Research Activity: Synthesis and Processing Science

Division: Materials Sciences and Engineering
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Portfolio Description:

Synthesis and Processing Science addresses the fundamental understanding necessary to extend from design and synthesis to the preparation of materials with desired structure, properties, or behavior. This includes the assembly of atoms or molecules to form materials, the manipulation and control of the structure at all levels from the atomic to the macroscopic scale, and the development of processes to produce materials for specific applications. The goal of basic research in this area ranges from the creation of new materials and the improvement of the properties of known materials, to the understanding of such phenomena as adhesion, diffusion, crystal growth, sintering, and phase transition, and ultimately to the development of novel diagnostic, modeling and processing approaches. This activity also includes development of *in situ* measurement techniques and capabilities to quantitatively determine variations in the energetics and kinetics of growth and formation processes on atomic or nanometer length scales.

Unique Aspects:

- The Materials Preparation Center (MPC) at the Ames Laboratory is operated for the purposes of understanding and further developing innovative and superior processes and for providing small quantities of unique, research-grade materials that are not otherwise available to academic, governmental, and industrial research communities.
- Through the Center of Excellence for the Synthesis and Processing of Advanced Materials (CSP), coordinated, collaborative research partnerships related to synthesis and processing of advanced materials are promoted between DOE national laboratories, universities, and the private sector.
- Non-equilibrium processing methods are studied using advanced modeling techniques and experimental methods, including innovative real-time, *in-situ* techniques enabled by synchrotron radiation.
- Advanced growth techniques and *in situ* diagnostics have been developed for the synthesis of improved thin-film structures of advanced materials.

Relationship to Others:

This program is intimately related to the other research activities in the Division of Materials Sciences and Engineering as the synthesis and processing of materials is critically important and must be tailored to achieve optimal structure, properties and behavior.

Through materials supplied by the Ames MPC, linkage is provided to the Office of Biological and Environmental Research, the academic community, the industrial community and international research institutions. In FY 2002 the MPC provided materials to 14 users at DOE laboratories, 3 at other federal laboratories, 46 universities, 72 industrial laboratories and 37 international universities and institutions. Total orders totaled $515 k in FY 2002. During FY 2002 the MPC continued its effort to prepare 24 kilograms of LaNi$_{5-x}$Sn$_x$ for the Jet Propulsion laboratory. This hydrogen sorption material is to be used in fabricating a Joule-Thompson cryocooler for background radiation measurements aboard the joint ESA-NASA PLANCK Mission vehicle scheduled for launch in 2007. A total of over 500 lbs of Sn-Ti alloy was prepared during FY 2002 for the fabrication of superconducting wire to be used for the South Korean K-Star Tokamack and the National High Magnetic Field Laboratory at Florida State University.

Additional linkages within the Department of Energy are provided through the Energy Materials Coordinating Committee. This research activity is also linked with Defense Programs via Nanoscience Network. Interagency coordination is provided by participation in the MatTec Communications Group on Metals, the MatTec Communications Group on Structural Ceramics, and the National Nanotechnology Initiative.

Significant Accomplishments:

This program has changed the way people understand and think about the preparation of materials. Experimental, theoretical and computational tools are developed and applied to advance the scientific understanding of complicated thermodynamic and kinetic phenomena underlying processes ranging from self-assembly to the far from equilibrium reactions that take place in welding. In the welding area, a coupled thermodynamic and kinetic model was developed to describe stability of the principal phases in stainless steels. This knowledge has led to the modification of the standard diagram used to choose welding electrode compositions for stainless steels. Additional modeling work utilizing massively parallel computers has permitted the linkage of macro- and microscopic scale phenomena during the melting and solidification of a weld. This permits simulation and visualization of weld microstructure as a function of processing conditions, e.g. during the melting, addition of new compounds, and resolidification that occurs during welding. Experimentally, tracking of real-time phase transformations that occur during weld solidification are made possible using synchrotron radiation and provide invaluable data to support scientific modeling and simulation and leads to better electrode design. The later work received the 2001 Spararagen Award from the American Welding Society. A current application in electrode design is the new understanding of the consequences of "self shielding"--that is preventing adverse reactions with air through additions of aluminum or other powders to the electrodes on compound (phase) stability leading to understanding of potential changes in electrode composition on properties of welded components. Recognitions include the two recipients of the Warren F. Savage Award from the American Welding Society

In the self-assembly area, developing scientific understanding of surfactant interactions with ceramic compounds and other materials, including biological tissues, has permitted the growth of ordered porous ceramic structures duplicating template structures from the nanoscale to the macro scale.

Specific achievements include:

