DOE/SC-0019

Report of the
Basic Energy Sciences Advisory Committee
Subpanel Review of the
Electron Beam Microcharacterization Centers
Past, Present, and Future

February 2000

U.S. Department of Energy
Office of Science

UNIVERSITY OF OREGON

10 March 2000

Dr. James Decker
Acting Director
Office of Science
U.S. Department of Energy
19901 Germantown Road
Germantown, Maryland 20874-1290

Dear Dr. Decker,

I would like to express my appreciation for your attendance and presentation at our Basic Energy Science Advisory Committee (BESAC) meeting last week. It is encouraging to see the proposed budget increases for the Office of Science and Basic Energy Sciences (BES). As a Committee we are committed to helping to make the proposed budget a reality.

At our meeting three Subpanel reports were presented addressing the recent charges given to us by former Director of Science, Martha Krebs. The three reports submitted by the Subpanels pertained to Neutron Scattering in light of the recent shutdown of the High Flux Beam Reactor (HFBR) at Brookhaven National Laboratory (BNL), a review of the Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory (LBNL), and a review of the Electron Beam Microcharacterization Centers at Oak Ridge National Laboratory (ORNL), University of Illinois, Argonne National Laboratory (ANL), and LBNL. The purpose of this letter is to forward to you the reports of these Subpanels and the response of BESAC to these reports. Overall, the BESAC members are supportive of the recommendations of the Subpanels. We are appreciative of the tremendous amount of work that Panelists and BES staff contributed to these important planning and review exercises.

Neutron Scattering Research Capabilities

The purpose of this Subpanel, chaired by Dr. Martin Blume, was to recommend steps to provide the best possible neutron scattering research capabilities in the United States in the near term. Subpanel deliberations took into account the shutdown of the High Flux Beam Reactor at BNL and assumed that the Spallation Neutron Source at ORNL would be operational in a timely manner. The Subpanel was also asked to provide advice on how to properly accommodate the neutron scattering groups at BNL, conditional on their submitting satisfactory long-term plans for programs to be funded by BES.

Neutron scattering is a critical tool in the arsenal of experimental techniques for studying condensed matter systems. It will be particularly valuable for studies in nanotechnology and nanoscience. BESAC is committed to assuring that neutron scattering science in this country retains its world-class standing and to supporting facilities that allow scientists to

conduct first-rate science in this area. BESAC commends the Subpanel for the high quality of the submitted report, recognizing the short time constraints imposed by the need to assure continuity in the field in light of the HFBR shutdown. BESAC supports the general recommendations of the report that is provided with this letter. However, with respect to the funding recommendations, first BESAC regards these numbers as estimates requiring detailed review. Second, several factors need to be considered before funding decisions are made, including determination of what costs are currently in the FY 2001 budget, the shutdown costs of HFBR, and the anticipated growth in the number of users over the next few years as the other neutron scattering facilities increase their operations. BESAC however felt strongly that any increase for the existing facilities should not come at the expense of core BES programs. The funding for research and instrumentation should be competitive with the core program.

Review of the Electron Beam Microcharacterization Centers

BESAC's charge was to help assess the scientific impact of the nation's need for the Electron Beam Microcharacterization Centers operated by BES. To this end a Subpanel of experts was assembled and chaired by Dr. John Stringer. The four centers considered were the Shared Research Equipment Program at ORNL, the Center for Microanalysis of Materials Research Laboratory at the University of Illinois Frederick Seitz Materials Research Laboratory, the Electron Microscopy Center for Materials Research at ANL, and the National Center for Electron Microscopy at Lawrence Berkeley National Laboratory. The Subpanel visited each of the four centers and met with members of their management, staff and user communities. The recommendations of this group are summarized in the enclosed report. The Subpanel's review was a monumental effort and BESAC expresses its appreciation for the efforts of the committee, the chair and the BES staff.

In general these facilities were found to operate well and produce excellent science. BESAC is supportive of the recommendations found in the report. The recommendations have been carefully derived and attention has been paid to the unique nature of different facilities. BESAC accepted the recommendations provided that any additional funds allocated to these centers as a result of the review be competitive with the core BES program.

Review of the Advanced Light Source

BESAC was charged in August 1999 with reviewing the Advanced Light Source (ALS) at LBNL. The purpose of the review was to examine those issues that were raised by the BESAC report on "DOE Synchrotron Radiation Sources and Science," known as the Birgeneau Report. In particular, BESAC was asked to explore ALS's vision for the future, the quality and diversity of the science program at the facility, the user demand, and the interaction and relationship with the user committee. The Subpanel charged with this study was chaired by Dr. Yves Petroff and consisted of expert scientists from a broad spectrum of scientific areas.

The Subpanel gave an enthusiastic review of the ALS. The response of the management of the ALS to criticism in the Birgeneau Report has led to a restructuring of LBNL to raise the ALS to the divisional level. The user hours have dramatically increased, and the user participation in the ALS decision making process has been welcomed by the users. Most important is the high quality of the science being generated at the ALS. LBNL

Director Chuck Shank and ALS Director Daniel Chemla are commended for this impressive turn around. BESAC accepted the recommendations of the subpanel provided that any increase in funding to the ALS as a result of this positive review not come at the expense of the BES core program. Increases in funding for beamlines should be competitive with the core program.

Thank you again for attending our BESAC meeting and giving us your insights into the FY 2001 budget process.

Enclosures

Sincerely,

/s/ by

Geraldine L. Richmond, Chair
Basic Energy Sciences Advisory Committee

cc:
Iran Thomas, Acting Director of Basic Energy Sciences
Patricia Dehmer, Acting Deputy Director of the Office of Science
Sharon Long, BES

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Report of the Basic Energy Sciences Advisory Committee Subpanel Review of the Electron Beam Microcharacterization Centers Past, Present, and Future

Kickoff Meeting - August 1999

Marriott Washingtonian Center
Gaithersburg, MD.

Center Reviews - December 1999

Shared Research Equipment Program (SHaRE),
Oak Ridge National Laboratory,
Oak Ridge, TN.

Center for Microanalysis of Materials,
Frederick Seitz Materials Research Laboratory,
University of Illinois,
Urbana, IL.

Electron Microscopy Center for Materials Research,
Argonne National Laboratory,
Argonne, IL.

National Center for Electron Microscopy,
Lawrence Berkeley National Laboratory,
Berkeley, CA.

Subpanel Review of the
Electron Beam Microcharacterization Centers
Past, Present, and Future

Subpanel Members

John Stringer, EPRI (Chairman)
Clyde Briant, Brown University
M. Grace Burke, Bechtel Bettis, Inc.
Vinayak Dravid, Northwestern University
Derek Hull, University of Liverpool
John Mundy, Retired
Gregory Olson, Northwestern University
John Poate, New Jersey Institute of Technology
Manfred Ruehle, Max Planck Institute fur Metallforschung
John Spence, Arizona State University
George Weatherly, McMaster University
Miguel Yacaman, Centro Nuclear 'Dr. Nabor Carillo'

Associate Subpanel Members from Centers

Charles Allen, Argonne National Laboratory
James Bentley, Oak Ridge National Laboratory
Joe Greene, University of Illinois
Kannan Krishnan, Lawrence Berkeley National Laboratory

Report Submitted on Behalf of the Subpanel:

***/s/* by**

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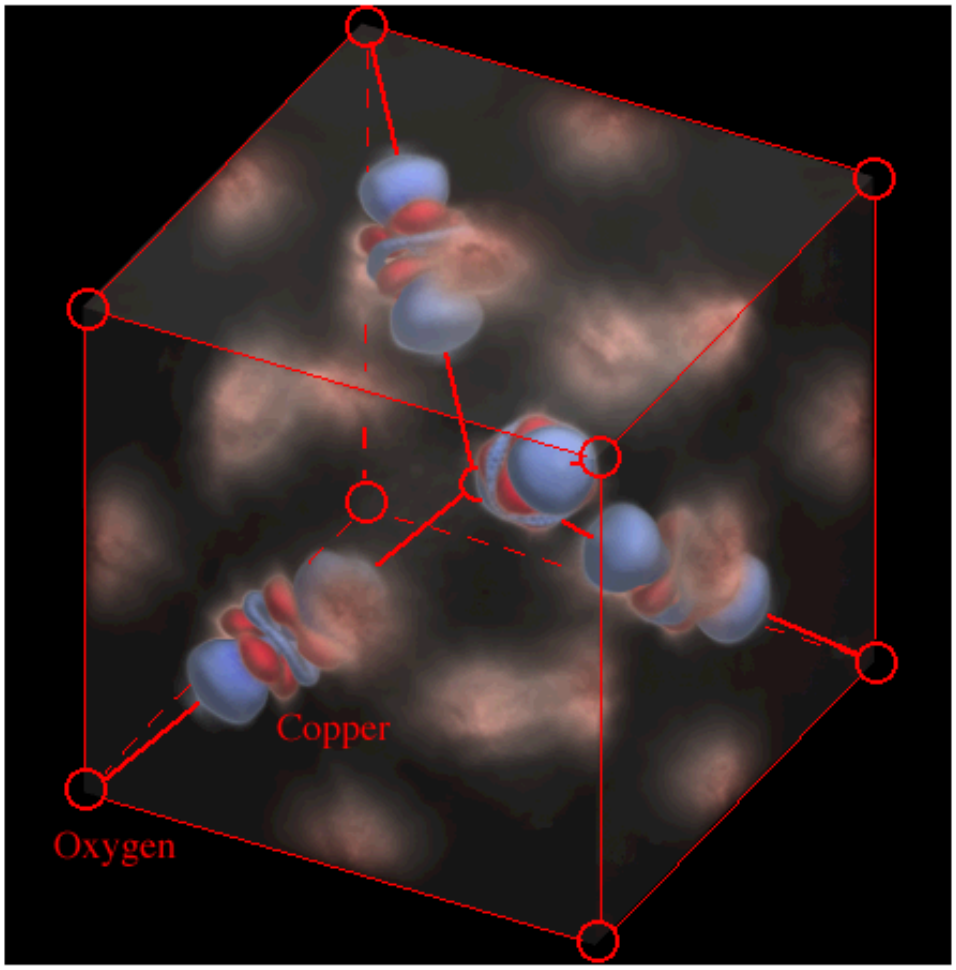
John Stringer, Chair

Subpanel Review of the Electron Beam Microcharacterization Centers

Past, Present, and Future



The cloud of bonding electrons in copper oxide (cuprite) is shown as measured by a combination of extinction-free quantitative electron diffraction (using a TEM) and X-ray diffraction. Subtraction of the ion background has produced the classical dz^2 orbital shape of a charge "hole" (shown in blue), and also shows metal-metal bonding. Modern TEM instruments produce highly accurate quantitative data. (Zuo, Kim O'Keeffe and Spence, Nature 401, p. 49, 1999, ASU)

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1.0 EXECUTIVE SUMMARY

1.1 Introduction

Over the last few years, the Office of Basic Energy Sciences (OBES) of the Department of Energy (DOE) have asked the Basic Energy Sciences Advisory Committee (BESAC) to review the user centers which it has established and maintains. The first group of these reviews was concerned with the major facilities: the neutron sources and the synchrotron radiation sources. This review involved the Electron Beam Microcharacterization Centers (EBMCCs). As the charge letter to this review panel points out, "The centers differ from Basic Energy Sciences major user facilities such as the synchrotron radiation light sources or the neutron sources in that they do not have distinct "operating budgets"; they are supported as part the Materials Science Division research budget. Furthermore, each of them can be regarded as a suite of instruments aimed at using electron beams to characterize materials with high resolution, both structurally and chemically."

The four centers reviewed include: the Shared Research Equipment Program at Oak Ridge National Laboratory (SHaRE); the Center for Microanalysis of Materials at the University of Illinois Frederick Seitz Materials Research Laboratory (CMM); the Electron Microscopy Center for Materials Research at Argonne National Laboratory (EMC); and the National Center for Electron Microscopy at Lawrence Berkeley National Laboratory (NCEM).

BESAC asked Dr. John Stringer to assemble a panel of experts to conduct this review. Prior to the selection of the panel, he asked the Directors of the four centers to identify the principal areas of materials science to which they believed they were contributing. These were:

- Interface Science
- Phase Transformations and Alloy Design
- Defects, Deformation and Radiation Effects
- Nanostructures
- Thin Film and Surface Science
- Microelectronic Materials

They noted that not all of the centers worked in all of these areas. A panel was then selected which had expertise in electron beam microcharacterization techniques and in the materials science topics listed above. The panel included four members from outside the United States to place the centers in an international context.

The review process itself involved first a meeting at which the centers as a group described the contributions that electron beam microcharacterization techniques are making to materials science, and described some of their most significant achievements in the areas listed above. Following this, the panel prepared a list of 15 questions for each center to address. The second part of the review was a visit to each of the centers to assess their individual contributions.

The following report describes the panel's findings in considerable detail. In this summary are listed the answers to the questions in the Charge Letter to the panel from the Chair of BESAC, Professor Geraldine Richmond; and the panel's recommendations to BESAC.

The panel wishes to express their gratitude to the four centers for their responsiveness to our questions, and for their courtesy extended to us during our visits to their establishments.

1.2 The Questions in the Charge Letter and the Panel's Answers

(1) What has been the scientific and technological impact of the microcharacterization centers during the past decade, and what is to be expected during the coming decade? In particular, what scientific studies are enabled by the centers that could not otherwise be done?

Collectively, the centers have contributed a significant amount to the development of the application of advanced electron beam microcharacterization techniques to issues of interest in materials science. Specifically, the unique capabilities such as the HVEM/IVEM-Tandem facility at EMC/ANL, the ARM at NCEM/LBNL, the APFIM at SHaRE/ORNL, and the controlled environment TEM at CMM/UIUC have all made important contributions. The studies enabled by the ability of the linear accelerators to inject energetic ions to the specimens in the high-voltage microscopes in the Tandem contribute a great deal to our understanding of the potential radiation damage of materials. This is particularly relevant at the moment, because of concerns related to the selection of materials for the long-time storage of highly radioactive wastes. The drive toward quantitative atomic-scale imaging at NCEM has led to important advances in our understanding of key features that determine the properties of materials, such as the atomic structure of nanophase particles, defects and interfaces. This effort has made a significant contribution and culminated in the recent introduction of the One-Ångstrom Microscope, which allows the investigation of solids that contain light elements such as C, N and O. The insights gained into the mechanisms of hydrogen embrittlement at CMM including the effect of hydrogen pressure on enhancing the dislocation velocity and the crack propagation rate led to more recent research on the consequences for dislocation-obstacle interactions. In particular, solute hydrogen has been observed to reduce the propensity for cross-slip by stabilizing the dislocation segment with high edge character. Perhaps the most remarkable developments over the last few years from the point of view of materials science have been those involving the developments in high spatial resolution analytical electron microscopy and atom probe field ion microscopy at SHaRE. In particular, the demonstration of the radiation-induced segregation to grain boundaries in austenitic stainless steels over distances of less than 5nm at temperatures typical of light water nuclear reactors has provided insight into the phenomenon of irradiation-assisted stress corrosion cracking.

All of these examples could not have been accomplished without the special capabilities in equipment and staff at the centers. It is highly important to recognize that all of the

above examples are representative of the unique strength of electron beam microcharacterization techniques, the capability due to small probe size (down to 1 Ångstrom) of examining nanoscale problems. In the next decade the drive toward nanotechnology will demand increasing use of electron beam microcharacterization and the users from a breadth of disciplines will require the experience and expertise of the four EBMCCs.

Also in the future, the currently-planned developments and recently developed instruments at the centers offer further promise. These include the interesting results starting to come from the Spin Polarized Low Energy Electron Microscope (SPLEEM), at NCEM, which is one of only two SPLEEMs in the world; the proposed 'ARM III', also at NCEM; the Kindbrisk ECOPoSAP energy-compensated position-sensitive atom probe at SHaRE, which is one of two in the U.S., one of only three ECOPoSAPs in the world; and the combination of high temperature (up to 1500 C) LEEM and STM at CMM.

The existing and projected facilities are discussed in considerably more detail in the panel report.

