeling

348. Fusion Welding of Advanced Borated Stainless Steels — FY 1992 — $131,000

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The United States has developed leading technology for the production of borated stainless steels for use in the transportation and storage of nuclear materials as well as for shielding and neutron flux control in nuclear applications. However, the impact of this leadership position will not be fully realized until successful joining processes are identified. Traditional weld processing of these materials has been shown to so dramatically impact the mechanical properties of these alloys as to severely limit their use in applications where structural code requirements are operative. Included in the structural code requirements are materials tests developed for traditional alloys (ferritic steels) which do not describe material performance or provide design criteria suitable for these advanced alloys. This research program addresses two major objectives. First, the identification and development of commercially viable welding technology, based upon a complete metallurgical understanding of the response of these materials to thermal processing including melting. Second, the identification, development, and implementation of appropriate mechanical testing techniques, based upon a scientific understanding of the fracture process in these composite materials.

Keywords: Metals, Weld, Microstructure, Microscopy, Steel, Melting, Joining, Embrittlement, Alloy

349. Fusion Welding of Fe-29Ni-17Co Alloys FY 1992
$116,000

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Weldability analysis of Fe-29Ni-17Co indicates that the behavior of this alloy is similar to 304L and 316 stainless steels. Continuing work will assess the effects of chemistry variations on weldability. Applications for the controlled thermal expansion alloy Fe-29Ni-17Co often require joining by fusion welding processes. In addition, these applications usually require hermetic and high reliability joints. The small size of typical components normally dictates the use of autogenous welding processes, so that the hot cracking tendency of Fe-29Ni-17Co is of concern. The solidification behavior and hot cracking tendency of commercial Fe-29Ni-17Co has been evaluated using differential thermal analysis (DTA), Varestraint testing, light and electron microscopy, and laser welding trials. DTA and microstructural analysis indicated that the solidification of Fe-29Ni-17Co occurs as single phase austenite, does not exhibit the formation of terminal solidification phases, and results in only minimal segregation of major alloying elements. Varestraint testing indicated that the hot cracking behavior of Fe-29Ni-17Co is similar to, though somewhat more pronounced than, 304L and 316 stainless steels.

Keywords: Metals, Weld, Microstructure, Microscopy, Joining, Alloy

350. Inertial Confinement Fusion (ICF) Target Fabrication FY 1992

$242,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Three-dimensional, free standing thin film structures are being fabricated for the Particle Beam Fusion Accelerator II (PBFA II) inertial confinement fusion research program. The structures are high z walls for light ion beam fusion targets. The structures will function as a source of X-rays for diagnosing particle beam intensity and as a barrier to prevent radiation from escaping from the target. The structure is in the shape of a hollow, truncated cone on the order of one centimeter in diameter and height. The walls of the structure consist of thin layers of gold and titanium on the order of one micron in thickness. Fabrication was accomplished by physical vapor deposition, magnetron sputtering of the Au and Ti onto a rotating plastic (PMMA) mandrel. The mandrel was subsequently dissolved away leaving the desired three-dimensional free standing structure. Calibration deposition runs were performed to determine the radial deposition rate on the rotating mandrel. Deposition rates were on the order of 10 Å/s. A series of process optimization runs were carried out to minimize the stress in the Ti layer and to keep the mandrel below 90°C.

Keywords: Metals, Film

351. Laser Beam Welding Calorimetry Studies FY 1992

$300,000

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Measurements of the energy transfer efficiency of the LBW process have been made and incorporated into predictive equations using dimensionless parameters. Laser beam welding (LBW) is an advanced manufacturing process used for final assembly of many expensive and critical components throughout the DOE weapons complex and in many U.S. industries. Despite more than 20 years of application, in many ways the LBW process is less controlled and not as well understood as the more conventional arc welding processes. We have been investigating the LBW process by measuring the efficiency of energy transfer to the weldment through calorimetric measurements. To date, detailed experiments on 304 stainless steel, 1018 steel, and pure tin have been completed. Preliminary results of these experiments have indicated that absorption of the laser beam by the weldment is directly related to the laser beam power density incident on the sample surface. Using two new dimensionless parameters, predictive equations have been developed to accurately determine the amount of energy absorbed from the laser beam by the weldment. We are postulating that the underlying heat transfer mechanism is likely controlled by the thermal boundary layer created by the moving weld pool.

Keywords: Metals, Weld, Packaging, Joining

352. Palladium Cleaning FY 1992
$345,000

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The mechanisms involved when palladium is cleaned using heat and gaseous treatments have been investigated in order to understand the variables and eventually to develop an optimized, robust cleaning process. Palladium must be cleaned in order to be an effective catalyst. The current cleaning procedure is based on previous empirical performance data, but not on specific experience with the current material or on real time measurements of the cleaning process. Our goal in this study is to develop an understanding of the cleaning process and simultaneously develop process diagnostics that will allow us to determine in real time when the material is clean. Additionally, we hope to optimize the process to provide a material of appropriate cleanliness in the smallest amount of time with the least effort. The final process must be robust to changes in material chemical impurity levels.The current cleaning method is a batch process using heat and reactive gases. We have analyzed the product gases evolved in each step of the batch process using Fourier transform infrared (FTIR) spectroscopy. Since many such steps are necessary, we have also started to develop a continuous cleaning process that shows promise of cleaning the palladium more efficiently.

Keywords: Metals, Cleaning, Surface

353. Plasmatron Improvement Program Materials Selection Activities FY 1992
$57,000

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A variety of materials selection issues were addressed for the MC3859 Plasmatron in FY92. The MC3859 incorporates a larger number of processing steps than standard sprytrons, and as such is uprobably the most difficult switch tube to produce. During FY90, materials personnel at SNL, AS/KCD and EG&G, Salem teamed to conduct a study of cracking in trigger probe area seals in this component: the results of this study indicated that cracking was caused by rebrazed area seals and excessive alloying of the braze due to the use of a long braze cycle and a new pin material. During FY92, a "Plasmatron Improvement Project" (PIP) identified a number of process/design changes which could enhance the manufacturability of this component. We supported the PIP by performing materials selection studies for a couple of key parts and subassemblies. One item whose materials callout was changed is the switch pin cap: this was formerly specified as 1100 aluminum alloy. Unfortunately, there is a relatively large degree of variability in chemistry for 1100 aluminum—especially in the maximum levels of Fe and Si. 1188 aluminum alloy—which is normally used as "pure" aluminum welding rod—controls these and other impurities to a greater degree than 1100 aluminum.

Keywords: Metals, Packaging, Braze, Alloy

354. Solder Joint Assessment of Resonator Packages

FY 1992
$15,000

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Au-Su solder seals and circuit board solder joints are being assessed for reliability towards the introduction of commercial surface mount resonators in weapon subsystems. Surface mount technology for printed wiring board assemblies permits the miniaturization of electronic systems. Oscillator circuits which use quartz resonators as frequency control devices, have benefited by this miniaturization as resonators have likewise diminished in size and are available in surface mount packages. Surface mount resonators are targeted for weapon systems to further reduce circuit board real estate in the unit. The surface mount resonator built by Statek Corp. (Orange, CA) is being used on the MC4226 clock circuit. Performance of the solder joints is being evaluated. The relatively large size of the package makes the solder joints particularly susceptible to thermal fatigue damage. Units are subjected to thermal cycling; the solder joints are cross sectioned and examined for crack development. Solder joint shear strength is used to quantify microstructural changes to the solder as a result of thermal fatigue.

Keywords: Metals, Solder, Joining, Electronic

355. Solvent Substitution for Heather Parts

FY 1992
$25,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Two separate studies are being done to replace halogenated solvents used in the manufacturing of Heather and related components with environmentally acceptable alternatives. Chlorinated fluorocarbon (CFC) solvents, such as trichloroethylene and Freon, used at Martin Marietta Special Componets (MMSC) to clean piece parts, can not be used after July 1993 per agreement with DOE. EG&G Rocky Flats has demonstrated that, an aqueous surfactant cleaner, can be used to effectively clean metal parts. Sandia and MMSC have designed a solvent substitution study in the form of a Specific Engineering Instruction

(SEI). The SEI specifies the various analysis techniques such as Auger Electron Spectroscopy, X-ray Photoelectron Spectroscopy, and Secondary Electron Spectroscopy on the metal surfaces, as well as, ion chromatography, infra-red spectroscopy, and gas chromatography to analyze the cleaning bath to be done at MMSC.

Keywords: Metals, Cleaning, Surface, Chemical

356. <u>Thin Film Solder Conductor as Weak Link Device</u> FY 1992
$100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Thin film solder conductor paths are being developed as weak link triggers to over temperature environments in weapon systems. The safety of nuclear weapons under adverse environmental conditions depends upon sensors in the arming, fuzing, and firing electronics to detect such circumstances. One such sensor, in this case a "weak link", disables the system upon exposure of the weapon to elevated temperatures. A first-principle weak link concept is the use of electrical conductors which open upon exposure to high temperatures. Such a device is being developed, based upon thin film conductor lines fabricated of the low melting temperature metals and alloy, 100In, 100Sn, and 50In-50Sn. The conductor lines are electron beam evaporated onto optically polished, z-oriented single crystal quartz substrates. The In-Sn alloy is deposited as separate layers of In and Sn, the thicknesses of which provide for the 50In-50Sn composition. When the substrate is heated, the metal films melt and disintegrate under their respective surface tension, causing the film to electrically open. Optimization tasks include the film thickness and the conductor line dimensions.

Keywords: Metals, Solder, Coating

357. <u>Conductive Adhesives</u> FY 1992
$993,000

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The purpose of this project is to investigate the performance and reliability of alternatives to conventional lead solder pastes in surface mount applications. Solder alternatives have advantages over conventional methods. Smaller leads and tighter spacings of surface mount devices are placing demands upon materials which traditional solders may not be able to meet. Also, increasing environmental concerns also favor the use of solder alternatives. Lead compounds are in the top ten as environmental dangers. Their use has already been prohibited in the plumbing and painting industries. The electronics industry may soon be forced to seek alternatives. Reductions in fluxing and cleaning, accompanied by a decrease in emissions of hazardous solvents, are a benefit to the environment. Conductive epoxies offer many advantages over conventional tin lead solder pastes. Fluxing, cleaning, and tinning can be reduced or eliminated. Reflow or cure temperatures are much lower, which reduce thermal stresses associated with processing and which reduce damage to heat-sensitive components. Lower rework temperatures help to limit damage caused by excessive heat. Many alternatives benefit from better physical properties and coefficients of thermal expansion (CTEs) which better match those of the laminate and the components.

Keywords: Organic, Adhesive, Polymer, Solder

358. Design and Fabrication of ICF, X-ray Laser, and Hohlraum Targets — FY 1992 $241,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: James H. Aubert, (505) 844-4481

Design and fabrication techniques of thin metallic and organic films and low-density foams were developed for ICF targets. Target experiments designed to study the stopping power of ion beams and the resulting radiation fields are being conducted on the Saturn and PBFA accelerators. Target designs are developed in order to test a specific physical process and corresponding code prediction. The designs are for specific ion beams (composition and energy) and often utilize materials that are both challenging to prepare and to manipulate into the designed geometric arrangement. These materials include thin metal foils, thin polymer films, and low-density foams. We have prepared 5000A polystyrene films (measured with ellipsometry) with a uniformity adequate for recent x-ray laser experiments conducted on Saturn. A new assembly technique was developed in order to move these films to the x-ray laser target body intact. Targets for Hohlraum experiments are being coordinated with Y-12 and LANL. The basic target is an aluminum foil of 4000A thickness in a cylindrical geometry of diameter 1 inch. Prior foils provided by LANL have been extensively characterized and found to be extremely variable.

Keywords: Organic, Foam, Microporous, Metallization, CVD

359. MDA-Free Printed Wiring Boards — FY 1992 $993,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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An extensive characterization of commercially available MDA-Free high performance PWBs has been conducted. This characterization includes electrical, mechanical, physical and thermal properties with emphasis on long term reliability. This project has the task of replacing the high performance printed wiring board (PWB) laminate material used in the DOE weapons complex, for support of existing and future weapon systems. The high performance laminate material presently in production contains methylenedianiline (MDA), a suspected carcinogen. In an effort to use environmentally conscious materials and processes in the weapons complex this PWB laminate material should be replaced. Due to the importance of maintaining the long term reliability and other benefits of the high performance laminates used in electronic packages, an extensive evaluation and characterization of a replacement material had to be instituted. To determine potential replacement materials, a market survey was conducted resulting in five materials to be evaluated. These candidate materials are two MDA-free polyimides and three cynate ester materials, all supplied by US manufactures. According to their specifications all the candidate materials should meet the weapons complex needs.

Keywords: Organic, Packaging, Polymer, Electronic, Dielectric, Composite

360. Materials Substitution for Transparent Armor FY 1992
$20,000

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Suggestions were made for substitute materials to be used in transparent armor stackups (for ballistic protection). Modified materials are now being evaluated. We had previously shown that aging due to solar UV exposure of the inboard polycarbonate ply was responsible for shortened effective life in windows and windshields used for ballistic protection. In order to reduce the mean time between replacement for windows made from these stack-ups, we had recommended incorporation of specific UV screening agents in the polymer interlayers. During this reporting period, a commercial supplier of interlayers with the recommended UV screen was found, and initial stack-ups made for ballistic testing purposes. These materials are mow being evaluated for ballistic protection, both at SNL and at PPG. Should qualification testing prove positive, we will place a small order for windows to be installed, as well as several for accelerated testing.

Keywords: Organic, Optical, Polymer, Composite

361. Patterned Metallization of Teflon FY 1992
$100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Control of the surface chemistry of Teflon‰ has allowed development of processes for area selective, metallization with resolution of at least 20 micrometers. Because of its low dielectric constant, Teflon‰ is an ideal substrate for electronic applications, especially for very high frequency applications where capacitive coupling becomes a major problem. The major barrier to Cu on Teflon applications, however, is the well-known chemical inertness of Teflon; current state-of-the-art high frequency Radar technology depends on Cu clad Teflon produced by hot-pressing etched Cu foil and Teflon. We have developed a family of three-step processes for direct patterned deposition of adherent Cu onto Teflon with resolution, to date, of 20 mm. Adherence of Cu to Teflon results form chemical etching of the surface with sodium naphthalenide which produces a highly porous defluorinated surface region extending to a depth of 4000 A; in one variation, patterned Cu deposition results from control of the etching by patterned pre-irradiation of the surface. Rutherford Backscattering Spectra (RBS) of thin Cu films electrolessly deposited on etched Teflon suggest that penetration of Cu occurs into this entire etched region.

Keywords: Organic, Metallization, Lithography, Film, CVD

362. Plasma Cleaning FY 1992
$100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Pamela P. Ward, (505) 844-2038

Plasma cleaning is a potential candidate for surface preparation of ferrites. Addition of water to an oxygen plasma increases organic removal rates but not as well as fluorine addition. Plasma cleaning is a process that is used industrially to prepare surfaces for improved adhesion. Immersion of the object in a glow discharge of the proper gas mixture results in removal of contaminants and surface activation. Plasma cleaning is an environmentally safe alternative to some current solvent cleaning methods, but for many applications plasma processing is too slow. We are exploring alternative plasma chemistries and methods which will increase the range of applications and make plasma cleaning a more viable solvent replacement. One component of this program involves evaluating various plasma treatments of a ferrite disk used in the stronglink assembly. Currently, the ferrite surface is prepared for metallization by immersion in phosphoric acid, but this treatment has reliability problems. Working with Carl Pennington, 2543, we have prepared ferrite disks with four plasma treatments prior to metallization. Initial results from adhesion testing appear promising for one of the plasma treatments.