- Investigations of self-assembled heteroepitaxial semiconductor quantum dots using real-time stress sensing and light scattering showed how elastic repulsion determines evolution of dot arrays. Repulsion promotes spatial ordering, accelerates ripening kinetics, and enhances quantum dot phase transition.
- A rapid, efficient self-assembly process for making nanophase composites that mimic the complex construction of seashells was developed and it resulted in a strong and tough (crack resistant on impact loading) material.
- Ceramic substrates were synthesized with tailored and regularly ordered nanoscale pore sizes of controlled shapes and sizes. It was then found that these substrates would remove deadly heavy metals such as mercury, lead and silver from water.
- A breakthrough in the fundamental understanding of the processing of ceramic aerogel films led to a new, non-toxic, low temperature and low-pressure process to produce such films in an environmentally benign manner. This discovery overcame the sixty-year barrier to the large-scale commercial utilization of these films, won the prestigious Iler Award of the American Chemical Society and was cited as an important discovery by the Wall Street Journal.
- Established a Materials Preparation Center to provide outside researchers from academia, industry and government laboratories with research quality and quantities of unique, carefully controlled research-grade materials and crystals that would not otherwise be available. The following technologies were enabled by this Center:
  - ♦ Lead free solder
  - ♦ Magnetocaloric gadolinium-silicon-germanium alloys
  - ♦ Recyclable lightweight automotive composite materials
  - ♦ Terfenol-D which is a mangetostrictive alloy containing terbium, dysprosium, and iron that was developed at the Center and led to the spin-off of a new private sector company which now markets this material
- Quasicrystal coatings produced by plasma-arc spray that have superior wear resistance and thermal insulation behavior coupled with reduced surface friction for potential thermal barrier wear resistant coating applications in aircraft-engine components.
- A uniform three-dimensional coating process known as "Plasma Ion Immersion Processing" was improved so as to fabricate hard coating, such as diamond-like carbon, that exhibit low sliding friction and superior wear resistance. This process achieves a uniform implantation rate over a very large surface, a very high rate of implantation, and has the ability to produce a uniform thickness and quality coating over complex three-dimensional geometries, and is cost-effective.
- A nanophase molecular template method was developed to synthesize films that exploit the dielectric properties of air to achieve ultra-low dielectric constants for the next generation of microelectronic devices and computers.

Mission Relevance:

This research supports the Department of Energy's overarching goals for improved energy efficiency and protection of the environment. Specific relevant applications include hard and wear resistant surfaces to reduce friction and wear; high-rate, superplastic forming of light-weight, metallic alloys for fuel efficient vehicles and other structures needed in fuel-efficient land and air transportation applications; high-temperature structural ceramics and ceramic matrix composites for high-speed cutting tools, bearings, engines and turbines (to enable fuel efficiency and low-pollutant emissions); ordered intermetallic alloys for harsh applications (requiring heat, load, wear and corrosion resistance), including engines and turbines (also to enable fuel efficiency and low pollutant emissions); non-destructive analysis for early warning of impending failure and on-line flaw detection and quality assurance during production; response of magnetic materials to applied static and cyclical stress; and plasma, laser, and charged particle beam surface modification to increase corrosion and wear resistance; and welding and joining, including dissimilar and non-metallic materials.

Scientific Challenges:

Understanding the physics and chemistry for the synthesis and processing, as well as the thermodynamics and kinetics of reaction of nanoscale materials and structures and the elements of the processing environment are critical to the preparation of larger components. There are significant experimental, theoretical and computational challenges in understanding what is occurring so that the benefits of nanoscale phenomena can be realized in larger scale components. Major scientific challenges also remain in the fabrication and the fundamental understanding in the non-trivial assemblies of inorganic, organic and biomimetic materials. This research activity will develop carefully designed experiments to directly compare measured behavior to results of systems modeling.

For thin films systems, future efforts are required to solve materials problems such as the adhesion and the thermal and environmental stability. Although there is steady progress in the synthesis and processing of materials, there still exists a serious deficit in the ability to produce (new) materials with desired properties and microstructures by rational design and synthesis. Experimental methods and theoretical models need to be developed to achieve mesoscopic structures perhaps via self-organized growth using various kinds of templates. Scientific challenges also lie in new composite materials with various matrices, and in ecologically-benign materials.

Funding Summary:

Dollars in Thousands

FY 2002	FY 2003 Request	FY 2004 Request
14,497	18,595	18,570

Performer	Funding Percentage
DOE Laboratories	78.5%
Universities	21.5%

These are percentages of the operating research expenditures in this area; they do not contain laboratory capital equipment, infrastructure, or other non-operating components.

Projected Evolution:

- Increased emphasis on understanding the opportunities and challenges presented by nanoscale materials and by the processing of larger components containing nanoscale materials.
- Science based understanding of advanced synthesis and processing methods such as self-assembly, molecular-directed nanostructure formation, and novel deposition methods will be investigated. This understanding will be applied of to attain new structures and compositions, to fabricate materials with new functionalities, and to reduce the energy and environmental impact of processing.
- Processing research will be extended to include new ceramic, intermetallic, semiconducting, organic and biomimetic materials and material structures, including nanocrystalline materials, films, coatings, and crystals. Analytical techniques and modeling will be developed and applied to determine and predict the relationship of synthesis and processing parameters to structure, purity, deformability, residual stresses, toughness, adhesion, and electronic, optical and magnetic properties.
- Welding process models will be refined and extended to solidification of aluminum alloys. The relationship of process conditions to microstructure development in various alloy welds will be studied by real-time, *in-situ* x-ray diffraction enabled by synchrotron radiation. Follow-on work will investigate supercooling effects in welding, and determine the residual stress distribution in welds and spatial distribution of different phases in different locations of the weld heat-affected-zone.
- Processing capabilities of the Materials Preparation Center will be expanded to provide intelligent process control and modeling and to provide new innovative synthesis and processing techniques. Plans include the use of a plasma-heated skull-melting furnace with capabilities to cast or atomize materials. Follow on efforts in microstructure-sensitive processing and providing real-time monitoring of processing and correlation with process models.
- An understanding of interfacial interactions at the molecular level will be developed, and their influence on molecular conformation and ordering will be shown for evolving nanostructures. An understanding of how short and long-range molecular forces mitigate the formation of ordered amorphous structures will be extended to develop an understanding of how biological systems control the formation of ordered assemblages of crystalline nanoparticles.