(2) *What are the user groups served by each of the centers? How do they differ? What is the user demand at each of the centers, and how is it expected to change?*

Three of the centers grew out of electron beam microcharacterization groups whose original function was to support the Materials Research groups in their institutions, and to a greater or lesser extent users from these groups are still a very significant part of their customers. The exception was the NCEM at LBNL, which was intended as a National Center from the beginning, and primarily was intended to develop high resolution electron microscopy. Even at NCEM, there was considerable interaction with the Materials Departments in LBNL and the University of California (Berkeley): the interaction with the University as such is mainly through staff who also hold positions at LBNL. CMM was created specifically to serve as an essential component of the BES MSD materials research program at FS-MRL. CMM receives no direct recurring funding from MSD and therefore presently operates primarily through user fees (67%) and direct support from the University of Illinois (24.5%).

Originally, the centers were called Collaborative Research Centers. The intention was to distinguish these from the major user facilities and to give the expectation that people wanting to make use of the instruments had to collaborate rather than expect formal procedures and user support. However, since the review of the centers by the Council on Materials Sciences (written in 1987) there has been a progressive move towards a user center approach. In all of the centers, there is, however, still a very clear pattern of engaging in collaborative research with users; the panel believe that this is not inappropriate to the field of advanced electron beam microcharacterization, because of the nature of the major instruments.

The centers presented the panel with very detailed analyses of their user demographics, and this is discussed in more detail in the panel report. Essentially, the users (or collaborators) are in six groups: those from the 'parent' Materials Science Department; those from other Departments in the same institution; those from other National Laboratories; those from U.S. academic institutions; those from foreign institutions; and those from industry. The data are presented in detail in the body of the report, and show that in general the dominant users are materials scientists from their 'home' institutions. This is most obvious in CMM, where 93% of the users were from UIUC one way or another. However, the total users at CMM are very large, and 62% of them are non-DOE supported; the small percentage of non-UIUC users (7%) is still 27 individuals. The panel was nevertheless concerned with the very high usage of the CMM by UIUC-based researchers. This was noted also by the 1987 Council on Material Science review, which remarked "Off-campus usage is not extensive, nor should it be encouraged further. There is quite sufficient work at Illinois without the facility being burdened by outside projects." The present panel believes that the change in the role of the centers now required by OBES makes this conclusion much less justifiable.

Generally, industrial users at the centers were less than 5%. There has been relatively little change in the user numbers over the last three or four years. The centers believe that the new instruments they hope to install, and the opening to users of some of the more advanced instruments, will allow them to increase their user base. The panel believes that there needs to be a greater effort on the part of the centers to develop increased user interest, and in particular feel that the very low usage by U.S. industry is a cause for concern: in the past, the high-technology materials-based industries have either had their own facilities, or have used instruments in local universities or local consultants, but with the development of newer nanotechnologies at increasingly finer scales, the more advanced techniques available at the centers ought to have a larger role to play.

(3) What special needs do each of the centers serve, and how do the centers complement one another?

This is dealt with in more detail in the answer to the next question, and in the panel report; but in summary:

SHaRE addresses the variations in chemical composition and phase structure at length scales in the range 1nm - 1μm in complex engineering alloys, relating these to the mechanical properties of the materials. They interact closely with materials scientists and engineers.

EMC offers a unique user facility capable of looking at radiation damage produced by energetic ions in situ. This relates to the damage experienced by structural materials in nuclear reactors, and the deterioration that might be experienced in radioactive waste storage.

NCEM studies materials at the atomic resolution scale, and is capable of determining the structure and properties of defects such as dislocations and interfaces. Their interactions are mainly with materials scientists concerned with the more fundamental treatment of the mechanical properties of materials. This is, of course, one of the most important areas of materials science: to quote James E. Gordon, it is why we don't fall through the floor!

CMM has expertise in some specific environmental studies, notably the effect of hydrogen on crack growth. They also provide training in electron beam microcharacterization techniques to a considerable number (>150 individuals per year) of investigators at the graduate and postgraduate level.

All of the centers are moving toward higher resolution techniques, capable of lattice imaging. Essentially all are moving towards instruments capable of providing chemical analysis at a fine scale. The issues of surfaces and interfaces in practical materials, for which electron beam microcharacterization is a very appropriate tool, are increasingly being studied at all the centers.

There is good interaction between the centers in various ways. Members from one center will use the facilities at other centers, as appropriate. The Advisory Committees for the centers frequently have representatives from the other centers. The three National Laboratory Centers are increasing their interactions with other capabilities on the same site: ECM and NCEM report that they are interacting with the light sources; ECM and SHaRE report that they are interacting with the neutron sources; and SHaRE is interacting with the High Temperature Materials Laboratory and with the group led by Dr. Steven Pennycook in the Solid State Division at ORNL. The CMM is very closely integrated with the activities of the Frederick Seitz Materials Research Center at UIUC.

At each of the centers, the panel had lunch with a group of users in the absence of the center staff. All of the users expressed nothing but the highest regard for the level of performance of the instruments, and the support they received from all the staff.

(4) *What is the vision of each center? Are the visions appropriate? How do the visions complement each other? Is there anything missing in the set of visions for the future?*

The visions of the four centers can be briefly summarized as follows.

The Shared Research Equipment (SHaRE) User Facility at Oak Ridge National Laboratory sees its contribution as providing, developing and maintaining state-of-the art instrumentation for the microscopy and microanalysis of materials, while developing, refining and applying a diverse array of microanalytical techniques for collaborative materials research and development efforts that are central to the mission of DOE. In particular, they aim to provide a resource of expertise and instrumentation for the materials research and development community to address problems of major scientific and technological impact through quantitative microscopy and microanalysis of materials

at length scales from one micrometer down to atomic level. Instrumentation and technique development will continue to focus on three areas of established core competency: analytical electron microscopy, atom probe field ion microscopy, and mechanical properties microanalysis. In addition the SHaRE user facility provides a unique resource for the microcharacterization of radioactive specimens on a routine basis.

The Electron Microscopy Center at Argonne National Laboratory provides facilities and personnel to serve the research needs of the Materials Science Division and other Divisions at ANL, to perform collaborative research with several area universities, and to serve a group of national and international users. The principal user capabilities historically have centered on a unique facility involving ion accelerators coupled with an older high-voltage electron microscope and a newer Intermediate Voltage Electron Microscope to study the microscopical effects of radiation damage in materials. New instrumentation will be coming to ANL in the near future, including high-resolution field-emission gun scanning and transmission energy-filtered electron microscopes.

The National Center for Electron Microscopy at Lawrence Berkeley National Laboratory sees its mission to provide forefront instrumentation and techniques for advanced electron beam microcharacterization of materials at high spatial resolution. The broadest challenge to electron beam microcharacterization over the next decade will be to develop the technique into a fully quantitative tool. To meet this challenge, NCEM is planning to further the development of:

- New methods for quantitative image analysis, processing and interpretation;
- New methods and tools for sample preparation; and
- New electron-optical instrumentation and stages.

The Center for the Microanalysis of Materials in the Frederick Seitz Memorial Research Laboratory at the University of Illinois at Champaign-Urbana regards its principal goals as:

- To contribute to the excellence of, as well as to enable, material science research at the Frederick Seitz Materials Research Laboratory with a focus on the mission of the DOE/OBES/DMS program.
- To develop the science of microstructural and microchemical analyses.
- To assist researchers within the community of materials science scholars at UIUC in applying modern microstructural and microanalytic techniques in their research.
- To educate graduate students and research associates in the use of modern microstructural and microanalytic techniques.
- To make modern microstructural and microchemical characterization tools available to the broader scientific community in the U.S.

The center regards itself as a major repository of instrumentation and expertise focused on the microcharacterization of materials, with a staff teaching the use of the instruments, assisting in the interpretation of experimental results, developing new instruments and

techniques, and carrying out research in the area of instrument science. The housing of the center in the FS-MRL leads to close interactions with the FS-MRL/UIUC staff.

The visions of the three National Laboratory Centers are consistent with their historical roles, and are complementary. They are also consistent with the expectations of the Office of Basic Energy Sciences. It must be remembered that, as indicated before, these three centers are, to different degrees, 'in construction'; and it is worth asking to what extent these traditional roles are still valid. The SHaRE and the NCEM are in good positions in their development, and the evolution of their visions seems entirely appropriate. The EMC is at an earlier stage, and it is worth asking to what extent they need to move beyond their traditional expertise in radiation damage. The national importance of the problem of the storage of radioactive waste suggests that this is still an important area. Their extension to other areas, particularly magnetic and superconducting nanostructures, and their intention to increase interactions with the APS and the INPS, seem appropriate: the developments in these new directions will need to be encouraged.

The vision of CMM is different, but is relevant to a center situated in a University, and embedded in a Materials Research Laboratory, in that it is largely aimed at their teaching role and the support of the materials research activities in the FS-MRL and the University.

What is missing is a positive effort to find out in what ways the centers could support the needs of U.S. industry; and this is an issue on which they need guidance from OBES.

(5) *How does the use of electron beams for characterizing materials complement the use of photons and neutrons?*

Complementarity of information has always been important to materials science because advances are made through a complex interplay of studies of processing characterization, properties and theory. From the earliest days of studying the behavior of materials, the importance of characterization has been recognized. At first, optical microscopy was the primary tool, and this is still an important technique. It provides information on aspects such as grain size and shape, phase distribution, and (with associated experiments such as heat treatment) kinetics of transformations. Although the information from a single examination is derived from the surface of the specimen, metallographers use sectioning techniques to extend the information into three dimensions. Next, X-ray diffraction was introduced, providing information on the crystal structure of the phases in the material, and, through studies such as line broadening, understanding of internal strains and compositional inhomogeneities. Pole figure studies allow determination of preferred orientation in polycrystalline materials, to mention one other contribution. Because of their shorter wavelengths, the penetration distance of the X-rays is significantly greater than that of light. Neutron scattering methods have made very significant contributions in recent years in crystallography and in internal strain studies, for example; the ability to detect hydrogen, and the considerable penetrating power of neutrons, particularly in ferrous alloys, have been of great value.

However, the properties of materials are very sensitive to crystalline defects such as vacancies, dislocations, and stacking faults; and to chemical and compositional features and variations on a fine scale such as grain boundary segregation. The introduction of electron beam techniques has allowed high resolution microscopy capable of providing three-dimensional information on atomic-scale defects, and high resolution microanalysis permits the determination of three-dimensional compositional variations with a resolution of the order of 0.1μm or less. In the case of the atom probe techniques developed at Oak Ridge National Laboratory, compositional variations at the atomic scale can be determined.

In terms of the general area of materials characterization, as Dr. J. Murray Gibson pointed out in his presentation to us at out meeting in Gaithersburg, there are a couple of thousand scanning electron microscopes with compositional analysis capabilities and approximately 400 transmission electron microscopes in the U.S. These provide an extremely powerful capability for the characterization of materials. The classical photon techniques of conventional optical microscopy and X-Ray diffraction are also widespread in materials facilities, and it is normal in materials research to use all these tools to address problems and to develop understanding.

Of course, we recognize that this question also refers to the research that is performed at the national neutron sources and synchrotron radiation light sources. As mentioned in the answer to question (3), EMC, NCEM, and SHaRE all report that they are increasing their interactions with the large user facilities on the same site. This is not yet particularly evident in the research results described to us: without the benefit of more careful analysis the panel had expected a greater amount of interaction. The Frederick Seitz Materials Research Laboratory maintains two sectors (four beam lines) at the Advanced Photon Source at ANL, and one sector (two beam lines) at the National Synchrotron Light Source at Brookhaven National Laboratory; CMM users interact heavily with these. A good example of how these three techniques provide complementary information is the work done on the 'high-temperature' perovskite superconductors. The first determination of the crystal structure of these layered materials came from neutron diffraction. The synchrotron radiation light sources have made important contributions to the understanding of how these materials become superconducting. However, in practical terms, the important aspect of these materials is that they are Type II superconductors, and their properties are related to the pinning of the flux vortices. This pinning is by crystalline defects of various kinds: and the determination of these, and such aspects as the effect of grain boundary segregation on the properties of the materials, have come as a result of the application of electron beam microcharacterization techniques. There are many other examples.

(6) *What are the opportunities for improving the techniques to maintain the facilities at the forefront?*

This question is of particular importance. For a center to be of value, and to attract users, it must have equipment that is at, or close to, the state-of-the-art. It is important to define what is meant by this statement. There are, of course, very advanced microscopes and

other microcharacterization facilities that are associated with specialist research teams; and it can be argued that these 'leading edge' instruments are ahead of what even the most advanced user (as that term is commonly understood) would have need for. A measure might be the most advanced equipment that is available from the manufacturers, with only a normal level of modification. Another measure might be equipment which has been developed by the specialist research teams, but has reached a level of maturity which makes it accessible to the general user, helped and guided by the center staff. The centers, to varying degrees, have suffered, or are suffering, from significant gaps of investment in updating their facilities over the last few years, although most appear to a limited extent to be updating their equipment at the moment. The level of equipment, and the particular capabilities required by each center, will be a function of their visions. What is required for a viable center is perhaps four or five instruments which are at least state-of-the-art which relate to their mission, and three or four 'core' instruments for preliminary assessment of specimens and for training. In the panel report there is a complete description of the equipment at each of the centers, and this will not be repeated here. Maintaining the facilities at the forefront involves three components.

First the existing equipment must be maintained in as high a quality as possible. Generally, this will mean that the state-of-the-art instruments will have service contracts from the manufacturers, backed up by a highly-skilled maintenance staff at the center. Second, the maximum lifetime of a state-of-the-art instrument is ten years, and a new state-of-the –art instrument will have to be introduced every two or three years. Third, the majority of the developments over the last decade have been not in completely new instruments, so much as the introduction of new devices which can be added to existing instruments. Examples include EDS, EELS, PEELS, Omega filters, imaging energy filters, Cathodoluminescence in STEM at helium temperatures, ALCHEMI, the CCD camera for TEM, video recording, Field-Emission Guns (FEG), Nanodiffraction using a FEG, Z-contrast detectors, LaB_6 sources, (there are several other examples). Together these devices have made the biggest difference to Transmission Electron Microscopy over the past two decades, with the exception of High Resolution microscopes, which (of course) are also very important.

At NCEM, a vigorous program of adding new capabilities has been in progress for the last three or four years, with a Philips CM200/FEG installed in 1996, a One-Ångstrom Microscope (OÅM) and a SPLEEM in 1997, and a FESEM (a JEOL JSM6340F) installed in 1999.

At the EMC, the most recent addition was in 1995, a 300kV Hitachi H-9000NAR high resolution microscope which allows *in situ* ion irradiation, and is the IVEM part of the HVEM/IVEM-Tandem Facility.

The SHaRE Facility has a somewhat more modern suite of equipment. Atom probes are a very important part of their capability, and a Kindbrisk ECOPoSAP was delivered in 1997: this is one of only two such instruments in the U.S., and one of only 3 in the world. Another energy-compensated atom probe has been designed and built at ORNL, but this

is used primarily for detector and software development, and is not available to outside users.

The statement from CMM points out that "each of the instruments in the CMM was purchased and is maintained in response to a significant, demonstrated need by DOE research programs in the FS-MRL. All of the instruments are also used by other researchers on the UIUC campus, throughout the State of Illinois, and nationally". Two new microscopes will be installed this year: a JEOL FASTEM 2010F, and a JEOL 2010. CMM also have other electron beam microcharacterization instruments: most recently a Hitachi S4700 low-voltage high-resolution FEG-SEM was installed in 1998; and a R. Tromp Design LEEM was installed in 1997

In this discussion of the appropriate fleet of instruments expected at a viable National User Center, it must be remembered that specimen preparation capabilities, and computer capabilities for the appropriate development and interpretation of information from the microcharacterization facilities, have also to be provided, maintained and updated.

In addition to these rather straightforward statements, we discussed the expected changes in electron beam characterization in the future. The centers discussed two areas with us: the first, which is already in progress, is the Materials Microcharacterization Collaboratory (MMC). This aims to bring the EBMC tools available at the centers to geographically-dispersed researchers working in industries, universities, and government laboratories using internet-based technologies. During our visits to the centers we were shown several examples of how this was working for a number of instruments. The panel recognizes the enthusiasm and initiative of the staff of the centers in introducing remote usage to the field of electron beam microcharacterization. The field is more complex than a number of other characterization techniques, and the staff are to be congratulated on their initiative and perseverance. Opinions of the panel members were somewhat mixed: most felt that it was an interesting and useful venture, and the way the future lies. These members also believed that the National User Centers were the appropriate places for this technology to be developed, and remarked what an excellent example of the close collaboration between the centers it was. Some panel members, without dissenting from these views, nevertheless expressed some concerns about the negative effect this approach would have on the close personal interaction between the users and the expert staff of the centers which they felt to be very important.