Keywords: Organic, Plasma, Polymer

363. Polymeric Optical Waveguide Sensors Integrated with Active GaAs Devices FY 1992
$296,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A simple sensor based upon integration of polymeric optical waveguides with epitaxial GaAs emitters and detectors has been demonstrated. Sensors utilizing guided optical waves in the transducer element, such as fiber optical sensors, have gained wide acceptance in recent years. In this project we have developed a simple sensor based upon an integrated optical circuit consisting of epitaxially grown GaAs emitters and detectors connected by a planar polymeric optical waveguide. This work was focused on determining the waveguiding properties of the polyimide used for the waveguides. Waveguide losses were measured by launching light into the guided modes of the waveguides and measuring the intensity of the light scattered from the guide as a function of the distance from the point of injection. These measurements revealed a loss of 1dB/cm for the TE_0 mode, and a larger loss of 9 dB/cm for the TE_1 mode. A higher loss is expected for the TE_1 mode since it interacts more strongly with the absorbing semiconductor substrate. The low loss of 1 dB/cm was only obtained for polyimides which were not "hard baked" (250 C for 60 min). Hard baking increased losses by approximately a factor of three.

Keywords: Organic, Waveguide, Polymer, Optical

364. Rocket Propellant Removal and Size Reduction by Cryocycling — FY 1992 — $449,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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The objective of this LDRD project is to develop a safe and environmentally conscious technology for removal and processing of hazard class (HC) 1.1 propellants from solid rocket motors slated for demilitarization. The project includes: (1) development of inert material to simulate the properties of HC 1.1 propellants; (2) determination of mechanical and thermal properties of a variety of inert and live propellants from 25°C to -196°C; (3) investigation of methods to reduce the particle size and remove the propellant from the motor casing; (4) modeling of crack initiation and propagation; and (5) demonstration on live propellant. During the previous six months significant progress was made on evaluating cryocycling as a removal technique that prepares the propellant for reuse, solvation/solvolysis, or destruction. Cryocycling uses repeated applications of liquid nitrogen followed by warm-up to ambient temperature to induce multiple fractures in the propellant grain. After a few cycles, these fractures reduce the average particle size of the propellant significantly. Inert materials have been developed to simulate the properties of live propellant for our laboratory studies.

Keywords: Organic, Modeling, Energetic, Polymer, Composite

365. Smart Materials and Structures FY 1992
$652,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Work has been initated on embedding optic sensors into composite materials. Smart materials and structures are highly integrated assemblies incorporating sensors, actuators, local intelligence, and communication. These materials offer adaptability in uncertain or inhospitable environments. Critical to the successful implementation of these materials is the use of composite manufacturing technology, as well as understanding the interrelationships between the host materials and the sensors and actuators. Our emphasis during the last year has been on incorporating fiber optic sensors into composite structures. These sensors were chosen because they offer significant processing and engineering advantages, and a TTI proposal involving 8711, 1315, and two outside companies has been funded. In the last six months our activities have been twofold. First, we have assembled a multi-use fiber optic system including sources, detectors, and data acquisition capability. The performance of the system has been validated in the laboratory and then further proven during a field test recently conducted at TTR. Second, we have been studying implementation issues such as sensor ingress and egress.

Keywords: Organic, Composite, Sensors, Polymer

366. Stockpile Shipping Container Evaluation FY 1992
$31,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Flexible polyurethane foams and "horsehair" (a natural or synthetic fiber bound with latex rubber) cushioning materials were evaluated for continued use in W-56, W-62, W-70, and W-78 weapon shipping containers. Shipping containers used by the older Livermore weapons programs (W-56, W-62, W-70, and W-78) were evaluated to make recommendations as to whether or not these containers should continue to be used to transfer weapons from sites to Pantex for disassembly. The materials evaluated were the flexible polyurethane foam and "horsehair" (a natural or synthetic fiber bound with latex rubber) cushioning materials. The tests conducted on these materials were density, compression set, and compression (load-deflection). The flexible polyurethane foams met the density and compression set requirements and nearly met the original load-deflection requirements. However, the "horsehair" material was badly degraded. Our recommendations were to continue using the flexible polyurethane foam, and replace the "horsehair" materials with a flexible polyether type polyurethane foam.

Keywords: Organic, Foam, Packaging, Fiber, Adhesive

367. Very Fine Line Ta_2N Resistors for Circuit Applications FY 1992
$205,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Thin film TaN_2 resistors on alumina hybrid circuits have normal linewidth and spacings of 4mils(100mm). By processing for advanced silicon devices, 0.1mil(3mm) lines and spaces have been generated. Significant (1100:1) circuit space savings result. The Hybrid Microcircuit Organization, 2411, now has the capability of generating Ta_2N thin-film resistors with features as small as 3-micron line widths and spaces on as fired alumina substrates. Standard line and space widths are 100 microns. This miniaturization represents an area reduction of more than 1100 to 1. The large reduction can be used to save space on hybrid microcircuits (HMC) or be used to generate megohm valued resistors in small areas. Silicon circuit type processing is used except that it is applied to (rough) alumina ($A1_2O_3$) substrates, or smooth insulators such as fused silica both used in fabricating HMCs. The miniature resistor processing includes removal of the unwanted Ta_2N areas using plasma etching rather than wet etching with hazardous HF solutions. The spin on photoresist for all sizes of resistors uses a water based developer rather than ozone depleting solvents. Hence, Sandia standard resistors as well as the miniaturized resistors reduce the hazardous waste stream from processing.

Keywords: Semiconductor, Plasma, Lithography, Etching, Film

Instrumentation and Facilities

368. Analytical Capabilities FY 1992
$4,701,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Advanced analytical techniques are a critical element of meeting DP needs for characterizing materials and processes used to produce and dismantle weapons. This project supports the maintenance and improvement of analytical capabilities required to develop and characterize materials and processes used to produce or dismantle weapons. Typical tasks include software upgrades, retrofitting equipment, and development of broadly applicable analytical techniques.

Keywords: Analysis, Capabilities

369. Chemometric Methods and Software FY 1992
$52,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Sandia's chemometric software has been improved and more completely tested. Investigations were initiated to improve the applicability of chemometrics to spectral analyses by studying methods to transfer calibration models between spectrometers. Chemometric methods are powerful new methods that have significant applicability for improving the accuracy, precision, and reliability of quantitative spectral analyses. At Sandia we have developed and programmed chemometric multivariate calibration methods on the VAX computer. This software has been converted to the PC and has undergone final beta testing. In addition, in order to increase the utility of these methods for widespread industrial applications, we must make the calibration models more robust to spectrometer drift and make the models developed on one spectrometer be able to be used on spectrometers at other sites without expensive recalibration.

Keywords: Analysis, Chemometric, Spectroscopy

370. Combustion Efficiency Measurements at the Combustion Research Facility FY 1992
$804,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: T. Tazwell Bramlett, (510) 294-2299

Techniques were developed to enable real-time measurement of combustion efficiency at the Combustion Research Facility. Research to quantify the efficiency of pulsed combustion process required the development of techniques to measure the concentrations of combustion gases. Real-time monitoring of reactant and product gas concentrations in a combustion reactor at the Combustion Research Facility (CRF) in Sandia/CA was performed using the GC/MS. The CRF is developing technology to combust VOCs on a large scale into environmentally harmless products. A test matrix using many concentrations of VOCs and oxidizing/fuel gases was tested to assess efficiency of combustion under these different conditions by on-line measurement of reactant/product concentrations. The data showed a clearly defined region in which the combustor can efficiently burn VOCs, including chlorinated VOCs. Major equipment modifications and elaborate sampling equipment was developed to enable the measurement of low concentration exhaust VOCs to the high concentration inlet (reactants) gas concentrations. Due to the high quality and resolution of the results, further funding is being pursued to continue the work.

Keywords: Analysis, Spectroscopy, Chromatography

371. Compatibility Studies with the Energetic Liquid Components of PEX FY 1992
$1,119,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A large series of organic and metallic materials were exposed to the liquid components of Paste Extrudable Explosive (RX-08-FK from Lawrence Livermore National Laboratory). These media are aggressive towards all tested metals and organic materials except Teflons and polyethylene. We are studying the interaction of Paste Extrudable Explosive with candidate materials for the construction of various transfer systems. The explosive solid constituent of PEX (HMX) is well characterized. We have focused on material interactions with the aggressive liquid components. Many metals and organics have been exposed to either the individual constituents or to the complete energetic liquid mixture both at room temperature and 74 °C for up to six months. All of the metals and alloys tested (304 SS, 316 SS, Monel 400, Al, Cu, Ni, Ta, Au) show evidence of interactions with the liquids. These interactions the form of corrosion, weight gain, dissolution and/or changes in residual mechanical properties. Only certain fluorinated polymers, polyethylene, and some high performance engineering resins remain apparently unchanged over the course of this preliminary exposure.

Keywords: Analysis, Chemical

372. Continuous Monitoring of Metal-Aerosol Emissions FY 1992
$422,000

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Laser Spark Emission Spectroscopy is being used to develop a device to continuously monitor levels of metals in aerosols. The initial focus is on chromium arising from plating-bath operations. The objective of this program is to develop and demonstrate a continuous monitoring device to measure emissions of potentially hazardous metal aerosols in weapons-complex manufacturing processes. The first device will be tailored to measure metal aerosols emitted by plating-bath operations at the Allied-Signal Kansas City plant, and the focus has been on aerosols containing chromium. In plating-bath operations, large volumes of ventilation air are pulled over the baths to protect workers from fumes. This air is then directed up stacks and released to the atmosphere. Metallic compounds entrained in the ambient-temperature air will generally be in aerosol form. Laser-spark emission spectroscopy (LSES), a technique previously demonstrated at Sandia to measure inorganic constituents of burning coal particles, is the proposed method to measure the composition of these aerosols. The LSES technique is being adapted to measure metal aerosols typical of the Kansas City plating-bath emissions.

Keywords: Analysis, Spectroscopy, Scattering, Resonance, Chemical

373. Diagnostics for Steelmaking FY 1992
$20,000

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Feasibility experiments on optical diagnostics for process control in steelmaking were conducted at Bethlehem Steel Corporation. Research and development of optical diagnostics for process control in Basic Oxygen Furnace (BOF) steelmaking has been supported by the Technology Maturation Program. The goal of this work is to develop real-time optical sensing methods for optimizing the carbon content and final temperature in the molten bath of a BOF. Activities in FY92 have included feasibility experiments at a Bethlehem Steel Corp. pilot-scale BOF facility involving optical measurements of the off-gas of a BOF during a steelmaking heat, and video-imaging and pyrometric measurements of the molten bath. Optical measurements of the BOF off-gas involved fixed-wavelength cw-infrared laser transmittance measurements, and we successfully transmitted mid-infrared laser beams through the highly particulate-laden off-gas stream throughout the heat. These results demonstrate the possibility of measuring both the average line-of-sight composition and spectroscopic temperature of infrared-active gases (CO, CO_2, H_2O) by tunable diode laser spectroscopy which can be related to the process variables of interest.

Keywords: Analysis, Spectroscopy, Chemical

374. Electron Beam Treatment of Oil Spills FY 1992
$42,000

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Phase 1 research on treatment of oils with electronics has been successfully completed with partial destruction demonstrated. Conventional methods of cleaning oils spills are both costly and messy. Research with the Pulsed Power Department is exploring the feasibility of using high energy electrons to decompose oil into environmentally benign compounds. Phase 1 of the research has been completed with preliminary results obtained for model compounds. Model compounds are used instead of crude oil to reduce the difficulty of analysis and interpretation of the results. Numerous products were identified from the destruction of the model compound using electrons ranging from low molecular weight fragments to high molecular weight polymers. Future work will concentrate on exploring parameters affecting the kinetics of the reactions and on further identifying compounds in the aqueous and organic phases.

Keywords: Analysis, Chromatography, Spectroscopy

375. <u>Focusing X-rays Using Capillary Optics</u> FY 1992
$65,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Recently, developments in the former Soviet Union to produce lenses consisting of capillary arrays for focusing hard X-ray radiation were published. The development of an X-ray lens has the potential to significantly enhance our capabilities in the areas of material science, astronomy, medical radiography, X-ray lithography, fusion power, and national defense. The purpose of our study was to determine current status of this technology and its possible application to areas of interest to Sandia. As a result of this study, the authors are convinced of the technical feasibility of utilizing capillary arrays to produce lens elements for hard (0.1nm) X-ray radiation. We proposed a research program be started to develop this technology at Sandia. The successful production of capillary elements would greatly enhance our ability to characterize materials using X-rays. In addition, X-ray lenses could lead to significant improvements in the resolution and speed of X-ray lithography. National defense applications include back lighting targets in fusion experiments, weapon effects experiments, and the detection of X-ray sources by focusing X-rays onto detectors from orbiting platforms. Medical applications are higher resolution radiographs and the development of an "X-ray scalpel" for the destruction of cancer tumors.

Keywords: Analysis, X-ray

376. <u>Hazardous Materials Characterization for Weapons Dismantlement</u> FY 1992
$238,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A sampling and analysis process for hazardous materials is being developed to support hardware inventory reduction and weapon dismantlement programs. One of the common "waste streams" generated throughout the nuclear weapon complex is component hardware originating from the nuclear weapons program. Federal regulations require that the hazardous characteristics of materials must be identified prior to disposal as solid waste. Chemical analysis methods are complicated by the complex, inhomogeneous, material composition of these components. We are developing sampling and analysis procedures for toxic materials characterization to support the Weapon Hardware Inventory Reduction Effort (WHIRE) at SNL/NM. This process is also applicable to weapon hardware generated by other DOE Weapon Dismantlement Program activities.

Keywords: Analysis, Chemical, X-ray, Spectroscopy

377. Hydration and Optical Absorption in Implanted Alumina

FY 1992
$120,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: George W. Arnold, (505) 844-3848

Ion-implanted alumina adsorbs H from the atmosphere (ion-beam measurements). Anomalously large H concentrations are measured from Al implants. Al-induced F-center absorption tracks the H uptake. Aluminum oxide (Al_2O_3) is an important technological material due to its high melting temperature (2045°C), transparency over a wide wavelength window (0.2-6.0 microns), and relatively high chemical stability and hardness. It has found wide usage as a substrate for silicon structures and is also attractive because it can be processed as an amorphous ceramic. The effects of ion implantation with active ions to produce phase separation has been extensively studied. We have recently found that the near-surface damage produced by ion implantation allows adsorption of molecular water from the ambient atmosphere. The same effect was previously found for implanted fused silica and quartz. Such effects are of importance in geologic waste media and in waste containment hosts. Although the maximum H concentrations are relatively small (on the order of 1 atomic %), the penetration into the bulk exceeds 200nm in some cases indicating that channels due to cracking have been initiated by the damage and the stress.

Keywords: Analysis, Ion Beam, Surface, Spectroscopy, Porosity

378. Interfacial Force Microscope Development

FY 1992
$220,000

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In conjunction with our BES-supported Fundamental Science of Adhesion program, we have been involved in an effort to develop force sensors capable of the force-feedback stabilization technique that characterizes the Interfacial Force Microscope (IFM). In addition, in an attempt to commercialize the technique, a CRADA has been negotiated involving Digital Instruments (the leading manufacturer of atomic force microscopes), AT&T and the University of New Mexico. The work described in the present report involves our progress in the overall area of IFM development. Our initial prototype force sensor was fabricated using printed circuit board technology and consisted of Au capacitor pads and contacts deposited on a glass substrate. The sensor element involved a "teeter totter" common capacitor plate etched in a 75 mm BeCu sheet. The capacitor gap was established by cleaved mica spacers and the unit was assembled using strain-gauge cement. This unit had good electrical characteristics but was difficult to assemble consistently. The second-stage prototype sensor used Si technology and consisted of a two-wafer design where the teeter totter etching and the capacitor gap were formed by porous Si etching techniques.