The second area which was discussed with us was contained in a preproposal entitled "National Transmission Electron Achromatic Microscope (NTEAM)" which was given to us late in our review process. We did not feel that we could formally include this document in our review, but we did discuss the concept it presented. The preproposal remarks that "Thanks to advances in aberration correction and quantitative transmission electron microscopy, we can build a new generation of microscope capable of sub-Ångstrom image-resolution and sub-electron-volt spectroscopic-resolution with adequate space to carry out a variety of important experiments on advanced materials." This is certainly an exciting concept, and the panel strongly encourages a detailed evaluation of it by OBES. However, our major concern in this review is ensuring the health and

vitality of the user centers over the next five to ten years, and we had some concern that the importance of this might be lost in devoting the effort that would be required if the centers were assigned a primary responsibility for the NTEAM development. There is a little more discussion of this idea in our report.

One further issue in relation to this question is the matter of staff development. The number of specialist staff – and highly-skilled technical support staff – in the centers is less than we believe adequate. Furthermore, several of the senior staff have been with the centers for a long time, and are approaching retirement. The quality of the specialist staff must be at least as high as the quality of the facilities, and we believe that the recruitment of staff of this caliber will only be possible if the staff of the centers themselves have the opportunity to be involved in leading-edge research in the field of electron-beam microcharacterization. This might, for example, involve arranging for their spending some fraction of their time with devoted leading edge facilities that are not necessarily part of the centers but are conducting research related to their missions.

1.3 Recommendations

(1) The panel believes that the concept of Electron Beam Microcharacterization User Centers is very valuable to the Materials Science community, and strongly recommends that funding for them should continue to be a high priority.

(2) The panel recommends that plans for the operation and development of the centers are essential, and that the planning must involve all the centers and the Office of Basic Energy Sciences. It is probable that involving external advisors familiar with the field would also be desirable. This planning must relate also to the wider field of the role of DOE in the future developments of EBMC techniques in the U.S., and the part that the centers may be asked to play in this.

(3) The panel recommends that the centers develop long-range plans for the maintenance of their capabilities, and that OBES should also have a plan for the centers, to the extent that this is possible.

(4) Having first class instruments in a center means nothing if the high level technical staff are not also first class. There are excellent people in the existing centers at the moment, but a number of them are approaching the ends of their distinguished careers. The panel recommends that plans for their eventual successors are developed in good time. It is important to remember that appointments have to be made well before individuals retire, to permit the transfer of knowledge.

(5) The panel strongly recommends that the critical issue of specimen preparation must be addressed by the centers, and by the establishments within which they are located. The panel believes that at the moment the centers fall short of the standards required, in part because of equipment deficiencies, but largely because skilled support staff are retiring and not being replaced.

(6) The panel believes that the levels of equipment and staffing in the centers are somewhat low, and in connection with the planning recommended above, we recommend that the appropriate size and funding levels appropriate for the centers should be carefully reviewed.

(7) The panel strongly recommends support for the additional facilities listed below. With them, and the addition of appropriate staff and support, the value to the materials community of the centers would become much clearer, providing an impetus for expansion of the user base.

- A 200 or 300kV FEG-TEM/STEM with EDXS, energy-filtered imaging and diffraction, high-angle annular dark-field detector, and holography capabilities (EMC/ANL)
- ARM III, a High-Voltage High Resolution TEM, with considerable capabilities (NCEM/LBNL)
- LV-EPMA (Low-Voltage Electron Probe Microanalyzer) including a bolometer EDS detector with better than 5eV energy resolution (SHaRE/ORNL)
- SAP/LEAP (Scanning Atom Probe/Local Electrode Atom Probe) (SHaRE/ORNL)

(8) The panel recommends that all the centers make similar efforts to those that SHaRE has undertaken to make the availability of their facilities known to University departments.

(9) The panel recommends that the centers make a positive effort to determine the needs of industry in the area of nanotechnologies, since this would appear to present an opportunity for the application of advanced EBMC techniques, and develop a strategy for expanding this part of the user base

(10) The panel recommends that OBES discusses with the centers ways of addressing the issue of the travel and accommodation costs for research students using the centers.

(11) EMC has issues concerning renewal of infrastructure and personnel that concern us (see below). However, it is clear to us that the management at ANL recognizes these issues, and is committed to addressing them; we recommend that ANL's efforts to solve these problems should be supported, with a review of progress in three years time.

(12) CMM also presented us with a problem, which is also described above. We recommend that OBES studies the role of CMM within the mix of EBMC user centers, to see whether it satisfies their requirements for this role. However, we support their continued funding as an EBMC within their present context.

(13) The panel recommends that the Materials Microcharacterization Collaboratory experiment is continued. The panel overall welcomed this development, and believes that it will lead to an expansion in the users of the centers. It can, for example, reduce the financial barrier to participation that the users we met talked about. We also welcomed it as a clear sign of the centers' collaboration. A cautionary note was expressed that there would be some loss in the personal contact between the users and the center experts.

(14) The panel recommends that OBES gives favorable consideration to the development of an instrument similar to that described in the National Transmission Electron Achromatic Microscope (NTEAM) preproposal. It is our opinion that this preproposal (which we were unable to discuss in depth) offers an accurate view of the direction for the next major development in electron microscopy. We suggest that consideration of this will involve creating a review committee drawn from the electron microscope community in the U.S. to assess the proposal, and to discuss the role that the EBMCCs might play in the development. However, we are anxious that involvement with this should not deflect their interest from the user functions we have discussed in this report.

2.0 INTRODUCTION

DOE's Office of Science has as one of its major missions the support of the scientific infrastructure for fundamental research. For the characterization of materials a major part of that support is the Office of Basic Energy Sciences provision and maintenance of an array of facilities both large (synchrotron radiation sources and neutron sources) and small (electron beam microcharacterization, materials preparation, and surface modification centers). DOE has played a critical role in fostering the development and use of electron microscopy in the U.S. and so it is not surprising that the four electron beam microcharacterization centers (EBMCCs) were set up in the 1980's to provide, as noted in the EBMCCs Report (F), " ... an uncommon array of tools and expertise that form the foundation for the research of a large and diverse group of users in the National Laboratories, universities, and industrial corporations". This report will discuss the merits of the past contributions and the present work of these four centers and their plans for the future. In recent years BESAC has formed panels that provided information on the present value and future prospects of two of the characterization techniques, namely synchrotron radiation light sources, and neutron sources (both reactor and spallation sources). The present report seeks to provide the same information for electron beam microcharacterization (EBMC) and will follow, so far as possible, a similar format to the previous reports. All three characterization techniques provide complementary information but it should be noted at the outset that there are a number of fundamental differences in the three techniques which have contributed to different rates of advancement, funding patterns, and modes of operation.

Complementarity of information has always been important to materials science because advances are made through a complex interplay of the studies of processing, characterization, properties and theory. The importance of characterization has always been recognized but until this century light (photons) played a dominant role. Light microscopes had essentially reached their resolution limit of 5×10^2 nm by the end of the last century. The twentieth century has seen the introduction of many new powerful characterization tools, some still based on photons, such as visible light and X-rays, and others are based on beams of electrons (or in some cases positrons) or neutrons (or in some cases protons). Characterization results in part from an understanding of the diffraction of these beams by the patterns of atoms in the materials. Each of the beams has widely different elastic mean free paths: approximately 10^7 nm for neutrons, 10^3 nm for photons, and 10 nm for electrons, and thus the techniques provide complementary information. However the complexity of materials requires a much broader characterization than a definition of crystal structure.

Materials are substances that can be used for some useful purpose. Their suitability depends on their properties, and there is a very wide range of properties that may lead to their being useful. In the case of metals used for the fabrication of structures, the properties may be strength, ductility, toughness; but also in some cases electrical conductivity, magnetic properties, or optical properties. The chemical properties may also be important. The fundamental premise of materials science is that these useful properties are a function of a combination of the physical properties of the phases of which the material is composed; the geometrical distribution of the different phases; the chemical inhomogeneities even within a single phase; and a host of similar variables. Collectively, the set of these variables for a given material is called its structure, and materials science is studying the relationship between structure and properties. Generally, one has some control over the structure through fabrication variables (for example), and the corollary is that if our understanding is good enough it will be possible to manipulate the structure to optimize the properties for the planned-for application. In the long run, our understanding may reach the point where we can design a material system for a specific application from *ab initio* calculations. The critical element in this process is our ability to measure quantitatively the structure of materials whose properties we have separately measured.

In most cases, it turns out that it is the local imperfections in materials that determine the properties. Even for a pure metal, which is of necessity single phase, various properties will be different for a single crystal than for a polycrystal; they will vary with the size of the crystals, and with their shape; with the character of the boundaries between the grains – whether they are essentially smooth or essentially jagged, for example – and, for an anisotropic material, what the spatial distribution of the crystal orientations is. The ductility of a metal is related to the motion of linear departures from geometrical regularity in the lattice called dislocations. The electrical properties of insulators and semiconductors are very sensitive to the presence of point defects – vacancies and interstitials. In all cases, the presence of impurities in the material may be very important. In steels, for example, the migration of impurities to the grain boundaries may result in embrittlement over time, particularly at higher temperatures.

Our understanding of these relationships has grown with our ability in the very early days of this discipline to see the presence of different phases in optical microscopes, and to observe the way their distribution changed - for example, with heat-treatment – and correlating that with their mechanical properties; to the present situation, where we can use the modern electron-beam techniques to determine the structure at very fine scales, and to use this additional knowledge to begin to develop fundamental understanding of the structure/property relationships.

The appropriate dimensions at which we need to determine structure depends on the material, the application, and structural variables that are critical in determining the properties. For the kind of coarse aggregate concrete that is used for making large dams, for example, the structural features of importance may have dimensions of centimeters. For polyphase alloys such as steels the dimensions of importance may be of the order of 10^{-2} mm. However, even in these steels, a critical issue may be embrittlement as a result of the segregation of impurities to grain boundaries during service, and this requires spatial and chemical characterization at dimensions of the order of 1 – 0.1 nm. In this sense, the development of understanding has been paced by the resolution limit of our real-space imaging and microanalysis instrumentation.

Some of the clearest understanding of materials science has been obtained through electron microscopy studies. Section 3 of this report will make this point more clearly. The contribution of electron beam techniques to characterization in materials science is not simply by very high resolution techniques, although much of this report will discuss that boundary. As indicated above, microcharacterization is necessary over a wide range of length scales, from the dimensions of individual atoms to the relatively macroscopic (fracture behavior). Electron beam microcharacterization extends over much of this length scale and at high resolution (millimeters to Ångstroms). The breadth of applicability and the variety of electron beam techniques account for the fact that at least a third of the articles published in the area of materials science in recent years have utilized electron beam microcharacterization techniques.

Major advances started at the end of the last century when the resolution of light microscopes was reaching a limit. X-rays were discovered leading to an enormous leap in the use of photons for materials characterization. Another dramatic change came when it was recognized that radiation is created when relativistic charged particles within an electron accelerator are deflected by a magnetic field. The photons that are generated are of high intensity, brightness, and stability, and have a broad energy range, leading again to a large jump in the use of photons. Shull and Brockhouse's discovery of neutron diffraction after the Second World War led immediately to the rapid use of neutrons for materials characterization and again a further leap was made when spallation neutron sources were discovered. Electron beam microcharacterization has followed a much steadier rate of progress. Although the possibility of achieving high resolution by the use of electron beams has been understood for a long time, the experimental difficulties meant that electron microscopy only started in 1932. Advancements in technique have to a large extent been incremental, as one or other of the technical problems has been solved.

The evolution of electron beam techniques has typically involved a new class of instrument appearing, which has been used by a single investigator, or a small group, who develop the technique, find out what new science can be done, and optimize the instrument. The manufacturers will then offer it for sale, and it will enter the portfolio of available materials science research tools, often becoming a standard item of equipment in university materials departments. With the development of larger materials research groups, electron beam microcharacterization groups appeared, providing specialized services in support of the associated materials group. As will become clear, three of the four centers we have reviewed began their lives this way, and to a greater or lesser degree a significant amount of their time is still directed towards their historical customers. The exception was the National Electron Microscope Center at Berkeley National Laboratory, which was established primarily to develop high resolution microscopy, and originally was staffed by researchers who conducted personal research in this area, working with visiting scientists as colleagues.

The decision by OBES to create National User Centers from these four centers represented a significant change, for them and for the materials community. It is still true that essentially all university materials departments have their own electron microscope: there are something like 400 transmission electron microscopes in the U.S. Scanning Electron Microscopes with analytical capabilities are even more numerous. There are several locations where electron beam microcharacterization is a significant component of the research, and these have suites of instruments and staffs that in some cases are at least comparable to those in the centers we have reviewed.

The logic for establishing users centers is that, as will be seen, the techniques available are numerous, and materials science and engineering departments in universities, research institutes, and high technology industries, would be unable to afford the equipment or the staff required to maintain and operate them, or to identify the best technique or techniques to address a particular problem. The rate of evolution of the EBMC techniques further places an unacceptable burden on the typical broad-based materials department. In a not very systematic – or complete! - telephone survey of materials departments with interest in materials characterization, the usefulness of having user centers of the type of these four, and the appropriateness of their being funded by DOE/OBES was generally supported.

The nature of their evolution and the role they are now expected to play defines very clearly what is expected of them, and gives our panel a set of objective criteria that we can use in our assessment, and this too will be discussed later. However, it is clear that a user center will be expected to have a suite of state-of-the-art EBMC instruments, which will have to be kept updated to stay in the forefront for as long as possible; new instruments must be added in a timely way as the techniques develop. The instruments must be well-maintained. Staff having a high level of competence in the instruments and knowledge of their capabilities as research tools for materials science will also be necessary. They will be expected to support the users, but ideally a second level of younger Ph.D.s whose primary function is user support should also be part of the staff. Specimen preparation is crucial for EBMC work, and this aspect ought also to be state-

of-the-art. It is not necessary – or desirable – for the instruments to be the same at all four centers, nor is it necessary for the user communities served by them to be identical. Training users to operate the advanced instruments, and teaching them in the art of EBMC is a desirable role for the centers, since in many cases a recognition of the power of a new technique will encourage visiting users to purchase a similar instrument for their home laboratory. To keep the staff at the forefront, as well as the instruments, is very important, and they should be able to spend some significant part of their time on personal or collaborative research at a high level. These points indicate the criteria we have used in our assessment of the centers as a group, and of the individual centers.

The four EBMC centers are part of a group of smaller centers which are regarded differently by the OBES to the 'major centers' which include the synchrotron radiation light sources and the neutron sources. To quote OBES: "These four centers differ from our major user facilities in that they are collections (primarily electron microscopes) that are supported as part of the Materials Sciences Division research budget, i.e. these centers do not have "operating budgets" as do the synchrotron radiation light sources or the neutron facilities." It is this difference that resulted in the panel being asked to confine our study to materials science, including metals, oxides, ceramics, and polymers. EBMC techniques are also capable of making important contributions to the fields of condensed matter physics, solid state chemistry, physical chemistry, structural biology, and medicine, but the EBMCCs have generally not addressed these latter areas. The panel heard comments at each center for the need and value of EBMC techniques to be increasingly applied to biomaterials, which in some respects is consistent with their mission; and biomedical research, which is not. This report does not address these issues but it is clear that the centers will need guidance as to the extension of their research into newer areas.

The charge letters concerning the review are provided as Appendix A (Charge letter to BESAC from DOE) and Appendix B (Charge letter to Subpanel Chair from BESAC). Prior to the selection of the panel members, the Chair held a number of discussions with the Center Directors, and attended a meeting with them. The Directors proposed a two-stage process: first, a review of the concept of the EBMCCs, in which they would present a concerted summary of their role and contributions. Following this, they asked that the panel would visit each of the four centers, to understand the differences between them. This proposal was accepted; and in addition, the Directors were asked to identify a limited number of the major scientific areas to which they believed their centers contributed. The list they provided was:

- Interface Science
- Phase Transformations and Alloy Design
- Defects, Deformation and Radiation Effects
- Nanostructures
- Thin Film and Surface Science
- Microelectronic Materials

They noted that not all of the centers worked in all of these areas.

The members of the panel were selected as two groups: one group was of people concerned with the electron beam microcharacterization techniques and the underlying science; the other group was of people familiar with one or more of the identified materials science areas. Of course, several members of the panel would meet both of these criteria. In addition, it was hoped to compare these U.S. Centers with the international equivalents, and four members of the panel come from outside the U.S. A list of the panel members is provided in Appendix C.

In addition, each of the centers was asked to nominate a member to act as an associate to the panel. These associates attended all the sessions of the panel apart from those in which there were discussions of the individual centers, and accompanied the panel on the visits to all of the centers. The names of these associates are included in Appendix C. The panel met first on the evening of August 12th, 1999 to discuss the task, and Professor Geraldine Richmond, the Chair of BESAC and the author of the Charge Letter, attended and described what was expected of the panel. Previous BESAC panel reports on BES facilities and other topics were also available and of help in orienting the panel members to the BESAC requirements.

The meeting with the Center Directors took place on August 13, 1999, and the agenda is given in Appendix D. The meeting date had been selected to follow a BESAC meeting at which the panel chairman had reported on the panel membership and the plans for the review. All the members of BESAC were invited to attend this first meeting, and we also invited members of BES staff from the Germantown site. The EBMCC Directors provided the panel with a detailed report on their overall view of the centers, and some of their contributions organized in terms of the scientific areas identified above. This report, entitled "Electron-Beam Microcharacterization Centers: A National Resource" (72 pp, June 1999) is provided as Appendix F, and a shorter outline entitled "Contributions, Challenges, and Opportunities in the Core Scientific Fields of the Four Electron Beam Microcharacterization Centers" (11pp, August 1999) as Appendix G. They also provided us with a list of the publications from the centers for 1996 and 1997.

Dr. Manfred Rühle (MPI, Stuttgart), a member of the panel and the director of the strongest EBMC group in Europe talked on "The Contribution of Electron Beam Techniques to Science" and demonstrated the enormous role EBMC has played, and continues to play, in study of materials science. Following this, Dr. J. Murray Gibson (ANL) talked on the "Electron Beam Microcharacterization Facilities: Opportunities and Needs" and provided an overview of his view of where the BES EBMCCs fit into the overall picture and a look at future directions for the centers. Each of the EBMCC Directors then discussed different programs where the centers' emphasis is concentrated and used highlights of their own and other centers' work for illustration of the success of these programs. Dr. Michael O'Keefe outlined "The Materials Microcharacterization Collaboratory", an on-line capability for remote users of equipment and expertise located at the centers. The program links all of the BES EBMCCs and also the microscope facility at NIST. In the final talk "Outlook and Future Challenges" Dr. Uli Dahmen (LBNL) provided the panel with the Center Directors' views as to the prospects for

advancement in the major fields worked on by the centers, and the exciting future challenges and opportunities made possible by the recent breakthroughs in EBMC technology.

Among other items, it became clear that the four EBMCCs are preparing to propose a cooperative program to solve the important nanostructural challenges using new advances in electron beam microcharacterization.

Following the presentations, the panel held a further closed meeting to discuss the critical points that they would focus on during their site visits and provide them with some of the basis for the structure and content of this report.

In the following month, the panel discussed via e-mail what we had learnt, and the form of our eventual report. As a result of these discussions, the centers were sent a list of 15 questions, which were designed to determine the different character of each of them. All the centers sent us excellent clear responses. Neither the letter nor the center responses are attached to this report, but much of the materials in the responses is quoted in the different sections of the report.

The visits to the four centers took place in the week of December 5th, 1999. The week-long itinerary is provided as Appendix E. We first went to SHaRE, then to CMM, then to EMC, and finally to NCEM. At each site, the procedure was exactly the same. We began with a brief introduction from the center, essentially to tell us what we would see on our tour. Then we divided into three groups, each containing at least one EBMC expert, one materials scientist, one of the international members, and one of the associate members, not including the associate member from the center we were reviewing. Each group was then guided through the same tour, and we had ample time to discuss what we were seeing with the researchers at each location on the tour. Following the tour, we had lunch with users in the absence of center personnel, and asked them to comment frankly on their experience in working with the center, and to inform us of any improvements they would suggest. Following this, we had a closed discussion to review what we had seen and heard. We then called in the Director of the Center, together with anybody else he wished to have present, and the chair summarized our general opinions, noting that this was an immediate response, which might change as we had more time to consider. At each center, we asked to see a senior member of the Institution, to receive assurance that the Institution regarded the center as an important part of their capabilities.

As we traveled to the next site, each of the three tour groups prepared a written report on their observations, which was distributed to all the panel members. Following the last of these visits, we spent a day and a half discussing the information we had accumulated during the whole of the review. Panel members were assigned to write reports on the scientific contributions on each of the six identified technical areas. The report that follows begins with the general field of electron beam microcharacterization and the capabilities of the different techniques is presented. Following this, the four centers are described, listing the Vision and Mission of each center, the principal instruments available, the current staff, and the overall costs. The scientific contributions of the work

done at the centers are summarized, both in terms of the overall performance of the four centers, and the relative contributions of each of the individual centers. The next section discusses the users of each of the centers, and the teaching and training in the specialist EBMC techniques provided by the center staff for visitors.

Finally, the conclusions and recommendations of the panel are presented.

3.0 THE SCIENTIFIC AND TECHNOLOGICAL CASE FOR ELECTRON BEAM MICROCHARACTERIZATION

3.1 Introduction

For materials scientists and engineers, electron beam methods have become pre-eminent wherever microstructural characterization of materials is required. In this section we place these methods in the context of others, and review the basic concepts needed to understand EBMC techniques. As an aid for the reader an acronym list is provided in Appendix I together with a brief outline of some of the techniques.

Three long-lived particles are commonly used as probes to study materials - neutrons, X-rays and electrons. These have recently been joined by an array of scanning probe methods for the study of surfaces, and many novel pump-probe methods which explore the time domain. All are now used with the aim of understanding and predicting the properties of matter - electronic, magnetic, mechanical and thermal amongst others. They are also used to test the latest theories in materials science and condensed matter, from the quasi-particles of superconductivity and colossal magnetoresistance to the remarkably sharp transition from ductile to brittle behavior in engineering materials. A common theme for much of this work is the demand for improved spatial resolution, in order to study increasingly fine-grained materials and the nanostructures of the electronics industry.

These tools may be classified according to their interactions with matter, and by the experimental arrangements used in each case. The interaction may probe the ground state (as in X-ray crystallography or STM) or excited states, as in the various spectroscopies. A popular trend with all the probes is toward spatially-resolved spectroscopies. Most may also be classified according to a photon/electron in - photon/electron out classification. Another form of classification distinguishes bulk from surface probes. Finally, we have the distinction between the biosciences and materials science. For all of these probes there have been dramatic recent advances in technique, especially in the area of source brightness, controlled sample environments (including UHV, liquid, and high pressure), and detector efficiency - field-emission guns, for example have now come into general use in electron microscopy, with brightnesses about four orders of magnitude greater than an undulator/synchrotron combination operating at 500 eV.

The usefulness of these methods will depend on many factors, including scattering cross-section, source brightness, availability of lenses for imaging or probe-focussing, detector efficiency, spectroscopic resolution, spatial resolution, and radiation damage effects. Frequently, strongly scattering probes are associated with difficult data interpretation due to multiple scattering (e.g. photoelectron spectroscopy, TEM), while weak scattering, simpler to interpret, must compete with noise and background.

The various probes provide complementary information - as one example amongst many, the magnetic properties important for magnetic field detectors and computer memory may be studied using circular dichroism with X-rays, by magnetic superlattice diffraction by neutrons, or by electron holography in TEM using an electron beam. A striking example of this complementarity has also occurred with the high T_c materials. Here the atomic structure was determined chiefly using neutron, the flux pinning centers identified using electron beams, while X-ray spectroscopies were used to test the prevailing theories of superconductivity and to identify the mechanism.

This report is concerned with the electron-beam facilities of the U.S. Department of Energy. We take this to include most of the electron-in/anything-out techniques, however much of the emphasis will be on TEM, SEM, Auger, cathodoluminescence and related methods. Modern TEM, STEM and SEM instruments have become extremely versatile, being fitted, for example, with detectors for characteristic X-rays and most of the secondary emissions generated by a kilovolt electron beam. By comparison with the other probes, the existence of very high brightness sources (the brightest in all of physics) and the availability of lenses capable of forming probes with diameters down to one Ångstrom (or images of similar resolution) are the great strengths of electron beam methods. However it is only recently that accurate quantification of the data has become possible, since this required very fast computers to deal with the multiple scattering problem (in CBED, ELNES and HREM) and, equally importantly, accurate specification of experimental parameters (in HREM). Recent developments, which we discuss, include the commercial availability of imaging energy filters, field-emission guns and aberration correctors. Improved sample preparation methods (ion guns, tripod grinders, plasma cleaners) are making sample preparation simpler and reducing contamination. In this section we review the basic concepts needed to understand these techniques.

3.2 Techniques

3.2.1. Microdiffraction in TEM and STEM

Modern STEM instruments are capable of obtaining transmission diffraction patterns, using a sub-nanometer diameter electron probe, from samples a few hundred nanometers thick or less. It follows that sample preparation is tedious, and elastic relaxation of the bulk material during thinning may make interpretation difficult. But for the phase-identification of individual nanoscale particles, microphases, inclusions, polytypes, intergranular phases etc., no other technique is as powerful. Following EDX analysis, microdiffraction and selected area diffraction have become the method of choice for micro-phase identification, based on d-spacing comparisons with data in the powder diffraction file. (For one campus-based Industrial Affiliates program, this type of work

accounts for 70% of the time spent on TEMs). Microdiffraction (CBED) with field emission guns and the Koehler mode of illumination (for spot patterns) have greatly improved the ability to correlate image and diffraction information. The STEM imaging mode provides the ultimate capability in this regard, allowing diffraction patterns to be obtained from individual unit cells that have been identified in an image. With the increasing trend toward finer grained materials and composites, we expect increased demand for the capability for phase identification based on indexed diffraction patterns obtained from sub-nanometer regions. Intergranular phases may also be identified in this way.

Strains larger than 10^{-4} may also be mapped out with nanometer spatial resolution (e.g. around quantum dots, near grain boundaries in ceramic superconductors) by CBED. Finite-element analysis has been used to correct for relaxation, however this remains a difficulty. A recent development, which takes advantage of imaging energy filters, is the precise quantification of elastically filtered diffraction (CBED) data and comparison with accurate multiple-scattering calculations. This makes it possible to measure X-ray structure factors by TEM with an accuracy of better than 1%, sufficient to "see" bonding effects and test the many-electron approximations made in band-structure calculations (This is illustrated in the cover picture of the cloud of bonding electrons in copper oxide). Thus, electron microscope signals may now be quantified with the same level of accuracy as more mature techniques such as X-ray crystallography. New effects are possible using coherent nanoprobes (with diameters down to one Ångstrom) which have only just begun to be exploited, such as the determination of the atomic structure of anti-phase boundaries in alloys, and the use of atomic columns to focus the beam for super-resolution schemes.

3.2.2. Electron energy-loss spectroscopy (EELS) in STEM

The high brightness and small probe of the STEM form the basis for a powerful method of spectroscopy, in which the number of beam electrons loosing energy in a small interval while traversing a thin film are plotted as a function of energy loss. For very thin samples, the resulting spectrum is proportional to the imaginary part of the reciprocal of the dielectric function, and so has similar form to a soft X-ray absorption (XAS) spectrum. The most straightforward application of EELS is therefore light-element microanalysis, and the method remains competitive with (or more powerful than, depending on the materials system) "windowless" EDX for second-row elements such as nitrogen. The best field-emission STEM instruments are capable of providing a probe current of about 1 nA into a probe of diameter 1 nm. EELS covers the same energy range (0-2000 eV) as soft X-ray absorption spectroscopy, with slightly poorer energy resolution (about 0.22 eV at best, with a 0.2 nm probe, giving a few thousand counts per second at the silicon L edge). Parallel-detection EELS spectrometers have also greatly improved collection efficiency over X-ray methods, and count rates are measured in kHz per 0.2 nm pixel at light element inner-shell absorption edges. Whereas X-rays are either annihilated or left unaffected by an interaction, a fast electron may loose any amount of energy. The result is a larger background due to multiple energy loss events in EELS than in XAS. In thicker samples, plasmon satellites, for example, are likely to appear downstream of inner-shell edges, however methods exist for the removal of multiple scattering effects. The forward-scattering nature of inelastic electron scattering at these

energies makes detection efficiency very high, compared to X-ray detection. The spatial resolution of EELS is unrivalled. Very recently several groups have obtained EELS spectra from single columns of atoms, in samples about 10 nm thick.

In the STEM geometry, these spectra can be obtained in correlation with an atomic resolution image of the same region, resulting in an extremely powerful technique, as shown in Figure 1 for the technologically important silicon-silicon oxide interface, on which many semiconductor devices are based.

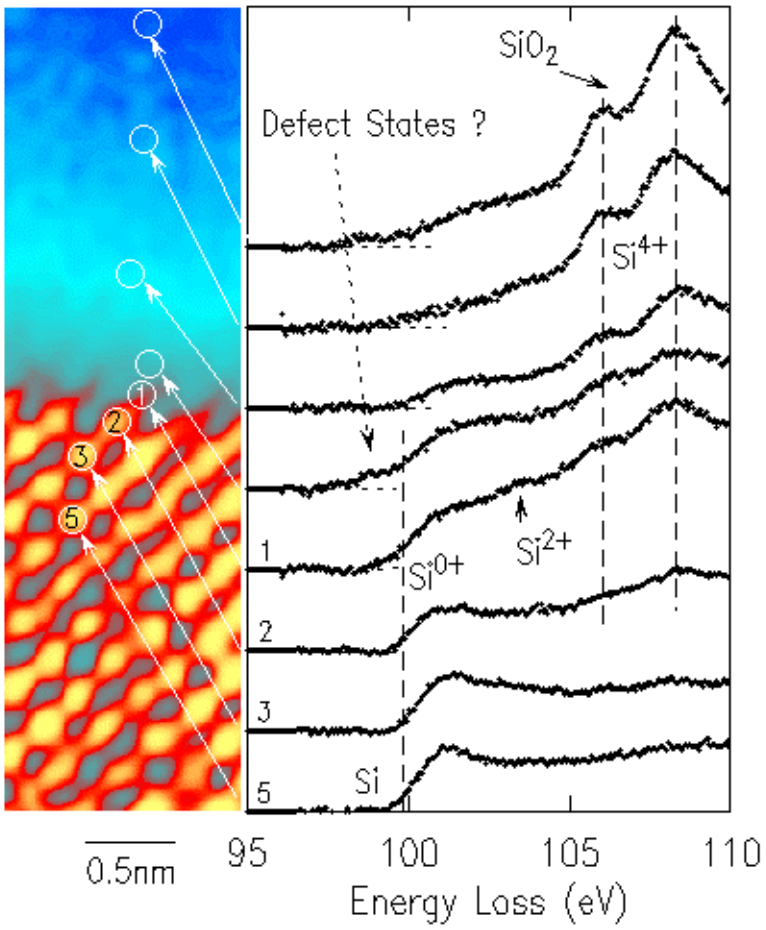

Figure 1. A STEM atomic-resolution image of a thin silicon crystal near the silicon-silicon dioxide interface also shown in Figure 3. Energy loss spectra of the silicon L edge have been obtained from individual columns of atoms as shown, revealing the gradual oxidation of the silicon. (Batson, Nature 366, p. 727, 1993, IBM).

As in XAS, the real and imaginary parts of the dielectric response function may be derived using Kramers-Kronig analysis. Both the near-edge structure and extended fine structure techniques of XAS have their parallel for EELS, and some fascinating channeling effects have been observed in EELS with close parallels to the standing-wave (SW) effects seen with neutrons and X-rays. These SW methods are almost unique in promising both high spatial and energy resolution, together with species identification. A modest literature is devoted to chemical fingerprinting (determination of oxidation state) from EELS near edge structure, with high spatial resolution. Dipole selection rules don't

always apply in EELS, since the momentum transfer is not negligible and may be controlled by choice of scattering angle, resulting in possible new information about elementary excitations in solids. The band-gap region of the spectrum has recently received much attention in EELS. In general, however, the total effort applied to the analysis of EELS spectra is miniscule compared to that devoted to XAS. Given the higher source brightness, stronger scattering cross section, unrivalled nanoscale spatial resolution and parallel detection capabilities of EELS, there seems to be no good scientific reason for this.