Keywords: Analysis, Surface, Tribological, Adhesion, Chemical

379. K-Map Characterization of InGaSb Buffer Layers and InAsSb/InSb Superlattices Grown on InSb

FY 1992
$150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Reciprocal space maps (K-MAPs) and two-theta X-ray diffraction spectra (K-MAP projections) have been used to characterize the structure and composition of a variety of InGaSb buffer layers and InAsSb/InSb strained-layer superlattices (SLS) grown on InSb substrates. The initial goal of the K-MAP measurements was to assess the amount and reproducibility of strain relaxation that could be obtained by MBE growth of multilayer InGaSb buffer layers. These strain-relaxed buffers are now being used as templates for the growth of InAsSb/InSb SLSs intended for use as long-wavelength infrared detectors. More recent K-MAP measurements have focused on the characterization of complete buffer/SLS sample structures with a primary goal of correlating growth parameters with the preparation of samples with superior electro-optical and structural properties. K-MAP results indicate that a linearly graded, 5-layer, In(.86)Ga(.14)Sb buffer structures can be routinely fabricated with a reproducible strain-relaxation of 78% by using a Be doping technique to activate dislocation formation and movement and suppress crack formation. K-MAP measurements indicate that the final in-plane lattice constant of these buffers is well matched to the average lattice constant of free-standing InAs(.2)Sb(.8)/InSb SLSs.

Keywords: Analysis, X-ray, MBE, Compound

380. Materials Testing for Use in Super Critical Water Oxidation of Mixed Waste

FY 1992
$1,548,000

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Simulated mixed radioactive and organic waste supplied by Westinghouse, Hanford has been oxidized in a supercritical water reactor to remove the organic component. The reactor materials have been analyzed for corrosion and fouling. The nuclear weapons complex has, for decades, been accumulating mixed radioactive and organic wastes that must now be disposed of. In order to apply standard immobilization technology, the organic component must be removed without releasing any radioactive components to the atmosphere. Supercritical water oxidation (SCWO) has the advantage of completely destroying the organic component in a fully enclosed system. The most difficult problem associated with this emerging technology is that it requires a high temperature, high pressure vessel that, when treating many of the most challenging wastes, is exposed to a highly corrosive environment. In addition, the reactor vessel can suffer from fouling as inorganic reaction products of feedstock oxidation precipitate due to local changes in physical and chemical conditions within the reactor.

Keywords: Analysis, Spectroscopy, X-ray, Surface, Microscopy

381. NMR Studies of Helium Bubbles in Aged Palladium Tritide — FY 1992 $279,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Temperature dependent NMR lineshape, relaxation, and coherence transformation measurements on Helium micro-bubbles within aged PdT have yielded bubble pressures, sizes, and He-metal interface characteristics. Large quantities of ^{3}He are created within tritium-containing materials by beta decay of tritium. This He can aggregate in clusters by migration and trapping. TEM studies of young metal tritides of Ti, Zr, Ta and Pd found 1-5nm inclusions, typically spherical, which were hypothesized to be bubbles containing high-density helium. Past nuclear magnetic resonance (NMR) studies of ^{3}He in PdT powders confirmed that the helium resides in highly pressurized bubbles by identifying the existence of liquid-solid He phase transitions in the 100-200 K temperature range. An understanding of the formation and characteristics of these bubbles is important to application of metal tritides as tritium storage and tritium handling materials.

Keywords: Analysis, Microstructure

382. Non-Destructive Characterization of Porous Si by X-ray Reflectivity — FY 1992 $220,000

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We have used X-ray reflectivity to determine the thickness, porosity and interfacial roughness of porous Si films. Since the observation of photoluminescence in the visible spectrum, porous Si has been the object of intense research. Two critical parameters for characterizing this material are the film thickness and the porosity. Even though this material has been studied for some time (for silicon-on-insulator technology), it has been difficult to determine these parameters non-destructively. We have recently used our technique of energy-dispersive X-ray reflectivity to determine if it can be used to characterize porous Si. A sample of porous Si was prepared by electrochemical etching. X-ray reflectivity allowed us to determine the film thickness (29.1 nm), porosity (45%), top surface roughness (0.7 nm) and porous Si/substrate interfacial roughness (2.7 nm). The thickness and porosity were in reasonable agreement with previous determinations from the same wafer obtained by destructive means. On a thicker sample (nominal thickness 400 nm), resolution was sufficient to determine the film thickness but not the porosity.

Keywords: Analysis, Silicon

383. Phase Identification by Electron Backscattered Kikuchi Patterns — FY 1992 — $135,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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A CCD based detector for electron backscattered kikuchi patterns has been developed and tested on a scanning electron microscope with excellent results. Phase identification is now a reality in the SEM. The existence of electron backscattered kikuchi patterns (EBKP) has been known for some time. These patterns are produced in the scanning electron microscope (SEM) by the inelastic scattering of backscattered electrons which are then diffracted as they exit the specimen surface. EBKPs have been detected by relatively conventional techniques which yield low quality patterns that are only suitable for orientation determination. The technique has been shown to be capable of analyzing areas as small as 50 nm, however the quality of the patterns have been of poor quality so that phase identification has not been possible. This work has been undertaken to develop an advanced detector for the collection of high quality EBKPs suitable for use in microstructural analysis through phase identification in the SEM.

Keywords: Analysis, Microstructure

384. Screened Rutherford Backscattering Cross Sections for Heavy Ion and Low Energy Backscattering Analysis — FY 1992 — $50,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Stephen R. Lee, (505) 844-7307

Ion scattering cross sections for heavy ion backscattering spectroscopy (HIBS) were experimentally and theoretically investigated. The quantitative accuracy of keV heavy-ion backscattering spectrometry (HIBS) as a high-sensitivity surface analysis technique depends directly upon our knowledge of the ion scattering cross section. HIBS is performed at energies well below 100 keV/m using relatively heavy ions (C, N, O or Ne) where the distance of closest approach for the scattered ion may lie near the K or L shell radius of the target atom. Thus, electronic screening effects are particularly important in determining the scattering cross section. Previous measurements and exact theoretical calculations of the screened cross section performed at ion energies relevant to HIBS analysis are in poor agreement. We have experimentally and theoretically revisited the transition from high-energy Rutherford scattering to low-energy electronically-screened scattering in order to determine appropriate cross sections for HIBS analysis.

Keywords: Analysis, Ion Beam, Silicon, Surface

385. <u>Sorting of Waste Plastics by Spectroscopy and Neural Networks</u> FY 1992
$27,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: M. Kathleen Alam, (505) 845-9621

A method has been developed using near infrared reflectance spectroscopy and neural networks to sort plastics for recycling purposes. Nine percent of the municipal solid waste stream is plastic. Yet only 1-2% of this stream is recycled, compared with a 25% recycling rate for the paper waste stream. The major impedance to plastic recycling is cost. In order for plastic recycling to be economically feasible, the recovered material must be less costly than the virgin feedstock and it must have virtually the same properties (tensile strength, resistance to cracking, moldability, etc.) as the original product. Although gains have been made in commingled recycling, the most value arises from the recovery of plastics as a high-grade, high-purity polymer. However, sorting of plastics into separate polymer streams is, at present, labor intensive and error prone. The purpose of the research has been to develop a non-invasive, rapid method for sorting post-consumer plastics, incorporating infrared spectroscopy and neural networks. Infrared spectroscopy provided the delineation needed to sort the plastics, since each plastic has characteristic absorption bands in the infrared region. Neural network software/hardware was used to correctly identify unknown polymer samples from the infrared data.

Keywords: Analysis, Spectroscopy, Chemometric

386. <u>Use of Parallel Beam Optics for X-ray Diffraction Studies of Thin Films</u> FY 1992
$255,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Parallel beam optics have been successfully used to study thin films using X-ray diffraction. This study required unraveling the defects of a long soller slit on the diffracted data. The use of long soller slits to make parallel X-ray beam optics for use in X-ray diffraction studies of thin films is a novel technique in laboratories employing sealed tube X-ray sources. Prior to our studies this technique was only tried using high powered sources such as synchrotron radiation. We are the first to use this technique in a laboratory environment using low power X-ray sources such as sealed tubes. This technique has the potential to be a widespread tool for studying polycrystalline thin films of all kinds. The use of parallel beam optics of this kind has been limited because of a lack in understanding the effects this optical arrangement has on the diffracted beam. When understood this technique can be used for studying phase assemblage, strain and crystallite size variations as a function of depth into thin films. It can also be used to study phase assemblages in very thin films (< 10 nm).

Keywords: Analysis, X-ray, Diffraction, Ferroelectric

387. Volatile Organic Monitor — FY 1992 $25,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Testing has begun on a continuous, portable gas chromatograph. A site at General Electric has been identified for testing the equipment. Control of air and water emissions by industrial operations has been vitally important for U.S. industry. Public concern for the environment, human health, and increasingly stringent federal, state, and local regulations are demanding that industry monitor effluents. In conjunction with the City of Albuquerque Environmental Health Department, a system will be developed to monitor gaseous emissions remotely and continuously. A gas chromatograph system was identified as the best sensor system for the project, given the levels of contaminants to be measured (1ppm or less). A system was purchased and work has begun on configuring the equipment for remote operation. The General Electric Plant in Albuquerque has agreed to allow testing of the device on their site. Two chemicals have been targeted for testing purposes—1, 1,2 trichloroethane and methylethyl ketone.

Keywords: Analysis, Chromatography

388. XRD Studies of PZT Thin Films — FY 1992 $91,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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XRD has been used to study crystallization and orientation of ferroelectric PZT films. Ferroelectric thin films are being studied as target materials for non-volatile memories. X-ray diffraction studies have been instrumental in studying processing parameters in the synthesis of PZT films and the electrode layers. Our work has been used to help understand the crystallization of perovskite phases and which parameters are key to limiting residual pyrochlore phases. Our work has shown that pyrochlore tends to remain at the surface of the film after most processing conditions. Also, in order to produce a crystallographically oriented film it is necessary to produce a well oriented electrode layer. Our work showed that (111) oriented platinum produced (111) oriented PZT and that (100) oriented platinum produced (100) oriented PZT. Since a (100) orientation was preferred, studies were done to perfect a technique which produced (100) oriented platinum electrode layers. X-ray diffraction was employed after all steps. Our work showed that developing a (111) oriented layer was the norm using most processing techniques and that producing a (100) oriented platinum layer proved to be difficult. Recently, a technique for developing (100) oriented platinum layers has been perfected by our customer.

Keywords: Analysis, Diffraction, X-ray, Ferroelectric

389. Fundamental Studies of Electronically-Stimulated Surface Processes — FY 1992 $150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Excitation of metallic, semiconductor, or insulator surfaces with electrons or photons often results in electronically-induced or *stimulated* chemical bond rupture, leading to desorption, molecular fragmentation, and even reactions. Stimulated surface processes are integral to *non-thermal materials modification* technologies such as ultraviolet and electron beam etching, plasma-surface interactions, and surface photochemistry. In addition, electron stimulated desorption (ESD) and photon-stimulated desorption (PSD) are *important surface analytical probes* because desorption yields and product distributions are very sensitive to the local electronic and geometric structure of the adsorbate/substrate complex. Stimulated processes are essentially the conversion of electronic excitation energy into nuclear kinetic energy. Thus, fundamental studies of simulated surface processes have concentrated on the elucidation of the localized excitations (both core and valence level) and the energetics of the desorption/dissociation/reaction products. Sandia's lead role in this effort has been advanced considerably by the application of laser spectroscopy as a *quantum-specific* probe of neutral gas-phase products.

Keywords: Metals, Surface, Modeling, Corrosion

390. Effects of Excitation Symmetry on Parallel-Plate RF Discharges — FY 1992 $542,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Paul A. Miller, (505) 844-8879

Performance of a laboratory plasma reactor was studied as a function of symmetry of electrical excitation. Symmetric excitation reduced harmonic generation, increased discharge impedance, and improved etch rate and uniformity. A laboratory plasma reactor known as the GEC RF Reference Cell has been studied at many different laboratory. Studies have emphasized both basic discharge physics and industrial problems of the microelectronics industry. The reactor has equal-area top and bottom electrodes contained in a grounded metallic vacuum chamber. It was desired to develop a mode of operation in which the two electrodes are both powered with equal-amplitude rf signals that are out of phase by 180∞. This mode would minimize interactions of the discharge with the chamber walls and make the plasma more one-dimensional (1-D) in nature, thereby enhancing comparisons with 1-D theoretical analyses. We developed electrical methodologies and circuitry that achieved this "symmetric" mode of excitation.

Keywords: Semiconductor, Plasma, Etching

391. Elimination of Chamber-to-Chamber Differences in Industrial Plasma Reactors

FY 1992
$342,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Paul A. Miller, (505) 844-8879

Workers in the microelectronics industry frequently report that seemingly identical chambers of rf-excited plasma-processing reactors do not perform identically when using the same process recipe. This situation hurts quality and throughput of microelectronics production lines. The same problem existed with the Drytek Quad reactors in Sandia's Microelectronics Development Laboratory. In recent work with laboratory reactors, we had developed diagnostics and methodologies relating to the rf circuitry that energizes plasmas in reactors. We felt that application of the laboratory techniques to the industrial-reactor problem might help cure it. A key feature of plasmas is electrical nonlinearity which causes generation of harmonics of the excitation frequency. These plasma-generated harmonics can interact with the electrical circuits (cables, matching networks) that couple energy to the plasma. We found that impedances of electrical circuits at harmonic frequencies were not identical from chamber to chamber because of varying stray capacitances and cable lengths that were not controlled by the manufacturer. When the circuits were modified to eliminate the impedance differences at harmonic frequencies, the chamber-to-chamber differences vanished.

Keywords: Semiconductor, Plasma, Etching

392. Femtosecond Spectroscopy of Optical Materials

FY 1992
$365,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
SNL Contacts: Michael B. Sinclair, (505) 844-5506

Laser induced absorption in ferroelectrics and conjugated polymers, as well as transient emission from surface emitting lasers have been studied using a newly developed femtosecond optical spectrometer. Investigations of the transient optical response of materials to ultrafast laser pulses are necessary in the development of photonic materials. Measurements of this type can determine if the photo-induced absorption or refractive index change of a material is sufficient to allow its use in an optical switching device. More importantly, these measurements yield critical information about the nature of the optical properties of the materials under study. This information is essential if better materials are to be developed. A new femtosecond spectrometer has recently been completed which allows us to perform these investigations. The spectrometer consists of a high power femtosecond laser system, a continuum (white-light) generator, and a CCD spectrograph. Depending on the optical configuration, the spectrometer may be used to study photo-induced absorptions and refractive index changes with a temporal resolution of ~100 fs. Alternatively, it can be used to investigate laser induced emissions with a temporal resolution of ~1 ps.

Keywords: Semiconductor, Laser, Compound

393. Particle Formation, Transport and Detection in Plasmas — FY 1992 $320,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Optical diagnostic techniques measure the formation, distribution, and motion of particles in plasmas. Instrumentation detects in situ generated particles in reactive ion etch systems. The formation of particles in a glow discharge is a poorly understood phenomenon with important consequences. In microelectronic fabrication, these particles have been implicated as one of the major sources of contamination resulting in reduced yield. Our research has focused on optical diagnostics for detecting and measuring particles, studies on the formation and transport of particles in low-temperature radio frequency plasmas, and the role of contaminants in the nucleation of particles. We have demonstrated that monochromatic 2-D imaging can be used for measuring spatial profiles of particles in plasmas. A plane of laser light passing through the plasma is scattered by particulate materials. Scattered laser radiation is imaged in a direction perpendicular to the laser beam. The time evolution of the particle distribution is used to study the growth, charging, and motion of particles.