As one example of recent work illustrating the power of the method, EELS spectra of the Si L edge have been spatially mapped with a 2 Ångstrom electron probe across the 4 nm width of a field-effect transistor, as shown in figure 1. (The plane of the gate oxide layer contains the beam direction). The sample was about 10 nm thick. In this way the local chemistry of the oxidation process can be mapped at the crucial Si/SiO_2 interface. Similar "single atom column EELS" results have recently been obtained from Mn doped strontium titanate.

3.2.3. TEM, High Resolution Electron Microscopy, STEM and EDX

The first major discovery for materials science due to transmission electron microscopy was the direct observation of dislocations. The first major discovery, which resulted from atomic-resolution TEM, was an explanation, at the atomic level, for the causes of non-stoichiometry in complex oxides. The most recent major discovery was the observation and synthesis of graphite nanotubes, which resulted entirely from HREM observation. Routine dark and bright-field TEM imaging may now be considered a mature technique, however it remains an extremely popular method in great demand for characterizing defects such as interfaces, stacking faults, grain-boundaries and dislocations in modern materials. The availability of elastic imaging filters has greatly improved the quality of these bright and dark-field images, and allowed imaging through thicker samples for a given voltage.

HREM has become the method of choice for the study of defect structures in the bulk when atomic resolution is needed. These defects control the properties of most materials. Thus, mechanical properties are controlled by the defects responsible for stress concentration, electrical properties are influenced by defects which limit charge-carrier life-times in semiconductors, and first-order phase transitions are mediated by moving defects.

HREM images of atoms may be obtained either in the TEM geometry (where the sample is flooded with illumination, and, as for a camera, scanning is not used), or in the STEM arrangement, where a fine probe is scanned over the sample and the transmitted electron signal detected. This second arrangement has many advantages, since it facilitates collection of analytical signals, however the first method provides a much larger field of view at atomic resolution. Under appropriate experimental conditions (specified by Otto Scherzer in 1949), the bright-field images provide a map of the electrostatic potential in a thin slab of crystalline material, projected in the direction of the beam. For this simple interpretation, the sample thickness must be less than about 10 nm, so that multiple

scattering can be neglected. This projected potential is convoluted by the impulse response of the electron microscope. (The impulse response is the image of a point, a peaked function that limits resolution). The columns of atoms in the sample, about 10nm long, are aligned with the beam. The width of the impulse response depends on electronic stability, mechanical stability, thermal stability, the aberrations of the objective lens, the focus setting, the source size and spatial coherence conditions, and the energy-spread of the electron source. Other factors, such as contamination and radiation damage may also be important. Plasma cleaners and improved oil-free vacuum systems have done much to reduce contamination. Aberration-correctors have recently been demonstrated.

For thicker samples, multiple scattering becomes important, and the above ideas, based on coherent linear imaging theory taken over from optics (or incoherent optical imaging theory for STEM), can no longer be used. Resolution can no longer be simply defined, since it becomes a property of both sample and microscope. Nevertheless, if the experimental parameters are sufficiently accurately known (including precise specification of sample orientation and thickness) the dynamical image may be calculated, and atom positions adjusted for best fit with the experimental lattice image. Based on prior experience in matching CBED diffraction patterns, the quantitative matching of images has now become an active field of research. However no consensus has emerged on the best Goodness of Fit index, to use or how best to incorporate a-priori information in a Baysian analysis, and the global-minimum search problem remains fundamental. More powerful computers are helping, but scaling laws are discouraging. The use of additional information (bond lengths, EDX data, CBED patterns etc.) will almost certainly be required to solve structures a-priori from lattice images.

The first such "lattice image", taken in the late fifties by Menter at 1.2nm resolution, was an interferogram which told us more about the instrument than the sample. The turning point came in the early seventies, when Albert Crewe in Chicago observed individual heavy atoms in the field-emission STEM instrument he had invented (and then applied to biology), and when Iijima and Cowley soon after showed that useful structural information could be extracted from images of complex oxides at 0.38 nm resolution.

It is now almost a decade since the important milestone of one-Ångstrom resolution was attained in transmission electron microscopy. This has allowed individual columns of atoms to be seen in projection for entirely new classes of materials, as shown, for example in Figure 2. This shows an atomic-resolution TEM image of a nanoscale diamond crystal, growing inside a "buckyball" onion, or Fullerene, consisting of shells of carbon atoms. This true atomic-resolution capability, combined with imaging energy filters, field emission electron sources, CCD camera detectors and fast computers for image analysis have transformed the field of HREM over the last decade. The very recent development of aberration correctors promises further exciting advances, both for HREM and the PEEM (Photoemission Electron Miroscope) instruments designed for synchrotrons. Fast computers have allowed rapid image simulation based on multiple-scattering solutions of the one-electron Schroedinger equation involving thousands of interacting Bragg beams, together with the incorporation of lens aberration effects.

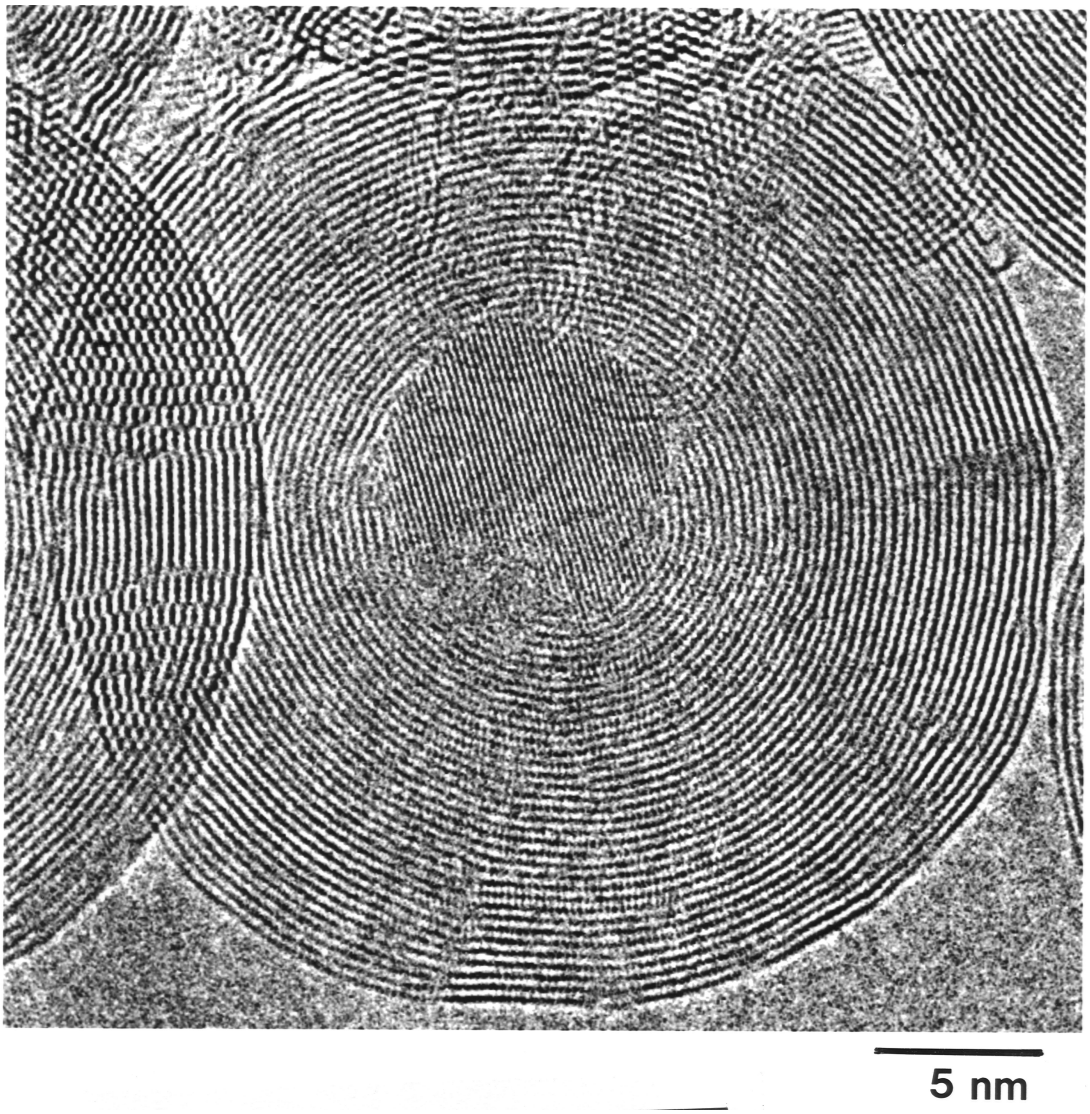

Figure 2. Spherical carbon "onion" containing a nano-crystal of diamond. Atomic resolution images such as these can reveal how the carbon shell acts as a pressure-vessel to allow the nucleation and growth of diamond. (Banhart and Ajayan, Nature 382, p. 433, 1996).

The most important finding from all this work is that real materials are far more imperfect on the atomic scale than the broad-beam spectroscopies and diffraction methods would suggest. These atomic-scale imperfections which control the properties of materials include point, line and planar defects. HREM has thus proven invaluable in revealing the host of microphases and polytypes present in minerals, the detailed structure of quasicrystals and fullerenes, the atomic processes involved in first-order phase transitions, the atomic structure of interfaces in both structural and electronic materials, the microstructure of magnetic ceramics, the core structure of dislocations, and grain boundary structures in superconductors, ceramics and alloys. The mechanism of phase-transformation toughening in ceramics was elucidated by HREM and other methods.

In short, HREM has become the technique of choice wherever microscale characterization of defects in modern materials is required. A large fraction of the literature is concerned with the determination of interface abruptness and structure at the atomic level - the study of intergranular phases in fine-grained sintered material and metal-ceramic interfaces has been an important application. (By altering interfacial energies, a thin lamella only a few nanometers thick may dramatically alter mechanical properties in composite materials). With some electronic device dimensions approaching a few nanometers (e.g. gate oxide widths), HREM has now become the only method capable of detecting structural imperfections in the millions of field-effect transistors which make up a modern computer.

Figure 3 shows such a transistor – the latest devices are considerably smaller. In this HREM image it is possible to count the number of atomic columns across the gate oxide width. Line defects have also been analyzed - by using these images to exclude and suggest models for atomic structures used in ab-initio quantum molecular dynamics simulations, the detailed mechanisms of dislocation kink motion have been suggested and energy barriers determined. Thus the atomistic basis of ductility may be understood in simple crystals. Similarly, the observation of the three-fold dissociation of screw dislocations in bcc metals has confirmed theories of the high flow stress of these metals at low temperatures. Less success has been obtained in determining the structure of point defects by HREM, and this, together with the determination of the structure of glassy materials, remains one of the great remaining challenges for the field.

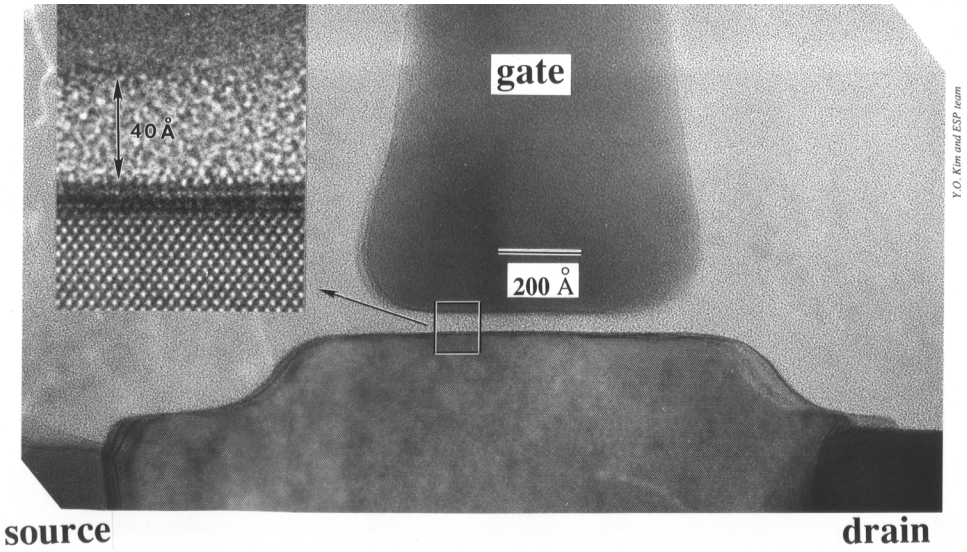

Figure 3. Atomic-resolution HREM image of a Ghz FET transistor, similar to those in modern computers. The inset shows detail at the atomic scale of the crucial solicon-silicon oxide interface, and the insulating gate oxide, about 4 nm thick. (Kim and Ourmazd, Lucent)

In addition to the study of microphases, planar and line defects, a final important class of HREM experimentation has consisted of in-situ studies of materials under controlled environments. The transformation between oxidation states of complex oxides for example can be directly observed in movies recorded at atomic resolution using an environmental cell. The surface oxidation of silicon has been observed at high resolution in an ultra-high vacuum microscope, giving an understanding of the atomic mechanisms involved in the early stages of crystal growth. The motion of phase boundaries in nickel silicide can been observed using real-time, atomic resolution movies, and quantum wires of gold just a few atoms in diameter have been observed at atomic resolution and their size correlated with quantized changes in resistance. The process of MOCVD has been imaged in a controlled atmosphere TEM.

The discovery of nanotubes by direct observation in HREM amounts to the first potentially useful commercial material to be discovered by HREM alone. The electronic properties of these nanotubes hold great promise for nano-electronic devices.

The STEM mode operates in similar fashion to the SEM, however electrons transmitted through a thin film are detected. The theory has been worked out in detail, and is related to that of TEM by the principle of reciprocity in Optics. If a STEM annular detector is used (equivalent, by reciprocity, to the use of annular illumination in TEM), the arrangement is conveniently combined with an energy-loss spectrometer, to collect the small angle inelastic scattering which passes through the central hole in the detector to the spectrometer. In this way EELS spectra of the inner-shell edges may be obtained from regions as small as a single column of atoms, in correlation with the dark-field or "Z-contrast" image produced by the elastically scattered electrons detected at larger angles by the annular detector. These Z-contrast images have demonstrated one-Ångstrom resolution and, although scattering is weak, increased sensitivity to atomic number is obtained and image interpretation may be simpler than for TEM, since incoherent imaging theory becomes a useful approximation. Then the near-edge and extended edge structures can be obtained with good statistics, providing crucial local chemical information from nanometer sized regions. In the thinnest samples, where multiple inelastic scattering of the beam electron can be neglected, these may be analyzed with all the power of the existing XAS theoretical tools.

This STEM arrangement also allows for the detection of characteristic X-rays, so that chemical maps of X-ray emission with high spatial resolution may also be obtained (EDX). Recently, using a STEM instrument optimized for EDX, an X-ray emission image of Cu segregation at grain boundaries in Al-4wt%Cu was obtained with a spatial resolution of 4 nm (foil thickness 100 nm). Typical count times are 5 seconds per 1 nm pixel for an EDX line scan. New X-ray detectors (Ge, superconducting bolometers) promise much improved performance, and new designs for wave-length dispersive X-ray spectrometers, with their very high energy resolution but serial data acquisition method, occasionally appear. Much more could be done to optimize STEM instruments for this mode.

In general the STEM geometry allows for flexible detection of all the decay products of secondary excitations, so that valuable spectroscopic and chemical information can be combined with atomic resolution imaging. Several brief examples follow: By collecting the visible light emitted by dislocations in diamond samples at liquid helium temperatures in a STEM, it has been possible to obtain emission spectra with high energy resolution from individual line defects in correlation with STEM images. EBIC (electron-beam induced conductivity) images have also been obtained in STEM mode. In the one modification of the VG STEM, a highly efficient secondary and Auger electron detector was fitted, allowing Auger images of Ag clusters to be obtained in a UHV environment with 4 nm spatial resolution. Operated as an SEM, images could also be obtained showing single-atom high steps from silicon surfaces. The spectacular results possible using "single-atomic-column" EELS spectra have already been mentioned, as has the X-ray mapping capability. This capability for combining the highest possible spatial and energy resolution from bulk defects is unique to the STEM. Its only disadvantage relative to TEM mode is the limited number of image pixels normally acquired, which limits field of view at atomic resolution.