Keywords: Semiconductor, Plasma, Ion Implantation, Etching, CVD

Lawrence Livermore National Laboratory

Materials Preparation, Synthesis, Deposition, Growth or Forming

394. Inorganic Aerogels FY 1992 $200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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The approach is to perform a controlled hydrolysis of metal alkoxides such that the partially hydrolyzed chemical can be limited from further condensing reactions by using an appropriate solvent diluent. Subsequently, this pre-hydrolyzed compound can either be further diluted to make very low density sol-gels, or gelled directly for higher densities. Aerogels are obtained by super-critical solvent extraction of the wet gels.

We have synthesized several new metal oxide aerogels utilizing the above process; titania, germania and neodymia. We have also synthesized aerogels of mixed compositions of these oxides with silica. We successfully synthesized ultralow-density aerogels (i.e., total density less than 50 mg/cc) of silica doped with other metal oxides including iron, aluminum and zirconium. We have also characterized new silica aerogels which were synthesized from preformed, cube-silica building blocks.

Aerogels have unusual optical, thermal, and acoustic properties due to the ultrafine porous microstructure. Applications for these materials include target material for direct drive laser fusion experiments, thermally insulated windows and skylights, and as collector material for hyper-velocity microparticle capture.

Keywords: Inorganic Aerogels, Sol-Gel, Laser Fusion Targets

395. Organic Aerogels FY 1992 $325,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: R. W. Pekala, (510) 422-0152

The aqueous polycondensation of (1) resorcinol with formaldehyde or (2) melamine with formaldehyde are two proven synthetic routes for the formation of organic aerogels. The former materials can also be pyrolyzed in an inert atmosphere to give vitreous carbon aerogels. The structure and properties of organic aerogels are analogous to their inorganic (e.g., silica) counterparts. In general, these materials have continuous porosity, an ultrafine cell/pore size (<50 nm), high surface area (400-100m^2/g), and solid matrix composed of interconnected colloidal-like particles or fibrous chains with characteristic diameters of 10 nm.

A major advantage of organic aerogels is their low Z (atomic number) composition. Recently, we showed that resorcinol-formaldehyde aerogels are even better insulators than silica aerogels when measured under ambient conditions. A record low thermal conductivity of 0.012 W/m-K was obtained at a density of 0.16 g/cc.

Carbon aerogels are of particular importance because they are the first electrically conductive aerogels to be synthesized. The low resistivity of the aerogel network and the large surface areas per unit volume are being exploited in supercapacitors that have both high power (7.5 kW/kg) and energy densities (4-20 Whr/kg). Carbon aerogels are also being investigated as candidates for the storage of hydrogen and methane.

Keywords: Organic Aerogels, Sol-Gel, Supercapacitors, Thermal Insulation

396. Nanostructure Laminates — FY 1992 — $264,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: Troy W. Barbee, Jr., (510) 423-7796

Multilayers are man-made materials in which composition and structure are varied in a controlled manner in one dimension during synthesis. Individual layers are formed using atom by atom processes (physical vapor deposition) and may have thicknesses of from one monolayer (0.2 nm) to hundreds of monolayers ($>$100 nm). At this time more than 75 of the 92 naturally occurring elements have been incorporated in multilayers in elemental form or as components of alloys or compounds. In this work deposits containing up to 225,000 layers of each of two materials to form up to 500μm thick samples have been synthesized for mechanical property studies of multilayer structures.

These unique man-made materials have demonstrated extremely high mechanical performance as a result of the inherent ability to control both composition and structure at the near atomic level. Also, mechanically active flaws that often limit mechanical performance are controllable so that the full potential of the structural control available with multilayer materials is accessible. Systematic studies of a few multilayer structures have resulted in free-standing foils with strengths approaching those of whiskers, approximately 70 percent of theory. Also, new mechanisms for mechanically strengthening materials are accessible with nanostructure laminates.

Keywords: Thin Films, Multilayer Technology

397. Sol Gel High Reflectors FY 1992
$325,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: I. M. Thomas, (510) 423-4430

We are developing high damage threshold sol gel multilayers to use as mirrors for advanced high power laser applications. Previously, we demonstrated high damage thresholds using hafnia-silica multilayers. During this past year, we demonstrated that the use of polyvinyl alcohol (PVA) as a binder in the hafnia produces a superior HR. PVA increases the index contrast between the hafnia and silica, permitting the mirror to be made with as few as 20 layer pairs, instead of the usual 36. The damage threshold was unaffected. These sol gel coatings do not require laser conditioning to reach their ultimate damage threshold, in contrast to electron beam coatings. In addition, the mirror is much more durable because of the PVA binder. In terms of mechanical strength, the sol gel multilayers are approaching the strength of conventional electron beam coatings.

We also demonstrated that these sol gel coatings can be applied to large, rectangular substrates using a meniscus coater. In the past, sol gel multilayers have been applied using a spin coater which is not ideally suited for the rectangular substrates envisioned for advanced laser architectures in the ICF Program. Meniscus coaters have been utilized to apply simple photoresist coatings, but nothing as complex as a sol gel multilayer. We borrowed a meniscus coater from Specialty Coatings Systems (which has a patent on the applicator tube) to demonstrate that high quality sol gel mirrors could be fabricated using this approach. We are in the process of procuring a full-size prototype machine for continued development.

Keywords: Sol Gel, Mirror, Coating, Multilayer

398. Modified Phosphate Laser Glass Composition FY 1992
$400,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
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Laser glass developed for the Nova laser has performed very well. It has a reasonably high emission cross-section, decent thermal-mechanical properties, and a high enough platinum solubility to meet the ICF Program's damage threshold requirements. Nonetheless, we believed that the composition could be modified in such a way as to improve all these aspects simultaneously. We started this effort to improve the performance of this glass in FY91, working with Schott Glass Technologies, which owns the patent for LG-750.

By increasing the phosphate content and altering the glass network and modifier constituents, we successfully demonstrated that the glass could be improved in all respects. The emission cross section is about 10 percent higher than LG-750. The nonlinear refractive index and emission bandwidth, both of which degrade laser performance, are about 10 percent lower. The platinum solubility is almost double that of LG-750. The thermal-mechanical properties are somewhat improved. Full-scale testing is all that remains to separately examine other manufacturing issues which can only be addressed at full-size.

Keywords: Laser Glass, ICF

399. KDP Growth Development FY 1992
$500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: J. J. DeYoreo, (510) 423-4240

Potassium dihydrogen phosphate (KDP) and its deuterated analog (KD*P) are important nonlinear crystals used both for frequency conversion as well as for a large Pockels cell. These crystals are very expensive, due in part to the very long times required to grow large boules (1-2 years) and the cost of D_2O for growing KD*P. We are developing alternative growth techniques which can either increase the growth rate of these crystals or reduce the volume of D_2O in the crystallizer.

In FY92 we made substantial progress in the design and fabrication of advanced prototype crystallizers. We designed and built a "forced flow crystallizer" which should substantially increase the growth rate of KDP and KD*P crystals. Testing in this crystallizer will begin in FY93. We also build a "conic downflow crystallizer" which dramatically reduces the volume of solution (mostly D_2O) necessary to grow a KD*P crystal. Initial results from the first growth cycle were promising. We have since redesigned the solution delivery system to more uniformly encapsulate the growing crystal; these tests will continue through FY93.

Keywords: KDP, Nonlinear Crystals, Crystallization

400. Advanced Finishing Development FY 1992
$700,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: J. S. Taylor, (510) 423-8227

Advanced optical fabrication methods will almost certainly be utilized to finish optical surfaces in advanced ICF laser architectures. We are working to understand the effect of finishing parameters on optical surface characteristics, including subsurface damage and resulting damage threshold. We are also developing rapid grinding and polishing methods which will simultaneously reduce the cost of fabricating optical surfaces while maintaining the high quality necessary for high power laser applications.

In FY92 we completed installation of about 2,500 square feel of clean room space with temperature and acoustic vibration control suitable for fabrication of very high quality optical surfaces. We installed a 9-inch double-sided grinder to begin development of rapid grinding process. We have demonstrated (at Kodak) that ion beam figuring may be used to attain the final figure on laser glass, which has tremendous potential to reduce the cost of finishing laser glass.

Keywords: Optical Finishing, Ion Beam Figuring, Laser Glass

401. Synthesis of Energetic Molecules — FY 1992 $500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: C. Coon, (510) 422-6311

A synthetic effort is underway to develop new energetic molecules that have a combination of greater performance and greater insensitivity than currently available. Target materials include but are not limited to: derivatives of amino-nitrotriazole, amino-nitro heterocyclics, variations of triamino-trinitrobenzene, dinitrobiguanide and biguanide nitrate, molecular complexes, and fuel-oxidizer solutions.

Keywords: Explosive, Synthesis

402. Doped Polymers for ICF — FY 1992 $500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contacts: R. Cook, (510) 422-3117 and G. E. Overturf III, (510) 422-7280

This program is developing covalently doped polystyrene derivatives for use as spectroscopic tracers in direct drive laser fusion experiments. The doped polymers are formed into small spherical shells that serve as the mandrel around which the ICF capsule is constructed. The dopant atoms should be atomically dispersed and thus must be covalently incorporated into the structure of the polymer. We have succeeded in producing soluble polystyrene derivatives doped with Cl, Br, I, Fe, and Cr at levels up to 3 atom percent. Current work focuses on a Ti-doped polymer.

Keywords: Polymers, Dopants, Laser Fusion Targets

403. Plasma Polymer Coating Development FY 1992
$900,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contacts: S. Letts, (510), 423-2681, R. Brusasco, (510) 422-3111 and R. Cook, (510) 422-3117

This program is developing the capability to produce ultra-smooth plasma polymer coatings on plastic microshells for use as targets in ICF experiments. These coatings are formed by reacting trans-2-butene monomer and hydrogen gas in an RF plasma created by a vertical helical resonator reactor operating at ≈ 40 MHz. This coating technology has demonstrated the ability to prepare coatings with surface roughness of the order of 50Å peak to valley. Our goal is to make coatings of this quality a routine procedure by investigating the fundamental mechanisms of surface roughening. We have the capability of measuring the plasma parameters within the coater which affects the kinetics of the polymerization process. Further, we have incorporated computer automated data collection and analysis capabilities in our newly constructed plasma polymerization reactor in order to study the effects of process changes and transients on the coating process.

Keywords: Plasma Polymer, Laser Fusion Targets, Surfaces, Coatings

404. Polymer Films by RF Sputtering FY 1992
$125,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: E. Hsieh, (510) 422-0753

We have experimented with a versatile RF sputtering approach to produce polymer films for ICF applications. In addition to producing pure polymer films, polymers can be doped with metallic elements using a co-sputtered configuration. We have sputtered Acrylic, Polypropylene, Polystyrene, Parylene-N and PTFE (Teflon) at a reasonable deposition rate of $1\mu m/hr$. The IR absorption measurements showed that the sputtered films compared favorably with that of the bulk materials. Most likely the polymer films' surface smoothness will be in the few 100Å range as evident in an ATM measurement on an acrylic film. As expected, the dopant level of co-sputtered polymer film has limitations, similar to the plasma polymerization process, beyond which the metallic atoms formed distinct crystallites. The limits on dopant levels are being explored.

Keywords: Polymers, Dopants, Laser Fusion Targets

405. Excimer Laser Micromachining

FY 1992
$30,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: R. Wallace, (510) 423-7864

This program is developing methods of using pulsed UV excimer laser photoablation techniques to micromachine plasma polymer coatings. Surface perturbations on microshells result in growth of Rayleigh-Taylor (RT) instabilities during the acceleration phases of an ICF implosion. To test current theories of RT growth and its effects on target performance, we are investigating methods of applying known perturbations, depth and mode distribution, to smooth capsules. Due to very high RT growth rates, excimer laser ablation etch rates in the range of a few hundred Ås are required. Micromachining at this level pushes the limits of excimer laser ablation due to low absorbtivity and incubation processes in polymer coatings.

Keywords: Excimer Laser Ablation, Plasma Polymer, Laser Fusion Targets

Materials Structure and Composition

406. Electronic Structure and Dynamic Properties in Superconducting Oxides

FY 1992
$500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: M. J. Fluss, (510) 423-6665

We have designated three experimental task areas: materials synthesis and properties, electron structure, and thin films, all of which are guided and supported by a broad theoretical task in band structure, ligand field effects, and pairing mechanisms. The selection of these activity areas provides the basis for a flexible, but focused program which is contributing to the solution of critical problems in the search for an understanding of the oxide superconductors. Additionally, the thrust is now focusing on opportunities for utilization of the new superconductors in future LLNL programs and has explored processing and applications oriented activities. The robust heteroepitaxy capability is leading toward the development of ultra high speed nanowatt digital logic "gates."

A variety of theoretical approaches have been used to study the nature of the charge carriers, the pairing mechanism and the formation of a superconducting state in superconducting oxide materials. The properties of paired charge carriers of fractional charge in a superconducting mechanism were investigated as was the condensation of a two dimensional Bose-Coulomb gas. Calculations of the electronic structure of superconducting oxides were done based both on band theory and Hartree-Fock with configurational interactions. Both of these approaches suggested the importance of specific hole states in the superconducting mechanism. Results that directly describe laboratory experiments were obtained from the Hartree-Fock method for Mossbauer measurements and from linear combination of atomic and molecular orbitals in a cluster approach to describe electronic structure measurements made with positrons on several materials.

Keywords: Superconductors, Electronic Structure, Positron Annihilation

407. Electronic Structure Study of the Thermodynamic and Mechanical Properties of Al-Li Alloys — FY 1992 $117,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: A. Gonis, (510) 422-7150

During the last fiscal year we effectively completed our study of Al-Li alloys. We completed and tested codes which allow the treatment of clusters of atoms in an alloy in terms of neutral Wigner Seitz spheres. Along with previously developed codes for the treatment of single atoms in an alloy, these codes allow us to study the effects of charge transfer in substitutionally disordered alloys. These effects can be significant in alloys of light elements and with extended charge distributions, such as Al-Li. We also developed, tested, and applied codes for the calculation of charge-density contours in substitutionally disordered alloys. Although charge density contours are plotted almost routinely for ordered systems, this is the first time that such contours have been plotted for disordered alloys.

Our studies of Al-Li alloys have yielded strong indications that the metastable ordered phase, thought to play a role in determining the mechanical behavior of Al-Li alloys, is caused by local strain fluctuations due to the large mismatch of the lattice parameters of the pure metals.

Keywords: Electronic Structure, Computation, Alloy

Materials Properties, Behavior, Characterization or Testing

408. Synchrotron Radiation-Based Materials Science — FY 1992 $700,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contacts: J. Wong, (510) 423-6385

The objective of this thrust area is to understand the role of atomic and electronic structures in determining the physico-chemical properties of materials and their processing. The tasks defined in this thrust area take advantage of the various unique characteristics of synchrotron radiation such as high intensity, high collimation, high polarization and broadband tunability from VUV to hard X-ray to probe the structure of matter on an element-selective basis at different levels. The research areas involve both an expansion of our existing capabilities in material characterization using these powerful photon sources and development of new capabilities (a) to probe dilute species in bulk materials, (b) to determine the geometric and electronic structure of thin films and nano-scaled materials, (c) to unravel chemical dynamics of high temperature reaction systems and phase transformation *in situ* in real time down to the milliseconds range, (d) to develop a novel quick-scanning EXAFS capability on our PRT X-ray beamlines at both SSRL and NSLS to study time-dependent phenomena in the second and sub-second time scale, and (e) to develop new soft X-ray monochromator materials for programmatic needs and the next generation synchrotron sources.

Keywords: Synchrotron, X-ray Diffraction, EXAFS

409. Structural Transformation and Precursor Phenomena FY 1992
$230,000

DOE Contact: C. B. Hillard, (301) 353-3687
LLNL Contact: P. E. A. Turchi, (510) 422-9925

Order and stability in bcc-based Fe-Cr alloys were examined with an ab-initio methodology. Around equi-composition, it was found that although FeCr ultimately phase separates, a metastable phase may exist at low temperature. This alloy becomes a proto-typical case where a competition between alloying effect (in favor or phase separation) and local chemical order may compete. This work was extended to a series of equiatomic bcc-based alloys, including FeV, FeTi, TiCr, TiV, TiMn, VCr. This study clarifies, at an atomic level, the possible role played by ternary additions in stabilizing complex phases or ordered superstructures which are not observed in the binary combinations.

On the experimental side, inelastic phonon scattering as well as diffuse elastic scattering close to the Bragg reflections (Huang scattering) on a $Fe_{0.53}Cr_{0.47}$ single crystal have been performed. In the area of surface physics, a LEED investigation of the possible surface-induced bcc to σ transformation in oriented FeCr single crystals along (110) and (111) has been performed at SNL (Albuquerque). This study precluded a more quantitative investigation based on X-ray grazing incidence diffraction, at the NSLS at Brookhaven. The results are still currently analyzed. This study will help understanding structural transformations from simple to complex phases which occur in a broad class of advanced materials.

Keywords: Electronic Structure, Phase Stability, Short Range Order, Complex Alloys, Surface Properties

410. Underwater Explosive Energetics FY 1992
$50,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: W. C. Tao, (415) 423-0499

We are developing a model to address the hydrodynamic and thermodynamic processes during the combustion of aluminum in water. The exothermicity gained from underwater metal combustion and the resulting impulse have been found useful in various applications ranging from underwater explosives to exciting acoustic projectors. Our comprehensive model incorporates heat transfer, chemical kinetics, and bubble hydrodynamic, and therefore, requires data from pressure-time history, temperature, and spatial distribution of chemical species measurements. We have modified our Ultrafast Microphotography System to allow for both near-field and far-field pressure-time history measurements. The results clearly show that we can characterize the contribution to bubble expansion from thermal and chemical sources. The feasibility of applying the energy derived from the combustion of large amounts of aluminum in water to drive acoustic projections is currently being investigated.

Keywords: Underwater Explosives, Reactive Metals

411. Composite Explosive Energetics FY 1992
$150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: W. C. Tao, (510) 423-0499

Using Fabry-Perot interferometry techniques, we have determined the early time rate of energy release from detonating PETN and TNT explosives filled with 5 and 10 wt% of either 5 μm or 18 μm spherical aluminum (Al) particles. From the measured particle velocity data, we are able to infer the reaction rate of aluminum with the detonation products, and calculate the extent of reaction 1-3 μs after the detonation. We observed that a substantial portion of the aluminum metal in all of the PETN and TNT formulations reacted within the timeframe of the one-dimensional experiment. In the PETN formulation filled with 5 wt% of 5 μm aluminum, all of the metal reacted within 1.5 μs, resulting in an increase of 22 percent in energy compared to pure PETN. A reactive-flow hydrodynamic model based on the Zeldovich-von Neumann-Doring (ZND) description of the reaction zone and subsequent reaction product expansion (Taylor wave) is used to interpret the reaction rate of the aluminum particles with detonation product gases. The diffusion-controlled reaction mechanism for aluminum and the global kinetic parameters used in the model have been found to be consistent for all of the PETN and TNT formulations.

Keywords: Composite Explosives, Metal Loading, Reactive Flow Modeling

412. Low Vulnerability Composites Explosives (LOVEX) FY 1992
$200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: D. M. Hoffman, (510) 422-7759

We have an ongoing program to develop explosives with significantly lower vulnerability to battle accident environments (bullets, fires) than conventional explosives such as TNT or Comp-B (RDX/TNT). The challenge lies in accomplishing this without sacrificing performance and increasing costs. We have developed a composite explosive which is comprised of inexpensive fillers such as the crystalline explosive powder hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX), sodium nitrate ($NaNO_3$), aluminum metal powder (Al), and a low-modulus polymeric binder system. The binder system uses polycaprolactone (PCL) as the polymeric component and trimethylol ethane trinitrate/triethyleneglycol dinitrate (TMETN/TEGDN) as an energetic liquid which acts as a plasticizer for the polymer. We have tailored a specific formulation, RX-35-BX, for use as the explosive fill in the Navy's Advanced Bomb Family Program (AFB). The RX-35-BX formulation is a high-performance, easily processed, low-vulnerability, and low-cost composition which passes all of the Navy's main-charge explosive qualification tests. The qualification documentation, UCRL-UR-110363, "LOVEX RX-35-BX: A Low-Vulnerability, High-Performance Explosive for Main Charge Applications," June 1, 1992, Lawrence Livermore National Laboratory, Livermore, CA, was submitted to the Navy for consideration of RX-35-BX as an Advanced Bomb Family candidate explosive. This report discusses the formulation, processing, rheology, and cure behavior of this composite explosive as well as performance, vulnerability, and cost aspects.

An additional aspect of the program is to gain an understanding of the effect of constituents on sympathetic detonation. Over the next year, several tests will be performed with 6" artillery shells to determine the effect of fuel/oxidizer ratios, particle size, and RDX content. Completion of this effort should allow optimization of the composite formulation for performance without a sympathetic detonation hazard in a particular hardware configuration.

Keywords: Insensitive Explosives

413. Fundamentals of Explosive Vulnerability FY 1992
$1,000,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: W. Tao, (510) 423-0499

The purpose of these studies are to develop models that can describe explosive response to abnormal environments. The focus is on multiple stimuli such as combined thermal-mechanical impulses. These results are used to model safety and vulnerability aspects of specific munitions.

Keywords: Explosives

414. Very High Energy Density Materials FY 1992 $100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: R. L. Simpson, (510) 423-0379

An experimental effort was initiated with the epsilon form of CL-20 to examine its sensitivity and performance properties. An ϵ-CL-20 analog to LX-14 was formulated. Data reported include: impact, spark, and friction sensitivities, differential scanning calorimetry and thermal gravimetric analyses, and one-dimensional-time-to-explosion (ODTX). Both the pure alpha and epsilon phases were examined as well as the epsilon based formulation. To examine questions concerning the effect of mechanical confinement on the ODTX results vented experiments were carried out. Performance measurements included a 24mm cylinder test, small scale metal acceleration experiments and particle velocity examination using impedance matched crystals.

Keywords: Explosives Characterization

415. Liquid Actinide Embrittlement of Refractory Metals FY 1992 $120,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: J. S. Huang, (510) 422-5645

Following our previous study on the embrittlement of Groups VB and VIB metals and alloys by liquid U, we continued to study the embrittlement characteristics of these metal and alloys by liquid Pu. It was found that Ta, Nb and W are embrittled by liquid Pu while V and Cr were not. It appears there is not a universal relationship between phase diagram and embrittlement susceptibility. For example, even though the phase diagrams of the Ta-Pu and V-Pu systems are similar, the behaviors of V and Ta in Pu are extremely different.

The embrittlement of Ta, Nb and W by liquid Pu is similar to that observed in the Ta-U and W-U systems, which is caused by rapid grain boundary wetting of the liquid phase. We also studied the behavior of binary V-Ta alloys. The addition of 10 to 20 wt. percent of Ta made the V based alloys embrittled by Pu. TEM investigation indicated no significant change in secondary grain boundary phase or in grain boundary angle of these alloys. It is suggested that the embrittlement is due to changes in the grain boundary energy. Grain boundary grooving experiments are being planned to study the relative change of grain boundary energy/surface energy ratio caused by the addition of Ta in V. This could possibly shed the light on the embrittlement mechanism in these metals and alloys.

Keywords: Deformation, Fracture, Actinide

416. The Formation of Bands of Ultrafine Particles During Rapid Solidification — FY 1992 — $67,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: J. W. Elmer, (510) 423-4655

We have shown that rapid solidification processing of Al-Be alloys can be used to produce a dispersion of ultrafine (10nm) Be-rich particles that are periodically arranged in banded arrays that lie 25 nm apart and are parallel to the solidification front. This unique banded microstructure has been modeled using a numerical method that incorporates the effects of liquid phase nucleation (LPN) to explain the formation of this microstructure. Similar ultrafine particle microstructures have been observed in Al-rich and Cu-rich alloy systems such as Cu-Cr, Cu-Nb, Cu-O, Al-In, and Al-Pb. These alloy systems have phase diagram characteristics similar to Al-Be, which indicates the likelihood of metastable liquid immiscibility. We are now investigating the possibility of LPN-induced ultrafine particle formation in the Cu-O and Cu-Nb alloy systems to determine the necessary composition and solidification-rate conditions required to form banded ultrafine particle microstructures.

Keywords: Rapid Solidification, Banded Microstructures, Ultrafine Particles, Liquid Phase Nucleation, Liquid Immiscibility

417. Failure Characterization of Composite Materials — FY 1992 — $330,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: Scott Groves, (510) 422-1331

We have characterized the three-dimensional performance of continuous fiber composite materials. This project has included data on three-dimensional failure and elastic properties as well as the dynamic strength behavior of these materials. Our accomplishments include the development of a unique multiaxial testing system for composite tubes, high strain rate testing fixtures, and enhancements to a drop tower for instrumented impact testing. We have also developed a three-dimensional orthtropic finite element code that will permit detailed ply-by-ply stress analysis.

Keywords: Composite Materials, Fibers, Failure Testing

418. Modeling Superplastic Materials FY 1992
$260,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: Donald Lesuer, (510) 422-9633

This project is developing a model that accounts for structural changes during superplastic flow and their subsequent influence on stress-strain behavior and deformation localization. We have studied the kinetics of strain-enhanced grain growth and cavitation and have assessed the ability of two rate dependent constitutive laws to predict the stress-strain behavior when the kinetics of microstructural evolution are included. The ability of instability criteria to predict strain localization during superplastic deformation has been assessed.

Keywords: Superplastic Flow, Deformation Modeling

419. Interfaces, Adhesion, and Bonding FY 1992
$500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: Wayne E. King, (510) 423-6547

We have developed a unique capability for calculation of the electronic structure at interfaces, where symmetry is reduced compared with the bulk. Specifically, the method, called the real-space multiple-scattering theory (RSMST) can treat interfaces and include the effect of atomic relaxation at the interface. We have coupled this method with the semi-empirical embedded atom method (EAM) which uses modified two-body potentials with molecular dynamics, molecular statics, or Monte Carlo techniques to determine atomic rearrangements.

Our experimental effort is producing results that are directly comparable with theoretical calculations. We are investigating planar metal/metal interfaces and metal/ceramic interfaces (in anticipation of improvements in the theory) of well defined misorientations. In order to span the entire range of length scales described above, macroscopic bicrystals a few millimeters thick, with interfacial areas on the order of a square centimeter will be required. In order to obtain such bicrystals, we plan to employ the diffusion bonding approach. An ultra-high-vacuum diffusion bonding machine has been developed in parallel with this research project.

Keywords: Interfaces, Bonding, Electronic Structure

420. Laser Damage: Modeling and Characterization FY 1992
$500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: M. R. Kozlowski, (510) 294-5637

We have been working to understand the damage mechanism in thin film coatings used on Nova for a number of years, with the ultimate goal of improving the damage threshold in coatings for future laser systems. In recent years, we have utilized atomic force microscopy (AFM) to characterize laser damage as well as the laser conditioning process which allows coatings to sustain higher laser fluences. We've shown that damage threshold correlates with the density of pre-existing defects, and that nodular defects often damage but that the craters (produced by nodules "popping" out prior to laser irradiation) do not damage.

This past year, we initiated a theoretical study of intense electromagnetic waves with "typical" nodular defects in a simple quarter-wave dielectric mirror coating. The model results demonstrated large field enhancements are produced by these defects, which are composed of the same dielectric material as the coating materials. Previously, it was thought that defects had to be absorbing (either carbonaceous or non-stoichiometric material) to produce the large field enhancements which produce damage. We will use these results to help modify the coating process to avoid the formation or incorporation of these defects into the coating, thereby improving the damage threshold.

Keywords: Coatings, Atomic Force Microscopy, Laser Damage

421. KDP Characterization FY 1992
$300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: J. J. DeYoreo, (510) 423-4240

We require very large, high quality crystals of potassium dihydrogen phosphate (KDP) and its deuterated analog (KD*P) for present and advanced high power laser in the ICF Program. The use of these crystals can be limited by strain which induces anomalous birefringence. In FY92 we embarked on a theoretical and experimental campaign to identify the sources of strain in these crystals and to measure strain birefringence in a large number of crystals with a known growth history. Our goal is to incorporate these results into development activities directed to improving the growth process in order to increase the overall yield of crystal plates from a boule.

We've demonstrated conclusively that the strain patterns present in "first-grown" material persist through much of the growth, but that the magnitude of the strain decreases in the growth direction. This result is consistent with strain induced by dislocations present in the seed crystal used to initiate growth. Our present work is focused on determining the effect of growth parameters on strain, including growth rate, impurities, the seed-mount, and short-term fluctuations in supersaturation (arising from process imperfections).

Keywords: KDP, Strain, Crystal

422. Damage Testing — FY 1992 $600,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: F. Rainer, (510) 422-4376

We maintain three lasers to measure damage threshold of optical materials at pulse durations of 3 and 10 ns, and at the first four fundamental wavelengths of Nd:YAG (1064 nm, 532 nm, 355 nm and 266 nm). We test and document several hundred samples per year, including coatings, crystals, glasses, and the effect of finishing processes on surfaces. This information is in turn utilized to help make important decisions in the development of these materials. The ICF Program maintains one of the world's largest databases of damage results for optical materials.

Keywords: Laser Damage, ICF

423. Transferable Insensitive High Explosives — FY 1992 $1,500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: M. Hoffman, (510) 422-7759

A materials development effort was initiated to develop an energetic material which meets DOE's insensitive high explosive criteria, has moderate energy, and can be transferred as a fluid over a wide temperature range. A variety of approaches are being pursued including slurried materials, and single and multiple component liquids. Fundamental studies on initiation, performance, and rheological behavior are being carried out.

Keywords: Explosive

424. Energy Transfer Dynamics in Energetic Materials — FY 1992 $300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: A. Ruggiero, (510) 423-1020

When an energetic material is shocked, optical phonon energy is up-converted to intramolecular vibron modes which ultimately leads to molecular dissociation. The anharmonic potential and energy transfer rate determines, in part, a materials sensitivity. These dynamics are being probed using LLNL's unique femtosecond laser capability and are being modeled using a molecular dynamics approach.

Keywords: Explosive, Laser, Dynamics

425. Processing-Structure-Property Correlations in Laminated Metal Composites

FY 1992
$171,000

DOE Contact: C. B. Hilland, (301) 353-3687
LLNL Contact: Chol K. Syn, (510) 534-8226

Alternating layers of metals (e.g., Al 5182) and metal matrix composites (e.g., Al 6061-25 vol.% SiC) are to be press-bonded with heavy deformation to form laminated metal composites with strength, toughness, and other properties far superior to those of the constituent materials. Interfacial bonding strength and microstructure will be correlated with the processing parameters and mechanical properties and the mechanical properties will be modelled using the Laboratory's finite element codes.

Keywords: Laminated Metal Composites, Deformation Bonding

Instrumentation and Facilities

426. Scanning Tunneling Microscopy (STM) and Atomic Force Microscope (AFM) as a Detector

FY 1992
$320,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: W. Siekhaus, (510) 422-6884

One vibrating needle AFM, built in conjunction with LBL, capable of operating both as STM and AFM has been used to study the forces applied when scanning tunneling microscopy is being performed on biological molecules in air. A commercial AFM has been used to (1) study the structure of mouse, bull, and human sperm-heads, determining the packing density and packing morphology of DNA in sperm-heads, (2) to study the relationship between surface morphology and laser damage threshold of sapphire surfaces and of anti-reflection and high-reflection optical coatings.