Users of advanced TEM techniques demand an instrument in which rapid mode changes can be made between the diffraction/analytical mode and the imaging mode, without optical realignment, in a way which permits the analytical signal to be related to the real-space image. STEM provides this capability very naturally, and rapid switching between, say, a 1 nm probe for analysis and a 0.15 nm probe for imaging is desirable.

The use of aberration correctors for STEM instruments is a particularly exciting development. It has been estimated that a pole-piece gap of about 2 cm can then be permitted with a probe size of less than 0.2 nm. This would then allow a much increased solid angle of collection for EDX spectra (currently about 0.3 sterad at best), providing EDX maps of chemical composition with better spatial resolution and improved detection sensitivity. The larger gap will also permit larger sample tilt angles and new applications of improved environmental cells. Finally, the use of an aberration corrector in STEM will lead to valuable increases in probe current.

3.2.4. Electron holography.

In conventional TEM-based electron holography, an electrostatic biprism is used to split the coherent beam from a field-emission gun. Part of this beam passes through a thin sample, and part around it, to be recombined at the detector. In practice, the image is magnified before being crossed with the reference wave to form carrier fringes, thus easing the requirements on mechanical stability. (Fringes about three times finer than the finest detail to be reconstructed image are required). Holography (and Lorentz microscopy) has proven an indispensable tool for imaging superconducting vortex interactions and their dynamics. The phase shift due to a single quantum of flux can be detected. (A small region only, however, of the phase diagram may be studied. At high densities the vortices overlap and the weak phase contrast disappears). More recently there have been promising studies of ferroelectric domains by electron holography, useful for the study of memory elements and domain switching. The quantification of this data is difficult since sample thickness must be accurately known. Many measurements of the mean inner Coulomb potential in crystals have appeared - this quantity depends sensitively on crystal bonding and the susceptibility. However the largest field of application of TEM-based electron holography is likely to be in the study of magnetic thin films and multilayers. In recent work, magnetization as small as 1500 Bohr (i.e. 500 Co atom moments) has been measured by TEM holography, corresponding to a flux of about 10^{-5} quanta. Using special pole-pieces and temperature controlled stages, movies showing magnetic phase transitions and domain motion may be obtained. With the continued growth of thin-film magnetic storage media and devices, this area of EBMC is likely to see considerable growth and offers great opportunities.

3.2.5. UHV TEM

Since the structure of the Si (7X7) (111) structure was solved by UHV TEM (not by STM) fifteen years ago, this technique has grown slowly in the hands of a few specialists around the world, including groups at Northwestern and the University of Illinois, Urbana-Champagne. These groups have made important contributions to surface science, both in solving surface reconstructions and their interactions, and in understanding the oxidation of metals and semiconductors, and sintering. Leadership in this field has

traditionally come from Japanese researchers, but we see a strong and exciting future for in-situ UHV work on nanoscale structures, particularly dynamic experiments with fast image recording at reasonably high resolution. Spectacular recent successes from overseas have included direct observation at atomic resolution of the bonding process under STM control in a TEM, and the correlation of quantized resistance changes in nanowires with atomic structure. The panel considers the incorporation of a UHV stage and bakeable environment into a commercial aberration-corrected TEM or STEM to be a worthwhile but very difficult and ambitious project from which a large scientific payoff can be expected.

3.2.6. Environmental and in-situ TEM.

Controlled-atmosphere cells now allow near atomic-resolution imaging at pressures up to about a third of an atmosphere pressure. Atomic-resolution images may thus be recorded under conditions of known temperature and pressure in the presence of known reactant gases. (For example, images have recently been obtained at 1 mbar pressure and 700^{o} C, showing near-atomic resolution). The resulting HREM images of catalysts and oxides show directly the effects of oxidation on atomic structure, as, for example, point, line and planar defects are generated to accommodate changes in stoichiometry. These atomic processes may then be correlated with activation energies derived from Arrhenius plots. In-situ dynamic studies of inorganic chemical reactions with gases offer a large scientific payoff now that new designs of cells have greatly reduced the compromise in microscope performance, which must be accepted, especially in the field of catalysis. A variety of other in-situ holders have been developed, including straining stages and heating and cooling stages. Temperatures down to about 15 K are readily obtainable. Many improvements in cell design will be possible when aberration correctors allow larger pole-piece gaps.

3.2.7. SEM.

The Scanning Electron Microscope (SEM) is by far the most popular EBMC instrument, providing topographic contrast from bulk material at resolutions down to about one nanometer. X-ray spectrometers are commonly fitted, in addition to cathodoluminescence (CL) apparatus, electron-beam induced conductivity detectors (EBIC) and temperature controlled stages. In addition to routine use there have been several recent important developments:

i). Field emission guns and immersion lenses. These devices have improved resolution to the level of a few nanometers for routine work, and below one nanometer in special cases. Sufficient signal is now available to obtain atomic-number contrast from semiconductor multilayers at high resolution.

ii). Environmental SEM. The environmental SEM uses a gas ionization cascade in the sample chamber to amplify the secondary electron signal from the sample, which can thus be held at a controlled pressure. It has opened up an entirely new world of observations, allowing, from the observation of cement drying to biological samples in a hydrated environment. The need for conductive coatings is avoided, and heating and straining stages may be used.

iii). Back-scattered Kikuchi patterns. Electron backscattered diffraction patterns (EBSD) may be obtained from SEM instruments in which a large area detector is arranged to collect the backscattered electrons (those which loose little energy) and to display the resulting Kikuchi pattern. This has proven invaluable for texture analysis of polycrystalline bulk materials, spanning the length scale between X-ray methods and TEM microdiffraction. Multi-phase materials may also be analyzed, and phase identification facilitated by combining EBSD with EDX, thereby avoiding the need for tedious TEM sample thinning. (Unit cell volume, interplanar spacings, X-ray emission lines and data from the Powder Diffraction File are usually sufficient to identify sub-micron phases). Local crystallography may be mapped around crack trajectories, for example. Images formed from portions of these patterns reveal the populations of grains in particular orientations (orientational imaging, or OIM). Methods for mapping strains by this technique are under development, and automated indexing software has been developed. At 10 kV, a resolution of about 40nm is predicted at best, using a field-emission SEM.

iv). Channeling contrast from sub-surface defects in SEM. The field-emission SEM was first used to image sub-surface line defects many years ago - a renaissance of this field of research has recently occurred and represents an important opportunity for materials science.

v). UHV SEM. The UHV, field-emission SEM has become an invaluable research tool for the study of crystal growth in semiconductor research. Recently single-atom high steps have been resolved with this instrument, which is conveniently combined with many other detectors.

vi). Energy filtering SEM. Filtering may be applied to the emitted secondary and back-scattered electrons for greatly improved contrast and depth resolution - this old idea is also undergoing a renaissance, for example, in the semiconductor industry, where it is used to distinguish layers at different depths.

vii) Low voltage SEM. Considerable opportunities exist for obtaining higher resolution, higher contrast, possibly reduced damage, and reduced charging effects by operating SEMs at lower voltages, where the ionization volume is reduced. The use of this mode in combination with new types of X-ray detectors (e.g. bolometers) is particularly attractive at energies in the range 3 - 5 kV.

3.2.8. APFIM

Atom Probe Field-Ion Microscopy (APFIM) permits the atomic-level characterization of both microstructure and microchemistry of materials. The Atom Probe consists of a field-ion microscope (FIM) coupled with a time-of-flight mass spectrometer. The FIM is basically an ultra-high vacuum projection microscope. The specimen consists of a fine needle, with a nominal tip radius of 50 nm. The material for analysis must be electrically conductive. The needle specimen is then inserted into the Atom Probe and is cooled to cryogenic temperatures (typically 50K). A small amount of inert gas (typically

Ne) is admitted into the FIM, and a positive voltage is applied to the specimen. As the voltage is increased, the gas atoms are attracted to the tip and field-ionization of the gas atoms occurs above the atomic ledges at the tip. The gas ions are then projected radially away from the tip towards the electron channel plate-phosphor imaging screen assembly where the field-ion image is formed.

As the tip voltage is increased further, the surface atoms in the ledge positions become ionized and follow a similar trajectory to the imaging assembly. In conventional atom probes, a small hole is located in the center of the imaging assembly (electron channel plate-phosphor imaging screen). This hole serves as the entrance aperture to the time-of-flight mass spectrometer. A single ion detector is located at the end of the flight path. The atom probe also requires a high-speed timing system for measurement of the flight times of the ions as they leave the tip and strike the detector. The microchemical analysis is performed by computer-controlled pulsed field-evaporation coupled with the measurement of the flight time. The evaporation voltage and the flight time of each ion reaching the single ion detector are used to calculate the mass-to-charge ration (m/c) of each ion. The m/c for each analyzed ion is then stored in the computer in the sequence of arrival at the detector. The depth resolution for atom probe analysis is an atomic layer, but the lateral resolution for the conventional atom probe can be on the order of nanometers because it is related to the probe aperture. Atom probe analysis requires careful experimental procedure because a variety of factors including voltage pulse fraction, specimen temperature and background pressure in the system can markedly affect the quality and content of the data.

A major breakthrough in the APFIM technique was achieved by replacing the single-ion detector with a position-sensitive detector. This development has vastly improved the amount of data that can be collected from a given specimen. The use of the position sensitive detectors has enabled a significant improvement in the lateral resolution for analysis (< 0.5 nm) while maintaining the single atom layer depth resolution. The m/c data coupled with the positions of the ions at the detector is stored for subsequent computer reanalysis. An example from the work of Deconihout shows a graphical representation of an analysis along a [001] pole in a γ' (Ni_3Al) precipitate in a Ni-base superalloy in which the Al-rich and Ni-rich planes are clearly visible in this true "lattice image" of the precipitate. This technique is now most appropriately termed "3D-AP" to reflect the tomographic capability. The optical position-sensitive atom probe (OPoSAP) and tomographic atom probe (TAP) are currently commercially available from Kindbrisk (UK) and Cameca (FR), respectively. The application of the 3D-AP to a wide variety of materials problems, such as radiation embrittlement, segregation and precipitation phenomena has met great success to date. This instrument is likely to become an important research and characterization tool.

4.0 DESCRIPTIONS OF THE ELECTRON BEAM MICROCHARACTERIZATION CENTERS

The following section is arranged in the following way. First: the Mission and Visions of the center are stated. Each center was asked to give us their view of their role in the OBES context, and were invited to present this as a short Mission statement, and then a somewhat longer Vision statement, indicating their thoughts, foresight, and opportunities for the future. Each center approached this in a slightly different way, and the statements which lead off the descriptions of the each of the centers are summaries of the main points they made. Second: the Major Equipment. This lists the major items of electron beam microcharacterization equipment in each center, and shows when it was purchased and (in most cases) what the expected remaining lifetime is. Again, this material is that provided by the centers. In addition, this section indicates the center's plans for major equipment upgrades. There is a more extensive equipment list in Appendix H. Third, the Staffing. Fourth, the funding for each center. There is some ambiguity in these sections, because in some cases the funding may come from different sources, and certainly for earlier years prior to the centers being named as such, the funding, particularly for the large capital items, came from several sources.

In one of the presentations made to us, it was remarked that a new instrument, provided it is well-maintained, and that appropriate upgrades are made as they become available, can continue to be regarded as state-of-the-art or close to it for about ten years, and then can be usable for routine studies and instruction for a further few years. It will be seen that several of the major instruments in the centers are well over ten years old, and it is clear, therefore, that even if it has been possible for the centers to perform the appropriate maintenance and upgrades, more than half of the fleet is no longer at the forefront.

In reading the information on equipment, it is worth distinguishing three categories. A 'leading edge' instrument is one that is qualitatively different from its predecessors, capable of doing significantly new science. Such instruments during the first five years or so of their lives are generally run by a research scientist or a small team of scientists, whose major concern is developing the capability of the new instrument and exploring the new science to which it gives access. During this period, such an instrument is not usually regarded as appropriate for a 'user', in the sense that that word relates to the clients of user centers. The instruments which then appear, building on the new technology revealed by the leading edge instruments are 'state-of-the-art' instruments. These are the really important group of instruments that one looks for in a user center, since with the guidance of the expert staff in a center they can provide valuable information to the users. Eventually, as indicated above, an instrument ceases to be state-of-the-art, but it still can provide useful service within a user center, allowing preliminary examinations of specimens of interest, and serving for teaching and training purposes. These are often called 'core' instruments.

Now, in the case of electron beam microcharacterization instruments, this classification is oversimplified. This is because upgrades become available, which can be added to existing instruments, and in some cases may even turn a state-of-the-art instrument into a leading edge instrument. The costs of these upgrades, while not trivial, are very much

less than the cost of a new instrument. For example, consider the development of the following techniques, which were "leading edge" at one time: EDS, EELS, PEELS, Omega filters, GIFF (imaging energy filters), cathodoluminescence in STEM at helium temperatures, ALCHEMI, the CCD camera for TEM, video recording, Field-Emission Guns (FEG), nanodiffraction using a FEG, Z-contrast detectors, LaB_6 sources, etc. None of these involved the purchase of completely new and very expensive "leading edge" instruments. All were new detectors, sources or attachments to existing machines, and together they have made the biggest difference to transmission electron microscopy over the past two decades. Only the pursuit of higher resolution (which is important) has involved completely new instruments, and the development of the VG STEM, which was a quantum leap. The point is that there is an urgent need for instrument development money, since history shows that this is where most of the big scientific payoffs have come!

A list of the instruments and the multitude of microscopies and spectroscopies they support is of course by itself insufficient to allow a potential user to recognize the range of capabilities that are accessible at the centers. However additional information can be obtained by accessing each of the centers' excellent web pages (the addresses are given below each of the next four sub-sections). Information on the staff at each center is listed. These web pages are all linked with each other, with the BES home page, with the Materials Microscope Collaboratory, and with a number of other informative web pages. Collectively they represent a considerable effort by the staff of the centers to alert materials scientists to the value of EBMC and the ready access that they may have to the equipment. The center web pages make it clear not simply that the equipment is available to users but also that there is considerable expertise necessary in sample preparation, equipment manipulation, data analysis and simulation also available. This effort relates to the centers' objective of expanding their user base. The panel highly recommends these web pages as an introduction to the world of electron beam microcharacterization, and to the support available at the BES centers.

4.1 Shared Research Equipment Program (SHaRE)
Oak Ridge National Laboratory
Oak Ridge, Tennessee

4.1.1 Mission and Vision

The Shared Research Equipment (SHaRE) User Facility at Oak Ridge National Laboratory sees its contribution as providing, developing and maintaining state-of-the art instrumentation for the microscopy and microanalysis of materials, while developing, refining and applying a diverse array of microanalytical techniques for collaborative materials research and development efforts that are central to the mission of DOE. In particular, they aim to provide a resource of expertise and instrumentation for the materials research and development community to address problems of major scientific and technological impact through quantitative microscopy and microanalysis of materials at length scales from one micrometer down to atomic level. Instrumentation and technique development will continue to focus on three areas of established core competency: analytical electron microscopy, atom probe field ion microscopy, and

mechanical properties microanalysis. Within the context of OBES, the center believes that this suite of capabilities provides a powerful resource for ORNL, regional, national, and international researchers. In addition the SHaRe user facility provides a unique resource for the microcharacterization of radioactive specimens on a routine basis.

The electron microscopy effort in the Metals & Ceramics Division at ORNL was originally focussed towards characterizing irradiated alloys, especially the development of low-swelling high-strength austenitic stainless steels. With its evolution into the SHaRE User Facility, the scope of projects has broadened, and the facility is available for a diverse array of projects that make good use of the facilities and expertise. However, the projects are still largely aligned with the Metals & Ceramics Division, with its emphasis on structural materials.

4.1.2 Major Equipment

The oldest instrument in the current fleet is a Philips CM12 120kV AEM which was installed in 1987. It is regarded as standard core equipment, and will be replaced within six months; a replacement Philips Tecnai 20 Twin-lens 200kV microscope is on order. A Philips CM30 300kV AEM was installed in 1988. Addition of a Gatan 678 Imaging Filter (GIF) revitalized the use of this instrument, and it could have some 5 years more life; but demand may fall off within two years because of the installation of GIF on a newer microscope last year.