Keywords: Instrumentation and Technique Development, Defects, NDE, DNA, Laser Damage Threshold

427. Decontamination and Waste Treatment Facility (DWTF) FY 1992
($9,400,000)*

DOE Contact: G. J. D'Alessio, (301) 903-6688
LLNL Contact: R. Quong, (510) 422-7093

Construction of the DWTF line-item is scheduled to be completed by year-end 1998. The project started in FY 86. Through FY 91, 13 percent of the $70 million authorized funds has been spent. Originally completed design, environmental, and permitting documents will be revised to reflect significant changes in project scope and design waste streams. A major change is the deletion of the rotary kiln incinerator system. An alternative technology will be incorporated in the future when identified and demonstrated. The remainder of the project will continue to be executed. The revised DWTF consists of seven new buildings (91,000 square feet of new building space) plus equipment to process and treat radioactive, hazardous, and mixed wastes. The facility will occupy a 6-acre site within the Laboratory, replace existing outmoded facilities, and will greatly diminish the need for off-site treatment and disposal of waste generated by LLNL.

Keywords: Facilities, Waste Treatment

428. Pyrochemical Plutonium Process Development FY 1992
$9,000,000

DOE Contact: Lori Myers, (510) 422-3483
LLNL Contact: Mark C. Bronson, (510) 422-3061

The objective of this effort is the development of new pyrochemical processes for the recovery, purification, and conversion to metal of plutonium. This is being carried out in support of design of the next generation plutonium processing facility in the reconfigured nuclear weapons complex, and emphasized waste minimization, worker dose reduction, and construction cost.

During FY 92, development efforts were carried out in direct oxide reduction utilizing tilt-pour furnace technology, preparation of $PuCl_3$, evaporative separation of Am from Pu, recovery by hydride/dehydride, crucible development, electrorefining, advanced calcination technologies, advanced bottom pour casting, and electrowinning of Pu from PuO_2 utilizing Ca.

Keywords: Pyrochemical, Plutonium Processing, Plutonium Recovery

*Line-item construction project. It is not included in subtotal or total.

Los Alamos National Laboratory

Materials Preparation, Synthesis, Deposition, Growth or Forming

429. Actinide Processing Development — FY 1992 $1,350,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. F. Stevens, (505) 667-4414

The aim of this project is the development and characterization of fabrication processes and the study of new processing technologies for plutonium. Research involves casting, thermomechanical working, and stability studies. Measurements of resistivity, thermal expansion, magnetic susceptibility, and formability are made to evaluate fabrication processes and alloy stability.

Keywords: Radioactive Materials, Plutonium Alloys, Ductility, Thermal Expansion, Electrical Resistivity, Stability

430. Plutonium Oxide Reduction — FY 1992 $150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: K. Axler, (505) 667-4045

The thermodynamics of interactions among the components used in the pyrochemical processing of plutonium are determined along with the relevant phase relations.

Keywords: Radioactive Materials, Plutonium, Thermodynamics, Phase Diagrams, Direct Oxide Reduction, Electrorefining, Molten Salt Extraction

431. Low-Density, Microcellular Plastic Foams — FY 1992 $200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: P. Apen, (505) 667-6887

Microstructural polyolefin foams with densities between 0.01 g/cc and 0.2 g/cc are manufactured by a nonconventional foaming process. Foams are both open and close celled and have large surface areas. This process is being expanded to other polymeric materials for a wide variety of applications. Foams have cell sizes from 25μm down to the 1μm range depending on the process. Composite foams are being produced with submicron cell sizes while maintaining structural properties.

Keywords: Foams, Polyolefins, Polyurethanes, Silicones, Polyesters

432. Physical Vapor Deposition and Surface Analysis

FY 1992
$300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. Scott, (505) 667-7557

Physical vapor deposition, one electron beam sputtering, and dual ion beam sputtering are employed to produce materials for structural applications, corrosion resistance, optical properties, and thin film transducers. Materials being developed include doped, *in situ* laminates of aluminum and Al_xO_y having high strength and smooth surface finish. Also included are ion assisted deposition and ion sputtering onto various substrates for corrosion resistance to gases and liquid plutonium, reflective and anti-reflective coatings for infrared, visible, ultraviolet and X-ray wavelengths. Novel photocathodes are being made and evaluated by these processes.

Keywords: Coatings and Films, Physical Vapor Deposition, Sputtering, Ion Plating, Corrosion, Nondestructive Evaluation

433. Chemical Vapor Deposition (CVD) Coatings

FY 1992
$150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contacts: J. R. Laia and M. Trkula, (505) 667-0591

Chemical vapor deposition (CVD) techniques are used to deposit thin-film and bulk coatings of a wide variety of elements and compounds. Coatings are deposited by the following techniques: conventional flow-by, fluidized-bed, plasma-assisted, and chemical vapor infiltration. To support and enhance our basic CVD program, efforts are underway to study the fundamental nature of the CVD process, including *in-situ* diagnostics in the gas phase just above the substrate and modeling efforts to predict gas flows, reactor design, and chemical behavior within the CVD systems. Another collaborative effort at Los Alamos is attempting to synthesize organometallic precursors to deposit coatings at temperatures $<300°C$. Substrates coated by the CVD technique range from particles 2.0 μm diameter to infiltrations of fabrics a square meter in area.

Applications include nuclear and conventional weapons, space nuclear reactor systems (fuels and structural components), inertial confinement fusion program, high temperature engine and structural components for advanced high-performance aircraft, hard/wear resistant coatings (tribological), corrosion resistant coatings, coatings of complex geometries, near-net-shape fabrication, heat-pipe structures, precision CVD of ultra-thin, freestanding shapes.

Keywords: Chemical Vapor Deposition, Coatings (metal and ceramic)

434. Polymers and Adhesives FY 1992
$430,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: D. A. Hemphill, (505) 667-8335

The objective of this project is to identify potential weapons engineering and physics applications for plastic and composite materials, select or develop appropriate materials, develop low cost fabrication techniques compatible with Integrated Contractor production capabilities, and characterize promising materials on a timely basis to provide optimum material choices for new weapons designs. Material or process development projects include: highly filled polymers, composite structural and spring components, cushioning materials, and high-explosive compatible adhesives, potting materials. This work will be compatible with all current and future ES&H guidelines.

Keywords: Adhesives, Composites, Plastics, Polymers, Weapons Design, Weapons Engineering, Integrated Contractors

435. Tritiated Materials FY 1992
$175,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: J. R. Bartlit, (505) 667-5419

Advanced research and development efforts are focused on tritiated materials with the emphasis on Li(D,T) (salt) and other metal tritides. New methods for preparing, fabricating, and containing such compounds are under investigation. We are also using laser-Raman techniques for *in situ* measurements of hydrogen-deuterium-tritium gas mixtures, and kinetic studies of hydrogen-metal interactions.

Keywords: Tritium, Metal Tritides, Li(D,T), Tritiated Materials, Radioactive Materials

436. Salt Fabrication FY 1992
$750,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: J. R. Bartlit, (505) 667-5419

Development and evaluation of new fabrication and containment processes for Li(D,T) LiH, and LiD. This includes preparation of device parts for WTS tests. Research topics include development of hot pressing, machining techniques for salt compacts, new containment methods, and studies of radiation induced growth and outgassing.

Keywords: Tritium, Hydrides, Machining, Radioactive Materials, Near-Net-Shape Processing

437. Slip Casting of Ceramics

FY 1992
$90,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: D. S. Phillips, (505) 667-5128

We are slip casting many ceramics including alumina, zirconia-toughened alumina (ZTA), and magnesia. The technology uses colloidal chemistry and powder characterization techniques, along with materials engineering. Considerable progress was made in the development of ZTA ceramic alloys with a superior microstructure and improved thermal shock resistance. The scope of work has expanded to include frits and insulation materials, as well as dense crucibles.

Keywords: Ceramics, Microstructure, Strength, Transformation Toughened Ceramics, Thermal Shock

438. Plasma-Flame Spraying Technology

FY 1992
$300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: R. Castro, (505) 667-5191

Free-standing shapes and metallic and ceramic coatings are fabricated by plasma spraying. Materials examined recently include Be, ^{238}U, $MoSi_2$ and ZrO_2. Parts of this work involve investigation of ultrasonic-assisted densification to produce high density coatings. Applications include: radiochemical detectors; temperature-, oxidation-, and corrosion-resistant coatings; and electrically insulating coatings.

Keywords: Coatings, Metals, Ceramics, Plasma-Flame Spraying, High Temperature Service, Surface Characterization and Treatment

439. Rapid Solidification Technology

FY 1992
$500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: P. Stanek, (505) 667-6914

RSR technologies such as melt spinning, splat cooling, and rapid solidification plasma spraying, are being developed to evaluate a range of RSR alloys, intermetallics and composites for defense and energy applications. Activities include alloy development, microstructural analysis, mechanical and physical properties testing, process development and modeling.

Keywords: Rapid Solidification, Low Pressure Plasma, Alloy Development, Composites, Intermetallics

440. Bulk Ceramic Processing FY 1992
$150,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: J. D. Katz, (505) 665-1424

Cold pressing and cold isostatic pressing, followed by sintering, are used to produce ceramic and metal components for various physics experiments and for plutonium processing. Materials fabricated include alumina, magnesia and boron.

In addition, a collaborative effort was established with the University of New Mexico Center for Micro-Engineered Ceramics to investigate the effect of 2.45 GHz microwave energy on the diffusion of cations in ceramic oxides. This research consists of both a theoretical and experimental component. The results have shown that although microwave enhanced diffusion of chromium in alumina does not exist, microwave sintering has been found to be a very effective engineering tool for densifying even large alumina ceramics.

Finally, considerable effort was devoted to developing methods for sintering, rather than hot pressing, boron carbide to achieve high density. This work involves a collaboration with the A.W.E. in the United Kingdom.

Keywords: Ceramics, Sintering, Microwave Sintering, Cold Pressing

441. Synthesis of Ceramic Coatings FY 1992
$30,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: C. P. Scherer, (505) 665-3202

The objective of this effort is to synthesize Ceramic Films for liquid metal containment. One approach entails the use of organic and aqueous solvents to deposit erbia films, which are subsequently heat treated to densification. The second approach involves the *in situ* conversion of a metal surface to a nitride by precise heating in a nitrogen environment.

Keywords: Ceramic Coatings, Sol Gel, Nitration

Materials Structure or Composition

442. Actinide Surface Properties | FY 1992 $700,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: D. C. Christensen, (505) 667-2556

Characterization of actinide metal, alloy and compound surfaces using the techniques of X-ray photoelectron spectroscopy, Auger analysis, ellipsometry and Fourier-transform infrared spectroscopy. Surface reactions, chemisorption, attack by hydrogen, and the nature of associated catalytic processes are being studied.

Keywords: Actinides, Hydrides, Surface Characterization and Treatment, Hydrogen Effects, Radioactive Materials

443. Neutron Diffraction of Pu and Pu Alloys and Other Actinides | FY 1992 $237,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: A. C. Lawson, (505) 667-8844

Physical structure and properties of plutonium are being studied by pulsed neutron diffraction at the Manuel Lujan, Jr., Neutron Scattering Center (Los Alamos) and the Intense Pulsed Neutron Source (Argonne). A time-of-flight technique is used to measure diffraction at cryogenic and elevated temperatures.

Keywords: Alloys, Radioactive Materials, Transformation, Microstructure

444. Surface, Material and Analytical Studies | FY 1992 $300,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: W. C. Danen, (505) 667-4686

Studies are underway in four key areas: surface and interfacial structures and properties, explosives dynamics, laser-based isotopic analysis, and metastable energetic materials. Current investigations in surface and interfacial studies include: surface modification, HTSC composition and structure, and the use of MeV ion beams. In explosives chemistry, we are using real-time optical- and mass-spectral methods to probe the early-time dynamics of detonation. Analytical studies have centered on the use of resonance ionization mass spectrometry to eliminate isobaric interferences in the measurement of high-dynamic range isotope ratio measurements. We continue to study the synthesis and characterization of a new class of high energy density materials consisting of atomically-thin multilayered composite materials.

Keywords: Surface, Explosives, Interfaces, Composite Materials

Materials Properties, Behavior, Characterization or Testing

445. Mechanical Properties of Plutonium and Its Alloys — FY 1992 $450,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. Stevens, (505) 667-4414

The mechanical properties of plutonium and its alloys are related to the pre-test and post-test microstructures of the materials using optical and electron microscopy and X-ray, electron and neutron diffraction.

Keywords: Alloys, Radioactive Materials, Microstructures, Strength, Transformation

446. Phase Transformations in Pu and Pu Alloys — FY 1992 $450,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. Stevens, (505) 667-4414

Mechanisms and crystallography of thermally and mechanically induced allotropic transformations are studied with differential scanning calorimetry, optical and electron microscopy and electron and X-ray diffraction.

Keywords: Alloys, Radioactive Materials, Microstructure, Transformations

447. Plutonium Shock Deformation — FY 1992 $350,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. J. Reisfeld, (505) 667-1375

Plutonium and actinide alloys are subjected to shock deformation, recovered without further damage and examined to determine how the shock affected their microstructures and mechanical properties.

Keywords: Radioactive Materials, Plutonium Alloys, Microstructure, Strength

448. Non-Destructive Evaluation FY 1992 $550,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: W. M. Mathieson, (505) 667-1973

Development of Nondestructive Evaluation Technology that produces quantitative estimates of material properties. Use of tomographic techniques to enhance radiographic inspection. Flash, cine radiography, high speed video recorded optical and X-ray diagnostics of dynamic and ultra-fast events. Real-time radiography. Image enhancement of output results from all techniques. Development of ultrasonic inspection techniques.

Keywords: Nondestructive Evaluation, Radiography, Ultrasonic Microscopy, Tomography, Cine Radiography, Bonding Processes, Real-Time Radiography, Image Enhancement

449. Powder Characterization FY 1992 $50,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: G. J. Vogt, (505) 667-5813

Synthesis and processing of ceramic or metal powders depends critically on the physical characterization of the starting powders being used. Typical starting powders include commercial powders of thoria, magnesia, alumina, tungsten, copper, tungsten carbide, and boron carbide. In the past year, considerable effort has been expended on characterizing palladium alloy powders. Physical properties of interest include particle size and distribution, surface area, bulk and packed densities, morphology, pore size and distribution, and zeta potential. The crystalline-phase composition of the starting powders and processed powders can be determined by X-ray diffraction.

Keywords: Ceramic Powder, Metal Powder, Particle Size, Superconducting Powder, X-ray Diffraction, Surface Area

450. Shock Deformation in Actinide Materials FY 1992 $200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: M. Stevens, (505) 667-4414

Measurement of shock-wave profiles in uranium, plutonium, and plutonium alloys. Use of soft-shock recovery test to examine the microstructural changes occurring during shock deformation. Measurement of spall strength in actinide materials and examination of fracture surfaces.

Keywords: Actinides, Shock Deformation, Microstructure, Spall Strength

451. Dynamic Mechanical Properties of Weapons Materials FY 1992 $225,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: G. Gray, (505) 667-5452

Measurements of dynamic stress-strain and fracture behavior of materials used for nuclear weapons. Development of plastic constitutive relations.

Keywords: Dynamic, Strength, Fracture, Microstructure

452. Dynamic Testing of Materials for Hyper-Velocity Projectiles FY 1992 $100,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: G. T. Gray, III, (505) 667-4665

Spall testing of high density materials. Microstructural characterization and fractography on spall fracture surfaces. Dynamic and quasi-static compression tests.

Keywords: Shock Deformation, Spall Strength, Microstructure, Strength

Device or Component Fabrication, Behavior or Testing

453. Radiochemistry Detector Coatings

FY 1992
$200,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: G. Reeves, (505) 667-4290

Physical vapor deposition of metallic and nonmetallic coatings is employed for preparation of radiochemical detectors.

Keywords: Coatings and Films, Physical Vapor Deposition, Radiochemical Detectors

454. Target Fabrication

FY 1992
$1,500,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contact No. W-7405-ENG-36) Contact: L. Foreman, (505) 667-1846
LLNL Contact: W. Hatcher, (510) 422-1100
General Atomics Contact: Ken Schultz, (619) 455-4304

ICF/AGEX targets are fabricated using PVD, CVD, precision micromachining, and polymer chemistry techniques. After the parts are fabricated, the components are assembled using a variety of techniques. These targets are used to provide laser materials interactions data for the inertial confinement fusion community.

Keywords: Inertial Fusion, Target Fabrication

455. Filament Winder

FY 1992
$30,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: B. Benicewicz, (505) 665-0101

The Entec filament winder in MST-7 Plastics is a 4-axis computer-programmed machine with a winding envelope extending up to 4 feet in diameter and 10 feet in length. It is being utilized to wind circumferential or helical cylinders, cones, spheres, and closed-end vessels from a variety of fibers including glass, kevlar, carbon, tungsten, and aluminum oxide. The applications cover a host of programs from within the Laboratory as well as from outside agencies.

Keywords: Filament Winding, Composites

456. High Energy Density Welding in Hazardous Environments FY 1992
$800,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: G. Lewis, (505) 667-9663

High power Nd/YAG lasers combined with fiber optic beam delivery systems have been evaluated for welding applications in hazardous environments. Applications include the manufacture of nuclear weapons components and nuclear power reactor repair. High quality structural welds have been achieved without exposing the operators or the welding power supplies to the hazardous environment.

Keywords: Laser Welding, Fiber Optic Beam Delivery, Hazardous Environments, Nuclear Applications

457. Uranium Scrap Conversion and Recovery FY 1992
$400,000

DOE Contact: G. J. D'Alessio, (301) 903-6688
LANL (Contract No. W-7405-ENG-36) Contact: Dan Knobeloch, (505) 667-4417

Maintain and develop technologies for conversion and recovery of uranium scrap. Maintain and upgrade facilities for processing enriched uranium and managing uranium inventories.

Keywords: Uranium, Uranium Scrap, Enriched Uranium, Recovery, Processing, Inventories

OFFICE OF FOSSIL ENERGY

	FY 1992
Office of Fossil Energy - Grand Total	$6,614,000
Office of Advanced Research	$6,614,000
Fossil Energy AR&TD Materials Program	$6,614,000
Materials Preparation, Synthesis, Deposition, Growth or Forming	$2,814,000
Fundamental Study of Diffusion Coating Processes	0
Procurement of Advanced Austenitic and Aluminide Alloys	29,000
Iron Aluminide Equipment	34,000
Development of Iron Aluminides	391,000
Ultrahigh Temperature Intermetallic Alloys	196,000
Microalloyed Iron Aluminides	64,000
Low-Aluminum Content Iron-Aluminum Alloys	135,000
Investigation of Austenitic Alloys for Advanced Heat Recovery and Hot-Gas Cleanup Systems	220,000
Evaluation of the Fabricability of Advanced Austenitic Alloys	0
Welding Equipment	9,000
The Influence of Processing on Microstructure and Properties of Aluminides	171,000
Solidification Behavior of Iron Aluminides	40,000
Investigation of Electrospark Deposited Coatings for Protection of Materials in Sulfidizing Atmospheres	127,000
Vapor-Liquid-Solid SiC Whisker Process Development	59,000
Engineering-Scale Development of the Vapor-Liquid-Solid (VLS) Process for the Production of Silicon Carbide Whiskers	328,000
Ceramic Composite Processing Equipment	49,000
Fabrication of Fiber-Reinforced Composites by Chemical Vapor Infiltration and Deposition (CVID)	196,000
Fabrication of Ceramic Composites by Chemical Vapor Infiltration and Deposition	17,000
Interfaces and Mechanical Properties of Continuous Fiber-Reinforced Ceramic Composites	147,000
Low-Temperature Fabrication of Transparent Silicon Nitride	196,000
Microwave Sintering of Ceramics for Fuel Cells	210,000

OFFICE OF FOSSIL ENERGY

	FY 1992
Office of Advanced Research (continued)	
Fossil Energy AR&TD Materials Program (continued)	
Materials Preparation, Synthesis, Deposition, Growth or Forming (continued)	
Development of Advanced Fiber Reinforced Ceramics	147,000
Modeling of Fibrous Preforms for CVD Infiltration	49,000
Materials Properties, Behavior, Characterization or Testing	$1,777,000
Investigation of the Weldability of Polycrystalline Iron Aluminides	73,000
Aqueous Corrosion of Iron Aluminides	49,000
Fireside Corrosion Tests of Candidate Advanced Austenitic Alloys, Coatings, and Claddings	49,000
Joining Techniques for Advanced Austenitic Alloys	101,000
Corrosion and Mechanical Properties of Alloys in FBC and Mixed-Gas Environments	312,000
Environmental Effects on Iron Aluminides	220,000
Investigation of Moisture-Induced Embrittlement of Iron Aluminides	59,000
Corrosion Protection of Ultrahigh Temperature Intermetallic Alloys	215,000
Development of Nondestructive Evaluation Methods and Effects of Flaws on the Fracture Behavior of Structural Ceramics	308,000
Joining of Fiber-Reinforced Silicon Carbide Composites	171,000
Ceramic Catalyst Materials: Hydrous Metal Oxide Ion Exchange Supports for Direct Coal Liquefaction	220,000

OFFICE OF FOSSIL ENERGY

	FY 1992
Office of Advanced Research (continued)	
Fossil Energy AR&TD Materials Program (continued)	
Device or Component Fabrication, Behavior or Testing	$1,548,000
Materials and Components in Fossil Energy Applications (Newsletter)	20,000
Mechanisms of Galling and Abrasive Wear	0
Fabrication of Full-Scale Fiber-Reinforced Hot-Gas Filters by Chemical Vapor Deposition	203,000
Development of Ceramic Membranes for Gas Separation	391,000
Investigation of the Mechanical Properties and Performance of Ceramic Composite Components	108,000
Advanced Materials and Electrochemical Processes in High-Temperature Solid Electrolytes	826,000
Instrumentation and Facilities	$ 475,000
Management of the Fossil Energy AR&TD Materials Program	391,000
Coal Conversion and Utilization Plant Support Services	49,000
General Technology Transfer Activities	35,000

OFFICE OF FOSSIL ENERGY

The Office of Fossil Energy responsibilities include management of the Department's fossil fuels (coal, oil and natural gas) research and development program. This research is generally directed by the Office of Coal Technologies (OCT), the Office of Oil, Gas, Shale and Special Technologies (OGSST) and the Office of Advanced Research in support of the National Energy Strategy (NES) Goals for Increasing Energy Efficiency, Securing Future Energy Supplies, Respecting the Environment, and Fortifying our Foundations. Three specific Fossil goals are currently being pursued:

- The first is to secure liquids supply and substitution. This goal targets the enhanced production of domestic petroleum and natural gas, the development of advanced, cost-competitive alternative fuels technology, and the development of coal-based, end-use technology to substitute for oil in applications traditionally fueled by liquid and gaseous fuel forms.

- The second is to develop power generation options with environmentally superior, high-efficiency technologies for the utility, industrial, and commercial sectors. This goal targets the development of super-clean, high-efficiency power generation technologies.

- The third is to pursue a global technology strategy to support the increased competitiveness of the U.S. in fossil fuel technologies, to maintain world leadership in our fossil fuel technology base, and provide expanded markets for U.S. fuels and technology. This crosscutting goal is supported by the activities in the above two technology goals.

Office of Advanced Research

Fossil Energy AR&TD Materials Program

Fossil Energy (FE) materials-related research is conducted under an Advanced Research and Technology Development (AR&TD) Materials subactivity and is an integral part of the R&D conducted by OCT and OGSST technology programs. The AR&TD Materials program includes cross-cutting research to obtain a fundamental understanding of materials and how they perform in fossil-based process environments and the development of new classes of generic materials that will allow the development of new fossil systems or major improvements in existing systems. The present program is focused on ceramics (composite structural ceramics, catalyst supports, solid state electrolytes, membranes, and ceramic filters), new alloys (aluminides, advanced austenitic steels, and coatings and claddings), corrosion research, and technology development and transfer.

The AR&TD research is carried through development and technology transfer to industry. Special emphasis is being given to technology transfer to ensure that the materials will be available for subsequent fossil commercial applications. This also enhances U.S. technological competitiveness not only in the fossil area but in the materials industry in general and other technology application areas as well. The research is conducted in industry, universities, not-for-profit agencies, and national laboratories. This widespread participation also helps maintain the U.S. materials technology capabilities.

The acronym PF indicates that the work in the designated year was supported by prior-year funds.

Materials Preparation, Synthesis, Deposition, Growth or Forming

458. Fundamental Study of Diffusion Coating Processes — FY 1992 $0 (PF)

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Ohio State University Contact: R. A. Rapp, (614) 292-6178

The purpose of this work is to conduct a study of diffusion coating of iron-base alloys which will lead to a fundamental understanding of these processes. The work provides the ability to specify pack compositions and conditions that will assure the deposition of corrosion-resistant coatings. The work also provides specifications such as coating thickness, diffusion zone thickness, and elemental concentrations for corrosion-resistant coatings.

Keywords: Alloys, Aluminizing, Chromizing, Corrosion, Coatings

459. Procurement of Advanced Austenitic and Aluminide Alloys — FY 1992 $29,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824

This task provides funds for the procurement of alloys necessary for alloy development and testing activities under the AR&TD Materials Program.

Keywords: Alloys, Aluminides, Austenitic

460. Iron Aluminide Equipment FY 1992
$34,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824

This task provides funds for the procurement of major equipment items necessary for iron aluminide development activities under the AR&TD Materials Program.

Keywords: Alloys, Aluminides

461. Development of Iron Aluminides FY 1992
$391,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: V. K Sikka, (615) 574-5112

The objective of this project is to develop low-cost, low-density intermetallic alloys based on Fe_3Al with an optimum combination of strength, ductility, and corrosion resistance for use as components in advanced fossil energy systems.

Keywords: Alloys, Aluminides, Intermetallic Compounds

462. Ultrahigh Temperature Intermetallic Alloys FY 1992
$196,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: C. T. Liu, (615) 574-4459

The objective of this project is to develop high-strength, corrosion-resistant intermetallic alloys for use as hot components in advanced fossil energy conversion systems. The successful development of these alloys is expected (1) to improve the thermal efficiency of fossil energy conversion systems, and (2) to increase the service life of hot components exposed to corrosive environments.

Keywords: Alloys, Chromium-Niobium, Corrosion, Intermetallic Compounds

463. Microalloyed Iron Aluminides FY 1992
$64,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: C. G. McKamey, (615) 574-6917

The objective of this project is to use microalloying techniques to extend the development of those Fe_3Al-based alloys identified as possessing improved room-temperature tensile properties. Emphasis is on low-cost, low-density, precipitation-strengthened Fe_3Al-based alloys with improved high-temperature creep resistance and an optimum combination of good room- and high-temperature tensile properties, weldability, and corrosion resistance.

Keywords: Alloys, Aluminides, Microalloy

464. Low-Aluminum Content Iron-Aluminum Alloys | FY 1992 $135,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: V. K. Sikka, (615) 574-5112

The objective of this project is to develop a conventionally fabricable low-cost, low-density iron-aluminum alloy with a good combination of strength, ductility, weldability, and corrosion resistance for use as components in advanced fossil energy conversion systems.

Keywords: Alloys, Iron-Aluminum

465. Investigation of Austenitic Alloys for Advanced Heat Recovery and Hot-Gas Cleanup Systems | FY 1992 $220,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: R. W. Swindeman, (615) 574-5108

The purpose of this project is to evaluate austenitic alloys for improved performance in high-temperature components in advanced heat recovery and hot-gas cleanup systems. Detailed alloy performance criteria for advanced steam cycle alloys have been established. Factors considered included strength, ductility, corrosion resistance, high-temperature stability, and fabricability. The lean stainless steels were selected for additional study, and industrial subcontractors supplied pilot heats of the chosen alloy, a modification of type 316 stainless steel. Work to optimize the composition and heat treatment and studies of fabrication methods, joining methods, and surface treatments were undertaken. With the near completion of work on the lean stainless alloys, attention has turned to the 20-30% Cr alloys. Evaluation of the higher chromium alloys has been extended to temperatures of interest to Pressurized Fluidized Bed Combustion (PFBC) hot-gas cleanup systems.

Keywords: Steam Cycle, Materials, Mechanical Properties, Austenitics, Hot-Gas

466. Evaluation of the Fabricability of Advanced Austenitic Alloys FY 1992
$0 (PF)

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Babcock and Wilcox Contact: Walter Mohn, (216) 829-7816

The purpose of this work is to evaluate the fabricability, weldability, and surface treatments of advanced austenitic tubing for superheater applications. The problem of the fabrication of tubing from alloys containing controlled amounts of minor element additions and surface treatments of the tubing for optimum strength and corrosion resistance is examined.

Keywords: Austenitics, Alloys, Tubing, Fabricability

467. Welding Equipment FY 1992
$9,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824

This task provides funds for the procurement of major equipment items necessary for welding activities that support new alloy development under the AR&TD Materials Program.

Keywords: Welding, Alloys

468. Influence of Processing on Microstructure and Properties of Aluminides FY 1992
$171,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Idaho National Engineering Laboratory Contact: R. N. Wright, (208) 526-6127

The purpose of this project is to determine the influence of processing on the properties of alloys based on Fe_3Al. Thermomechanical processing is pursued to improve their room-temperature ductility. The response of the microstructure to annealing will be characterized in terms of the establishment of equilibrium phases and degrees of long-range order. The mechanical properties are determined at room and elevated temperatures and related to the microstructure.

Keywords: Aluminides, Processing, Microstructure

469. Solidification Behavior of Iron Aluminides FY 1992
$40,000

DOE Contacts:J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Oak Ridge Associated Universities Contact: S. Viswanathan, (615) 574-5112

The purpose of this project is to evaluate and improve the casting characteristics of iron aluminides.

Keywords: Aluminides, Casting, Solidification

470. Investigation of Electrospark Deposited Coatings for Protection of Materials in Sulfidizing Atmospheres FY 1992
$127,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Westinghouse Hanford Company Contact: R. N. Johnson, (509) 376-3582

The purpose of this task is to examine the use of the electrospark deposition coating process for the application of corrosion-, erosion-, and wear-resistant coatings to candidate superheater alloys. Materials to be deposited may include MCrAl, MCrAlY, highly wear-resistant carbides, and other hardsurfacing materials.

Keywords: Coatings, Materials, Deposition

471. Vapor-Liquid-Solid SiC Whisker Process Development FY 1992
$59,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Los Alamos National Laboratory Contact: J. D. Katz, (505) 665-1424

The purpose of this project is to provide assistance in transferring the laboratory-scale Vapor-Liquid-Solid (VLS) SiC whisker growth process to an industrial organization for engineering-scale development.

Keywords: Ceramics, Whiskers, Composites

472. Engineering-Scale Development of the Vapor-Liquid-Solid (VLS) Process for the Production of Silicon Carbide Fibrils — FY 1992 $328,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
The Carborundum Company Contact: R. S. Storm, (716) 278-2000

The purpose of this work is to transfer to industry a specific technology developed by DOE under the AR&TD Materials Program for the production of silicon carbide fibrils for the reinforcement of ceramic matrices. The Vapor-Liquid-Solid (VLS) process has been developed at Los Alamos National Laboratory (LANL) for the growth of silicon carbide fibrils of up to 75 mm in length which can be reduced in length by subsequent processing. The purpose of the work is to develop the VLS process into an engineering-scale process that will enable the U.S. industrial sector to commercialize the process for the production of fibrils for the reinforcement of structural ceramic components.