In 1994 a Philips CM200FEG 200kV AEM which has STEM capability was installed.

The principal thrust of SHaRE's work has been concerned with the mapping of compositional details, and the larger part of their equipment has this as a major thrust. In 1992, a Hitachi S4100 FEG SEM was installed, and in 1994 a Philips XL30FEG SEM was added. Both of these instruments are expected to have at least five more years life.

In 1985 a VG FIM 100 energy-compensated APFIM with ORNL-designed electronics and software was installed; there was a major voltage pulsing upgrade in 1997. This is one of only two such instruments in the U.S., and has the highest mass resolution of any atom probe. With a new cryostat and specimen stage, it is expected that this instrument will have a further 5 – 10 years life. In 1997 a Kindbrisk ECOPoSAP energy-compensated position-sensitive atom probe was installed. This is one of two in the U.S., one of only three ECOPoSAPs in the world. With appropriate upgrades, it has probably ten years remaining life. There are two other atom probes which are not available to outside users.

This lists only major instrumentation. There are some other workhorse machines – for example, a Tecnai 20 microscope.

For the future, SHaRE would like to add an advanced analytical electron microscope: it is possible that the NTEAM proposal could enable the development of this instrument. A low-voltage electron probe microanalyzer would also be very useful: they comment that there has been little development in EPMAs over the last two or three decades, in spite of

considerable developments in electron sources, electron optical design, and light-element X-ray spectrometers. They believe that a major breakthrough is possible. Finally, a Scanning Atom Probe/Local Electrode Atom Probe (SAP/LEAP) would allow new approaches to APFIM of thin films, such as are important for microelectronic and magnetic storage industries; and also offer major advantages for traditionally problematic metals with low bulk conductivities.

4.1.3 Staffing

The staff of the center consists of five high-level specialists: Dr. Ian M. Anderson (AEM), Dr. James Bentley (AEM), Dr. Edward A. Kenik (AEM), and Dr. Michael K. Miller (APFIM), all of ORNL; and Dr. George M.Pharr (MPM), of UT; a Program microscopist/administrator, Dr. Neal D. Evans (AEM/all), of ORISE; three technical support staff, Mr. J. Wade Jones (AEM), Ms. Kaye F. Russell (APFIM) and Mr. Victor W. Pardue (AEM), all of ORNL, one postdoctorate, Dr. J. Gregory Swadener (MPM) of UT, one graduate student, Mr. N. Ryan Williams (MPM) of UT, and an Administrative support, Ms. Renetta D. Godfrey of ORNL. Until two years ago, the facility also supported a full-time professional staff member concerned with maintaining the electron microscopes; he retired and was subsequently available part-time under a subcontract. However, he has now fully retired and with current budget constraints, it will be difficult to replace this position.

Service contracts are maintained on all of the electron microscopes and X-Ray detectors. These service contracts amount to ~0.5 FTE. The s4100 SEM is located in a separate building, and its service contract and staffing support are maintained by funds outside the facility budget. For the atom probe effort, there are no service contracts except for hardware and software maintenance agreements on the Silicon Graphics workstations.

The center comment that the planning of staffing levels is complicated by an ever-shrinking budget, which ends to make it difficult to replace even staff who leave or retire. There is definite need for support staff, who can prepare specimens and maintain the microscopes. This year, a technician was hired to work with the senior specimen preparation expert, to safeguard this expertise. However, since these technicians support many tasks within the Division and through the laboratory for TEM and other specialized specimen preparation, these positions do not pose a large cost to the operation of the facility.

There is no doubt that this lack of a significant support in advanced specimen preparation capability is a cause of concern: the panel regards specimen preparation as a most important component of an EBMC user center.

4.1.4 Costs

SHaRE activities are funded from 2 sources. The ORNL field work proposal (FWP) covers facility upkeep, maintenance, development, and (some) research activities of facility staff and outside users. The ORISE FWP covers Neal Evans 100%FTE, administrative support at ORISE ~10%FTE, travel costs for users of ~$50k/y.

Budgets for the last 3 years average $1750k/y from the ORNL Microscopy and Microanalysis FWP, and $240k/y from ORISE SHaRE Program FWP. It is worth commenting that the support from both these sources has been declining over the last three years.

For the last four years, the budget breakdowns have been:

FY	1996	1997	1998	1999
Maintenance	**171** (+70)	**320** (+65)	**345** (+35)	**232**
Technical Support	**321** (+90)	**353** (+90)	**145**	**199**
User Support	**1724**	**1389**	**1409**	**1297**
Facility Administration			**120**	**220**
Total operating	**2216** (+160)	**2062** (+155)	**2019** (+35)	**1948**

Note: the numbers in parentheses are additional funds from overhead cost centers.

Capital funds. Prior to SFI, capital funds were allocated from the ORNL BES Metal and Ceramic Sciences Program total of ~$500k/y. Typically, the SHaRE facilities received more than 50% of the total available. Capital funds were also obtained from other programs, such as Fusion materials, but at a steeply decreasing rate. With SFI, $250 k/y was allocated to SHaRE. When SFI funds were incorporated into the base program, allocations returned to the former system. Special allocations from DMS have enabled recent major purchases; in 1993 ~$800k for a 200kV FEG AEM; in 1996 ~$400K for a 3D atom probe; in 1999 ~$400k for a 200kV workhorse TEM.

Users do not pay any costs for use of the facilities.

SHaRE has had very few requests for proprietary work. The necessary procedures are in place and used extensively at other user facilities at ORNL.

Web page: http://www.ornl.gov/share

4.2 Center for Microanalysis of Materials
University of Illinois at Urbana-Champaign
Urbana, Illinois

4.2.1 Mission and Vision

The Center for the Microanalysis of Materials in the Frederick Seitz Memorial Research Laboratory at the University of Illinois at Urbana-Champaign regards its principal goals as:

- To contribute to the excellence of, as well as to enable, material science research at the Frederick Seitz Materials Research Laboratory with a focus on the mission of the DOE/OBES/DMS program.

- To develop the science of microstructural and microchemical analyses.
- To assist researchers within the community of materials science scholars at UIUC in applying modern microstructural and microanalytic techniques in their research.
- To educate graduate students and research associates in the use of modern microstructural and microanalytic techniques.
- To make modern microstructural and microchemical characterization tools available to the broader scientific community in the U.S.

The center regards itself as a major repository of instrumentation and expertise focused on the microcharacterization of materials, with a staff teaching the use of the instruments, assisting in the interpretation of experimental results, developing new instruments and techniques, and carrying out research in the area of instrument science. The housing of the center in the FS-MRL leads to close interactions with the FS-MRL/UIUC staff.

4.2.2 Major Equipment

The CMM is an integral part of the Frederick Seitz Materials Research Laboratory, and according to the center, each of the instruments was purchased and is maintained in response to a significant, demonstrated need by DOE research programs in the FS-MRL.

The oldest microscope in the current fleet is a Philips 120kV TEM, which was installed in 1982; this will be replaced this year. A JEOL 4000EX 400kV TEM was installed in 1986, and is regarded by the center as one of their 'unique and specialized' microscopes; it is modified for environmental cell operation. A special cell and double differential pumping allows operation with a gas pressure as high as 100 Torr in the vicinity of the specimen. This microscope is expected to be functional until 2005. A Philips CM-12 120kV TEM was installed in 1990, and will operate until 2004, and a Hitachi H-9000 300kV High Resolution TEM was installed in 1991 and will operate until 2005.

A VG HB501 100kV UHV-STEM with FEG, EDX, parallel EELS, was purchased in 1993 and will be operational until 2006.

Two new microscopes will be installed this year, and will be in service until 2015. These are a JEOL FASTEM 2010F, and a JEOL 2010 which is the replacement for the old Philips.

In addition, they have three scanning electron microscopes: a Hitachi S800 FEG/SEM installed in 1987; a Zeiss 960 SEM with a LaB_6 electron gun installed in 1990, and a Hitachi S4700 low-voltage high-resolution FEG SEM installed in 1998. The Hitachi will be retired this year.

Another 'unique and specialized' microscope is a low-energy electron microscope (LEEM) to a R. Tromp design with a number of capabilities, designed to incorporate Molecular Beam Epitaxy (MBE) capabilities.

The center also supports two other microscopes: a JEOL 2000EX TEM "SHEBA" extensively modified to provide UHV conditions and sample access, again with MBE and CVD (chemical vapor deposition) sources for *in situ* surface modification studies; and an extensively modified JEOL 200CX UHV TEM allowing *in situ* sputter deposition of nanoparticles onto clean surfaces. These also are regarded as 'unique and specialized, and are not really part of the user center.

There are several scanning tunneling and atomic force microscopes, a CAMECA SIMS, a Physical Electronics PHI 660 Scanning Auger microprobe, and an XPS Surface Science XPS/LEED/AES instrument.

So far as their plans for additional instruments are concerned for the next four or five years, they are mainly addressed at increasing their capabilities in techniques other than classical electron miroscopy: small angle x-ray scattering, an imaging XPS, a Time-of-Flight SIMS, a high-resolution Scanning Auger Spectrometer, a Focussed Ion-Beam Microscope, and a Scanning Atom Probe. In 2002, they hope to purchase an Environmental SEM.

4.2.3 Staffing

The center is headed by Dr. Ivan Petrov, who reports to the Director of the Frederick Seitz Materials Research Laboratory. There are five major sections within the center: the Electron Microscopy group, the X-Ray Diffraction group, the Microchemistry/Surface Analysis group, the Accelerator group, and the Scanning Probe Microscopy group. There are five staff scientists in the Electron Microscopy group: V. Petrova, Dr. R. Twesten, Dr. J. Mabon, Dr. W. Swiech, and Dr. Y. -W. Kim; one staff scientist in the X-Ray Diffraction group, Dr. M. Sardela, and one technical support staff, K. Colravy; three staff scientists in the Microchemistry/Surface Analysis group, J. E. Baker, N. L. Finnegan, and Dr. R. Haasch, and two technical support staff, S. Burdin and E. A. Sammann; one technical support staff member in the Accelerator group, B. E. Clymer; and three staff scientists in the Scanning Probe Microscopy group, V. Petrova, N. L. Finnegan, and Dr. J. Mabon, and two technical support staff, S. Burdin and E. A. Sammann. This totals 10 instrument scientists who are responsible for the teaching of new users, for scientific interactions with the users, developing the frontiers of instrumentation science, and carrying out their own research. The 4 technical support staff have prime responsibility for maintenance and instrument upgrades. They are experts in electronics, mechanics, and vacuum technology. Thirteen UIUC faculty members are associated with the center, 6 from Materials Science, 3 from Physics, 3 from Chemistry, and 1 from MATSE.

In general, service contracts are not used, since the center has the necessary facilities, trained technical assistance, and outstanding electronics and ultra-high vacuum machine shops available. They do have service contracts for the JEOL 4000 ($42k/y) and the Philips CM12 ($18k/y) TEMs

4.2.4 Costs

The operating costs for 1998 and 1999, and the expected costs for the next two years are listed below.

Operating and Capital Expenses
(Dollars in Thousands)

	Operating Expense			Capital Expense
FY	Salaries	Non-Salary	Total	Capital Equipment
1998	594	293	887	726
1999	658	227	885	2050
2000	740 *	276 *	1016	1305
2001	916 **	292 **	1208	1000

* Includes new hires: 1 staff scientist and 1 research associate
** Includes new hires: 1 staff scientist and 1 research associate

CMM receives no recurring funds from DOE or either operating expenses or capital equipment. Operating funds for the CMM are obtained from user fees (φ 65%) direct support from the College of Engineering and the University (φ 26%), and the FS-MRL Director's discretionary funds (φ 9%) from the State and University.

The Director of the FS-MRL is currently in negotiations with OBES/DOE and UIUC to eliminate user fees. The center believes that this type of support would strengthen their position as a national laboratory. User fees are currently charged to all users of CMM facilities, at rates of the order of $90/hour (there are variations in the charges, but this is to give an idea of the magnitudes). The income from these user fees is approximately $600k/y.

Maintaining CMM as a first-class state-of-the-art facility requires the replacement or upgrade of one to two major instruments per year (this is consistent with our other estimates elsewhere in this report). The special allotment support received from DOE for major equipment on a non-recurring basis has been crucial. The FS-MRL has been fortunate (their words) in securing other funding sources. Averaged over a ten-year period the support for new instrumentation has come from:

- 42% - FS-MRL decision to reallocate research support for critical instrument needs
- 34% - successful FS-MRL faculty and staff equipment grants from the federal government
- 9% - DOE special allotments
- 9% - the University and College of Engineering
- 6% - FS-MRL UI discretionary funds.

Over the past two years, the FS-MRL faculty and CMM staff have been successful in winning major NSF equipment grants for $1,000,000 and $450,000 to purchase a FEG/STEM/TEM and an Imaging XPS with an *in situ* experimental chamber.

Web page: http://ntweb.mrl.uiuc.edu/cmm/cmmhome

4.3 Electron Microcopy Center for Materials Research
Argonne National Laboratory
Argonne, Illinois

4.3.1 Mission and Vision

The Electron Microscopy Center at Argonne National Laboratory provides facilities and personnel to serve the research needs of the Materials Science Division and other Divisions at ANL, to perform collaborative research with several area universities, and to serve a group of national and international users. The principal capabilities historically have centered on a unique facility involving ion accelerators coupled with an older high-voltage electron microscope and a newer Intermediate Voltage Electron Microscope to study the microscopical effects of radiation damage in materials. New instrumentation will be coming to ANL in the near future, including high-resolution field-emission gun scanning and transmission energy-filtered electron microscopes.

4.3.2 Major Equipment

This center was based originally on a unique capability: the ability to view the impacts of energetic ions on specimens within a transmission electron microscope (TEM). Originally, it combined two ion accelerators, a National Electrostatics Corporation 2 MV Tandem Ion Accelerator and a NEC 650 kV Ion Injector which can produce ion beams from 10 keV to 8 MeV of most stable elements in the periodic table, with a High Voltage TEM, a Kratos/AEI EM7 with a maximum voltage of 1.2 MV. The HVEM was necessary because for the results to be relatable to the behavior of real structural materials the specimen has to be relatively thick. This facility is referred to as the Tandem-HVEM, and dates from 1979. The Kratos has been continuously upgraded, and has an impressive set of stages, including high-tilt stages, hot and cold straining stages, a high-temperature stage, and (more recently) an environmental stage. However, the electrical control system has been slowly deteriorating, and operation at the higher voltages is possible only for short times; the majority of the work now uses a voltage of 900 kV or so. By modern standards, the point-to-point resolution of 2 nm is relatively poor. In 1995 a Hitachi 9000N AR microscope was added to the HVEM-Tandem facility. This has a maximum voltage of 300 kV, and a point-to-point resolution of 0.25 nm. This year, a light-element EDXS system will be added to this microscope. Historically, the HVEM-Tandem facility derives from the interest of ANL in the effect of irradiation on the properties of structural materials, and this interest is now supplemented by interest in materials involved in radioactive waste storage.

In 1981, a JEOL-100 CXII100kV TEM/STEM was added; this is still in service, but the center believe that it will be retired in 2004. A Philips EM420T 120kV was installed in 1985; this has light element EDXS EELS, STEM, energy filtered imaging and diffraction and electron dosimetry. It is expected to be retired in 2002, but it is being transferred out of EMC in February 2000.

A Philips CM30T 300kV with light-element CDXS and PEELS was added late in 1987; the center believe it will still be serviceable in 2007.

A High Resolution JEOL 4000 EXII 400 kV microscope with 0.165 nm point-to-point resolution was installed in January 1992, and will continue to be in operation until 2012.

A VG HB603Z 300kV Dedicated STEM with a wide range of capabilities was delivered in 1992, but this is not part of the EMC; it is part of the Defects and Disordered Materials Group.

In addition to these transmission microscopes, a Hitachi S-4700-II 30kV Cold FEG-SEM will be installed in February 2000.