Keywords: Whiskers, Fibers, Ceramic

473. Ceramic Composite Processing Equipment — FY 1992 $49,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824

This task provides funds for the procurement of major equipment items necessary for ceramic composite development and characterization activities under the AR&TD Materials Program.

Keywords: Ceramics, Composites

474. Fabrication of Fiber-Reinforced Composites by Chemical Vapor Infiltration and Deposition (CVID) — FY 1992 $196,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: D. P. Stinton, (615) 574-4556

The purpose of this task is to develop a process for the fabrication of fiber-reinforced ceramic composites having high fracture toughness and high strength. This process utilizes a steep temperature gradient and a pressure gradient to infiltrate low-density fibrous structures with gases, which deposit as solid phases to form the matrix of the composite. Modifications to the process which are being explored include controlling the porosity and permeability of the fibrous preforms and variation of the deposition conditions. Alternate matrices, alternate fiber types, and pretreating or coating of fibers will be investigated to optimize the mechanical properties of the composites.

Keywords: Composites, Fiber-Reinforced, Ceramics

475. Fabrication of Ceramic Composites by Chemical Vapor Infiltration and Deposition

FY 1992
$17,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
University of Tennessee Contact: D. M. Walukas, (615) 574-4551

The objective of this research is development of the process of chemical infiltration and deposition (CVID) for fabrication of ceramic composites. This work is part of the University of Tennessee-Oak Ridge National Laboratory initiative for composites and structures to develop an interactive program in composites. A model of densification rates shall be evaluated. As part of this evaluation, experiments shall be performed to determine the key parameters in the model.

Keywords: Ceramics, Composites

476. Interfaces and Mechanical Properties of Continuous Fiber-Reinforced Ceramic Composites

FY 1992
$147,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: R. A. Lowden, (615) 574-7714

The purpose of this task is to optimize the strength and toughness of fiber-reinforced ceramic composites by tailoring the strength of the bonds between the fiber and the matrix. Methods must first be developed to characterize the fiber-matrix bond strengths in fiber-reinforced ceramic composite systems. Coating or pretreatment processes can then be utilized to tailor the fiber-matrix bonding within various composite systems and to optimize the strength and toughness of the composite.

Keywords: Composites, Ceramics, Fiber-Reinforced, Interfaces

477. Low-Temperature Fabrication of Transparent Silicon Nitride — FY 1992 $196,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
National Institute of Standards and Technology Contact: S. G. Malghan, (301) 975-6101

The objective of this research is the production of dense, hard, transparent ceramics from nanosize particles without the use of sintering aids. The work will concentrate on the fabrication of samples of transparent silicon nitride using the cryogenic compaction technique. A special apparatus for compacting nanosize particles under controlled atmospheres, temperatures, and rates of compaction will be used for sample fabrication and for rheologic studies of compacting powder. TEM, SEM, X-ray diffraction, and laser light scattering will be used to characterize the microstructure. Hardness at various temperatures will be measured to assess the creep resistance of the material. Fracture toughness and bending strength will also be measured. The work will provide a foundation for possible industrial fabrication of dense, transparent ceramics without the necessity for sintering processes.

Keywords: Ceramics, Mechanical Properties

478. Microwave Sintering of Ceramics for Fuel Cells — FY 1992 $210,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: M. A. Janney, (615) 574-4281

The purpose of this activity is to explore the feasibility of using microwave heating as a means of fabricating electrode, electrolyte, and interconnect materials having improved electrical properties for monolithic solid oxide fuel cell designs being advanced by DOE. The ultimate goal is to develop the technology (materials and process) for fabricating a complete monolithic fuel cell module in one operation.

Keywords: Ceramics, Microwave Sintering, Fuel Cells

479. Development of Advanced Fiber Reinforced Ceramics — FY 1992 $147,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
Oak Ridge National Laboratory Contact: N. C. Cole, (615) 574-4824
Georgia Institute of Technology Contact: T. L. Starr, (404) 894-3678

The purpose of this research effort is to conduct a theoretical and experimental program to identify new compositions and processing methods to improve the physical and mechanical properties of selected fiber-reinforced ceramics. The ceramic matrix material is amorphous fused silica or modified silica glass, and the focus is the development of fiber-reinforced silica.

Parameters studied include: (1) differences in elastic modulus between matrix and fiber, (2) differences in thermal expansion, (3) nature of interfacial bond, (4) densification of matrix, (5) nature of fiber fracture/pull-out, (6) fiber diameter and fiber length-to-diameter ratio, (7) fiber loading, and (8) fiber dispersion and orientation. A model will be developed based on the information generated in the experimental phase of the program.

Keywords: Ceramics, Composites, Fiber-Reinforced

480. Modeling of Fibrous Preforms for CVD Infiltration — FY 1992 $49,000

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The purpose of this project is to conduct a theoretical and experimental program to develop an analytical model for the fabrication and infiltration of fibrous preforms. The analytical model will: (1) predict preform structure (density, porosity, fiber orientation, etc.) based on fabrication technique and fundamental fiber parameters (diameter, aspect ratio, etc.), and (2) predict permeation and heat conduction through the preform structure and, thus, predict the CVD infiltration performance.

Keywords: Ceramics, Composites, Modeling

Materials Properties, Behavior, Characterization or Testing

481. Investigation of the Weldability of Polycrystalline Iron Aluminides — FY 1992 $73,000

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The purpose of this project is the investigation of the weldability of polycrystalline aluminides. The major thrust of the project is to determine the role of microstructure in the intergranular cracking of aluminides, with special emphasis on weld cracking susceptibility. The weldability of polycrystalline Fe_3Al-X alloys is being evaluated, and the weldability is correlated with composition, phase equilibria, grain size and morphology, domain size, and degree of long-range order.

Keywords: Joining, Welding

482. Aqueous Corrosion of Iron Aluminides FY 1992
$49,000

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The objective of this project is to investigate the aqueous corrosion of iron aluminides based on Fe_3Al. The effort provides basic corrosion information over a wide range of pH values for each of several experimental iron aluminide compositions and allows comparisons to be made among iron aluminide compositions, as well as with other corrosion-resistant materials of interest to fossil energy systems.

Keywords: Alloys, Aluminides, Corrosion

483. Fireside Corrosion Tests of Candidate Advanced Austenitic Alloys, Coatings, and Claddings FY 1992
$49,000

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The purpose of this project is to provide comprehensive corrosion data for selected advanced austenitic tube alloys in simulated coal ash environments. ORNL-modified alloys and standard comparison alloys have been examined. The variables affecting coal ash corrosion and the mechanisms governing oxide breakdown and corrosion penetration are being evaluated. Corrosion rates of the test alloys are determined as functions of temperature, ash composition, gas composition, and time.

Keywords: Austenitics, Alloys, Corrosion

484. Joining Techniques for Advanced Austenitic Alloys FY 1992
$101,000

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Weldability is an important consideration in the selection of a suitable alloy for the fabrication of boiler components such as superheaters and reheaters. It is often a challenge to select joining materials and establish procedures that will allow advanced materials to function at their full potential. The purpose of this research is to examine important aspects of newly developed austenitic tubing alloys intended for service in the temperature range 550° to 700°C.

Keywords: Alloys, Austenitics, Joining, Welding

485. Corrosion and Mechanical Properties of Alloys in FBC and Mixed-Gas Environments — FY 1992 $312,000

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The purposes of this task are to (1) evaluate the corrosion mechanisms for chromia- and alumina-forming alloys in mixed-gas environments, (2) develop an understanding of the role of several microalloy constituents in the oxidation/sulfidation process, (3) evaluate transport kinetics in oxide scales as functions of temperature and time, (4) characterize surface scales that are resistant to sulfidation attack, and (5) evaluate the role of deposits in corrosion processes.

Keywords: Corrosion, Gasification, Creep Rupture, Fluidized-Bed Combustion

486. Environmental Effects on Iron Aluminides — FY 1992 $220,000

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The purpose of this task is to evaluate the corrosion properties of Fe_3Al-based alloys as they relate to fossil energy applications. A primary objective is to measure the resistance of the alloys to mixed-oxidant [oxygen-sulfur] environments that arise in the combustion or gasification of coal. This includes a determination of the effects of sulfur on oxidation kinetics and oxide microstructures, the effects of rare earth additions on sulfidation and oxidation resistance, and the mechanical behavior of reaction product scales in mixed-gas environments.

Keywords: Corrosion, Aluminides, Mixed-Gas, Scales

487. Investigation of Moisture-Induced Embrittlement of Iron Aluminides FY 1992
$59,000

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The purpose of this work is to study hydrogen embrittlement of iron aluminide alloys. Moisture in air can significantly reduce the room-temperature tensile ductility of Fe_3Al-based alloys by combining with the aluminum in the alloys to form atomic hydrogen. The atomic hydrogen diffuses rapidly into the material causing embrittlement. Experiments are being conducted on selected Fe_3Al alloys that will lead to an understanding of the phenomenon. The work focuses on the effects of moisture on relevant mechanical properties such as fatigue and tensile strengths, and correlates important microstructural variables such as degree of order, grain size, and phases present with the alloy's susceptibility to embrittlement.

Keywords: Aluminides, Embrittlement, Moisture

488. Corrosion Protection of Ultrahigh Temperature Intermetallic Alloys FY 1992
$215,000

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The purpose of this activity is to support the development of high-strength, corrosion-resistant intermetallic alloys by conducting critical experiments and analyses to evaluate the best alloy design and surface treatments to offer protection for environmental degradation at high temperatures.

Keywords: Corrosion, Chromium-Niobium, Mixed-Gas, Scales

489. Development of Nondestructive Evaluation Methods and Effects of Flaws on the Fracture Behavior of Structural Ceramics FY 1992
$308,000

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The purpose of this project is to study and develop acoustic and radiographic techniques and possible novel techniques such as nuclear magnetic resonance, to characterize structural ceramics with regard to presence of porosity, cracking, inclusions, amount of free silicon, and mechanical properties, and to establish the type and character of flaws that can be found by nondestructive evaluation (NDE) techniques. Both fired and unfired specimens are being studied to establish correlations between NDE results and failure of specimens.

Keywords: Nondestructive Evaluation, Ceramics, Flaws, Fracture

490. Joining of Fiber-Reinforced Silicon Carbide Composites — FY 1992 $171,000

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The purpose of this project is to explore and develop joining techniques for silicon carbide fiber-reinforced silicon carbide ceramics produced by chemical vapor infiltration and deposition (CVID). The research goals include identifying appropriate joining methods, establishing experimental procedures for fabricating joints, and characterizing the structure and properties of joined materials. An understanding of the factors that control joint performance is sought through studies of the relationships among processing variables, joint microstructures, and mechanical properties.

Keywords: Joining, Ceramics, Composites

491. Ceramic Catalyst Materials: Hydrous Metal Oxide Ion Supports for Direct Coal Liquefaction Exchange — FY 1992 $220,000

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The purpose of this research is to investigate the role of ceramic material properties in the catalytic activity of a novel class of catalytic supports, known as hydrous titanium oxides (HTO). Catalysts prepared on these materials show particular promise as economically and environmentally attractive alternatives to present commercial catalysts for the direct liquefaction of coal. In these studies, improved understanding and control of the synthesis process is being pursued in order to tailor the composition, molecular structure, microporosity, and physical/mechanical properties of the HTO thin films. The effects of altered structure, composition, and other material properties of the thin film ceramic support material on catalytic activity are being assessed.

Keywords: Ceramics, Catalysts

Device or Component Fabrication, Behavior or Testing

492. *Materials and Components in Fossil Energy Applications* (Newsletter) FY 1992
$20,000

DOE Contacts: J. P. Carr, (301) 903-6519 and E. E. Hoffman, (615) 576-0735
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The purpose of this task is to publish a periodic (bimonthly) newsletter to address current developments in materials and components in fossil energy applications.

Keywords: Materials, Components

493. Mechanisms of Galling and Abrasive Wear FY 1992
$0 (PF)

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The purpose of this project is to investigate wear mechanisms that occur at the piston ring/cylinder liner contact in the coal-fueled diesel engine. The main emphasis of the work is the establishment of a test method that more closely simulates the ring-on-cylinder wall configuration than does the pin-on-disk method. Tests have been conducted with the improved configuration using selected materials, lubricants, and particles that allows the comparison of the two methods.

Keywords: Erosion, Wear, Galling

494. Fabrication of Full-Scale Fiber-Reinforced Hot-Gas Filters by Chemical Vapor Deposition FY 1992
$203,000

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The purpose of this project is to scale-up the chemical vapor infiltration and deposition (CVID) process developed at Oak Ridge National Laboratory for fabricating ceramic fiber-ceramic matrix composites. The goal is to use the scaled-up CVID process to produce composite filters that have the requisite strength and toughness, but which also have sufficient porosity to be permeable to gas streams and the appropriate size and distribution of porosity to be an effective filter. A practical process for fabricating porous ceramic fiber-ceramic matrix candle filters (full-size) with increased surface area will be developed.

Keywords: Ceramics, Composites, Filters

495. Development of Ceramic Membranes for Gas Separation — FY 1992 $391,000

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The purpose of this activity is to fabricate inorganic membranes for the separation of gases at high temperatures and/or in hostile environments, typically encountered in fossil energy conversion processes such as coal gasification. This work is performed in conjunction with a separate research activity that is concerned with the development and testing of the ceramic membranes.

Keywords: Ceramics, Membranes, Filters, Separation

496. Investigation of the Mechanical Properties and Performance of Ceramic Composite Components — FY 1992 $108,000

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Virginia Polytechnic Institute Contact: K. L. Reifsnider, (703) 961-5316

The purpose of this project is to develop a test system and test methods to obtain information on the properties and performance of ceramic composite materials. The work involves a comprehensive mechanical characterization of composite engineering components such as tubes, plates, shells, and beams subjected to static and cyclic multiaxial loading at elevated temperatures for extended time periods.

Keywords: Ceramics, Composites, Mechanical Properties, Testing

497. Advanced Materials and Electrochemical Processes in High-Temperature Solid Electrolytes

FY 1992
$826,000

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The objective of this research is (1) to identify, develop, and demonstrate advanced materials for use as alternative electrodes and current interconnections in solid oxide fuel cells, and (2) to develop an understanding of the synergistic effects of materials properties, structures, and compositions on electrochemical processes related to high-temperature solid electrolyte use in electrochemical cells.

Keywords: Fuel Cells, Electrochemical, Electrolytes

Instrumentation and Facilities

498. Management of the Fossil Energy AR&TD Materials Program

FY 1992
$391,000

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The overall objective of the Fossil Energy Advanced Research and Technology Development (AR&TD) Materials Program is to conduct a fundamental, long-range research and development program that addresses, in a generic way, the materials needs of fossil energy systems and ensures the development of advanced materials and processing techniques. The purpose of this task is to manage the Fossil Energy AR&TD Materials Program in accordance with procedures described in the Program Management Plan approved by DOE. This task is responsible for preparing the technical program implementation plan for DOE approval; submitting budget proposals for the program; recommending work to be accomplished by subcontractors and by Oak Ridge National Laboratory (ORNL); placing and managing subcontracts for fossil energy materials development at industrial research centers, universities, and other government laboratories; and for reporting the progress of the program.

Keywords: Management, Materials Program

499. Coal Conversion and Utilization Plant Support Services — FY 1992 $49,000

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The objective of this task is to provide support to the staffs of the DOE Energy Technology Centers and of operating coal conversion and utilization facilities in the areas of materials testing, evaluation, selection, and failure analysis.

Keywords: Testing, Failure Analysis

500. General Technology Transfer Activities — FY 1992 $35,000

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The task provides funds for the initiation of technology transfer activities to identify and develop relationships with industrial partners for the transfer of AR&TD Materials Program technologies to industry.

Keywords: Technology Transfer

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