In the next 1-2 years, the center believe their most pressing need is for a 200 or 300kV FEG-TEM/STEM with EDXS, energy-filtered imaging and diffraction, high-angle annular dark-field detector, and holography capabilities. Such an instrument would see use by all Argonne programs currently using the EMC and requiring high spatial resolution structure and chemistry. Additionally, new groups would be interested in local magnetic field measurements by electron interference microscopy (holography). Quantitative measurement of contrast from defect microstructures would be used in studies originating at the HVEM-Tandem Facility.

The center remark that the HVEM-Tandem microscopes are open to outside ANL use for approved projects; the remaining microscopes are primarily (though not exclusively) associated with use by the Materials Science Division and other scientific divisions at Argonne.

4.3.3 Staffing

The staff of the EMC consists of two Principal Investigators, five Scientific Associates, two post doctorates, and a secretary (30% time). Group Leader and PI is Dr. Mark Kirk, PI for the HVEM-Tandem Facilities is Dr. Charles Allen. Three Scientific Associates, Ed Ryan (Operations Manager), Tony McCormick (assistant Operations Manager) and Stan Ockers, are associated with the HVEM-Tandem Facilities, managing, maintaining instruments and assisting users. Two Scientific Associates, Dr. Russ Cook (Philips analytical TEM and Hitachi SEM instrument scientist) and Dr. Roseann Csencsits (JEOL high resolution TEM instrument scientist) are responsible for four microscopes, sample preparation facilities, image processing facilities, and user assistance. All microscopes but the HVEM are under service contracts. To varying degrees, McCormick, Cook and Csencsits also perform research in collaboration with ANL scientists outside the EMC.

When the S4700 FEG-SEM becomes operational in Feb 2000, another Scientific Associate, Richard Lee, an expert in SEM research, will assist in working with users, as a large number from his division, Energy Technology, are expected.

The two post doctoral positions are jointly held with the EMC and currently the Ceramic Thin Films and Interfaces Group (Dieter Wolf, group leader), and the Materials

Chemistry Group (Dieter Gruen and Alan Krauss, co-group leaders). Every several years these post doc positions are intended to rotate among primarily MSD groups, so that expert TEM research is made available to programs with substantial need. This is also designed to encourage those groups to eventually fund their own TEM post doc position. With the acquisition of an FEG-TEM/STEM in the next two years, a new PI position will be established with expertise in a critical aspect of the new capabilities of such an instrument, such as Z-contrast, electron holography, or energy filtered imaging.
A position for a sample preparation expert is expected within the EMC in the longer term, when the present expert, Bernie Kestel, who is currently within another MSD group, retires.

4.3.4 Costs

The total budget for the EMC for FY99 was $1852K, of which 49% went to salaries and 35% to laboratory overhead. The remainder for materials and supplies was about $300K, of which about $150K went to microscope service contracts.

Generally, there are no user charges for non-proprietary research. Charges for proprietary research would be levied on the basis of full cost recovery. However, in the history of the center (1984) and of the HVEM-Tandem Facility before that (1981), we have never had a user engage in a proprietary study. Thus the operating budget of the center is derived from DOE funding entirely.

Web page: http://www.msd.anl.gov

4.4 National Center for Electron Microscopy Lawrence Berkeley National Laboratory Berkeley, California

4.4.1 Mission and Vision

The National Center for Electron Microscopy at Lawrence Berkeley National Laboratory sees its mission to provide forefront instrumentation and techniques for advanced electron beam microcharacterization of materials at high spatial resolution. The broadest challenge to electron beam microcharacterization over the next decade will be to develop the technique into a fully quantitative tool. To meet this challenge, NCEM is planning to further the development of:

- New methods for quantitative image analysis, processing and interpretation;
- New methods and tools for sample preparation; and
- New electron-optical instrumentation and stages.

4.4.2 Major Equipment

The National Center for Electron Microscopy (NCEM) was formally established in the Fall of 1981 as a component of the Materials and Molecular Research Division, Lawrence Berkeley Laboratory.

The first microscope installed was a Kratos High Voltage Electron Microscope, with a 1500 kV accelerating voltage. This is still the highest voltage microscope in the U.S.

However, the high voltage system failed in October 1998, and is currently being repaired in Switzerland. It is not altogether clear at the moment whether this microscope will ever run again. Even if it is, the remaining life is only 2-3 years.

The second major microscope installed was a JEOL 1MV Atomic Resolution Microscope (ARM) in 1983. It has a point-to-point resolution of 0.16 nm, and a high-angle biaxial specimen tilting capability ($\pm 40^{o}$). The remaining life for this instrument is estimated as 3-5 years.

A JEOL 200CX AEM was installed in 1984. This instrument is optimized for microanalysis using EEELS and EDS. The remaining life is estimated as 2-4 years.

The next microscope was not installed until 1993, when two microscopes were added: a Topcon 002B, which is regarded as an introductory HREM with a point-to-point resolution of 0.18nm at $\pm 10^{o}$ tilt; and a JEOL 200CX *in situ* microscope. This latter microscope is very heavily used, and has a remaining life of about 3 years as a standard microscope for teaching and specimen screening.

In 1996, a Philips CM200/FEG optimized for analysis of the physical, chemical and magnetic microstructure at high spatial resolution was added. This instrument is regarded as 'standard but state-of-the-art'.

In 1997, the One Ångstrom Microscope (OÅM) project was started. This was based on a Philips CM300FEG/UT with Cs = 0.65mm and a native resolution of 0.17 nm. Holography, focal series restoration or HREM methods are used to extend the resolution to the desired 0.1 nm. This is currently the highest resolution TEM in the U.S.

Also in 1997 a Spin Polarized Low Energy Electron Microscope (SPLEEM) was added, based on an instrument donated by IBM. This is currently the only working SPLEEM in the world. It has an estimated 5 years of unique performance, and two further upgrading stages are envisaged.

In 1999, a JEOL JSM6340F FEG/SEM was installed. This is a standard instrument, except that it has been modified for electron beam lithography.

So far as the future of the facility is concerned, NCEM has been particularly involved in very high resolution microscopy. There are two possible directions to go in: one is to have a fully aberration-corrected microscope. The second is to have a microscope which is an extension of the high-voltage (1.25MV) ARMII microscope in Manfred Rühle's laboratory in Stuttgart. On balance, NCEM feels that since all of the high-voltage microscopes in the U.S. are very close to the end of their lives that a high-voltage microscope is to be preferred. This is referred to as ARMIII in their plans, and in their view can be installed relatively soon. The NTEAM microscope is unlikely to be available for deployment in less than five years at best, and the estimated lifetimes of the instruments shown above make it clear that a new state-of-the-art high resolution microscope is needed before then. As the center describe it: 'the two unique high voltage

microscopes at NCEM will be replaced with an ARMIII, a high voltage / high resolution instrument that can provide real time sub-Angstrom resolution with integrated microanalytical capabilities. A new post-projector electron decelerator designed by LBNL's Life Sciences group (Downing) will improve electron detection efficiency by two orders of magnitude. In addition this instrument would be the first to fully integrate microanalytical capabilities, an important improvement over present designs. This microscope would be a unique resource for U.S. science.

While 9 new HVEMs have been installed worldwide over the last decade (8 in Japan, 1 in Germany), U.S. capability in this area have fallen behind. Even when aberration corrected machines are operational, it is anticipated that there will be a significant need for an instrument capable of penetrating thick foils, generating local defect gradients, and provide energy-filtered spectroscopic images at reduced ionization damage.

4.4.3 Staffing

NCEM has a permanent staff of 13 for its core operation – 6 scientific, 6 technical and 1 administrative. Additional temporary staff is responsible for instrument development (the SPLEEM) and the collaborative program. The facility provides visitor space and hosts an average of about 10 users at any time.

Permanent scientific staff is dedicated to:
HREM (1.5 FTE),
Computing (2 FTE),
In-situ Microscopy (1.5 FTE),
Analytical and Magnetic Materials Microscopy (0.75 FTE).
Term scientific staff is responsible for SPLEEM (no service contract) (1.2 FTE)
Collaboration, HVEM (1 FTE)

Permanent technical staff is responsible for:
HVEM (no service contract) (1 FTE)
ARM (partial service contract) (0.5 FTE)
CM300, CM200, 002B (service contract) (1 FTE)
FESEM (service contract) (0.5 FTE)
AEM, In-situ (service contracts) (1 FTE)
Specimen Preparation Facility (2x0.5 FTE)
Image Analysis Facility (1 FTE)

Administrative staff is responsible for:
Operations + user access (1 FTE)

Adequate staffing for the next 5 years will require the addition of:
2 FTE for specimen preparation support (an engineer for instrument and technique development and a technician for user assistance),
1 FTE for collaborative projects,
1 FTE for administrative support.

4.4.4 Costs

The total operating budget of the facility is currently about $2,500K and breaks down roughly as follows:

- 70% for fixed salary expenses (permanent staff of 13),
- 10% for service contracts,
- 10% for instrument development (SPLEEM, stage development)
- 5% for utilities and
- 5% for other operating expenses.

Recent Annual Operating Budgets
(Dollars in Thousands)

1995	1996	1997	1998	1999
2.0	2.4	2.5	2.5	2.5

Projected cost increases in the future will be from service contracts and additional staff for instrument support and collaboration.

There is no user fee on any instrument except the FESEM, where we need to charge a $60 hourly fee to avoid undermining competing University and Lab instruments. Proprietary work is fully recharged at a rate of $125/h for self-operators and about twice that with technical help. However, such work is accepted only if it is shown that it cannot be performed at any commercial materials analysis lab. Requests for proprietary use are rare.

Web page: http://ncem.lbl.gov/ncem

4.5 Users

A brief description of the User Mode for each of the centers can be found on the Basic Energy Science web page at www.doe.gov. A more comprehensive report was provided by the EBMCs at the initial meeting of the panel. The report contained user demographics for the past five years, the procedures that are followed to enable a user to work at a particular center, and the names of the people that serve on their advisory, steering, and proposal review committees. The report is attached as Appendix F. The following comments result from an analysis of that report and from information obtained during the site visits.

The DOE Scientific Facilities Initiative (SFI) allowed the overall number of users to grow from 1994 to 1996 by ~50% to a total of ~900 and since then has remained at that number. The funding from the SFI was a great reminder that relatively small influxes of operating funds can make large differences in the usage of expensive and rapidly depreciating capital equipment. The overall number of users appears large but ~44% are students (mostly graduates), and another 15% are postdoctoral fellows. Education of students is clearly important at all centers but especially so at CMM (>90% graduate

students or postdoctorals). The users come largely from U.S. universities (with support from DOE, NSF, DOD, and NASA) and from DOE laboratories. The travel and living costs that foreigners incur may result in the small percentage from foreign universities and laboratories (7%). Only the SHaRE program has funds to assist visitors and the panel's concerns about living costs has already promoted action to reduce these costs at all the centers. The small percentage of industrial users (6%) may be as a result of the charge levied for proprietary investigations or simply that industry does not wish to expose their leading edge research. All of the centers have an Advisory Committee, a Review Committee, or a Proposal Review Panel. This lack of uniformity in the operation of their facilities is clearly the centers' response to the varied backgrounds of their specific users. For all but UIUC there appears to be a much smaller number of hours scheduled for or delivered to users than the number of hours for users allowed by budget.

The User Demographics shows that the education capability of the centers is clearly very strong. Not only are graduate students trained but professional staff are encouraged to come in to the centers and be trained in the use of the equipment. The User Demographics show and the Panel observed during their site visits that CMM/UIUC operates in a very different mode to the other centers. At EMC/ANL, NCEM/LBNL, and SHaRE/ORNL about a third of the users were graduate students or postdoctorates. At CMM/UIUC over 90% were graduate students or postdoctorates. The panel sees no reason why this user mode should change and believes it plays an extremely important role in the education of students in electron beam microcharacterization techniques. Nevertheless it is not clear to the panel that there are benefits to either themselves or to BES/DOE to be considered as an EBMCC. The panel was unanimous in encouraging BES to maintain and strengthen their support for CMM/UIUC but questions why it should be considered as a center.

During the course of their on-site visits the panel talked with a group of users unaccompanied by EBMC staff. These meetings were valuable in establishing the pros and cons of each center's user group. In contrast to the criticisms expressed by some users groups in the 1987 Materials Council report on the EBMCs, we found all users groups well served by the specific EBMCC. At a number of the centers the question of living cost was raised, but as noted above, steps are being taken to alleviate some of these costs. All users spoke highly of the staff of the centers and of their willingness to work overtime to help complete programs. Nevertheless it is clear that the users impose a heavier work load on some of the center staff and it appears likely that OBES is unaware of the problem. Access to microscopes is complicated both by safety regulations and a concern for the welfare of the equipment. It is a problem that needs to be addressed if full utilization is to be made of the capital investment in the instruments.

5.0 THE SCIENTIFIC AND TECHNOLOGICAL CASE FOR THE ELECTRON BEAM MICROCHARACTERIZATION CENTERS

5.1 Introduction

In science and technology, as with other fields of human endeavor, quality and quantity often appear to be inversely related to each other. Overall, in any field the published record contains a small amount of truly superb research, a larger amount of high quality, important work, and plenty of unexciting but often necessary accomplishments. As observed in Section 3, much of the excellent research in electron beam microcharacterization, both technique development and application to materials science has been performed both in the U.S. and abroad in laboratories that are not operated as centers.

We received several different inputs relating to the work done by and with the centers, some of which have been referred to before. At the Panel's first meeting, we received a document prepared by the centers, entitled "Electron-Beam Microcharacterization Centers: A National Resource" (72 pp, June 1999)" which is added to this report as Appendix C. This document lists a number of significant papers produced by or at the centers, grouped in terms of the six scientific and technical areas that the centers had identified early in our discussions. We also received a list of publications for 1996 and 1997. There were approximately 260 journal publications and 100 proceedings articles each year. The panel has no special expertise in defining a good or bad number of publications but if one translates the cost of all the centers into scientific man years (relative to total P.I.s in all centers) then the number of journal and proceeding publications would amount to somewhat over 10 publications per scientific man year. Even in an era of "publish or perish" the output is impressive and it should be noted that all the publications were in well known, refereed journals, and approximately 10% were PRL or APL publications.

Following the first meeting, the panel discussed via e-mail a number of issues, and a letter was sent to the Center Directors requesting further information. One of the questions referred to the contributions that the centers had made to world-leading science over the last few years, and asked each of them to identify the 'ten best' achievements of their center, asking for a mix of those relating to EBMC techniques and those relating to 'materials science breakthroughs'. During the panel's visits to the centers we received further information relating to research in progress. Of course, in addition to these materials made available to us by the centers, many of the panel members were familiar with much of the research in progress there.

In the evaluations we made of the research, we used a classification proposed by Manfred Rühle, who defined three research levels:

- Top Research: New techniques for specific problems
  Quantification of result
  Instrumentation and electron optics
  New techniques for specimen preparation
- Advanced Research: Application of newly developed techniques to specific problems in science
- Routine Research: Routine application of well established techniques for characterization of materials

The panel believes that at a National User Center while the bulk of the research would be in the second of these levels, some of the work should be in the first level, and this is what we were looking for.

As mentioned earlier, the centers had identified six areas of materials science where they believed they were making significant contributions, and the panel therefore organized its review of the research in the same classifications.

5.2 Interface Science

5.2.1 Introduction

In most materials systems there are interfaces of various kinds, and in many cases the properties of the system depend to a significant extent on the properties of the interfaces and the changes in the interfaces with time in service. In the simplest case, a pure metal may be an assembly of crystals, and the boundaries between the crystals which are called grain boundaries are the simplest form of interface. In this case the important property is the relative orientations of the two crystals on either side of the boundary. If the orientations are very similar, the two crystal lattices will form a boundary in which regions are crystallographically continuous, separated by linear discontinuities in the boundary which are called interface dislocations. This is called a low-angle grain boundary. As the difference in orientation increases, the spacing between the interface dislocations decreases, until eventually this description becomes untenable. The boundary may appear to be disordered, and this is called a high-angle grain boundary. There is a region on either side of the boundary where the atoms are displaced to some extent from their regular positions on the crystal lattices of either crystal, and the width of this region is called the thickness of the grain boundary. The properties of this grain boundary region may be significantly different from the bulk of the crystal. For example, the diffusion along the grain boundary may be significantly faster than in the bulk. If the grain size of the material is very small, the volume fraction that is in the grain boundaries becomes significant, and such a material will have properties which may be very different from a single crystal or a coarse grained polycrystal. For example, it may exhibit superplasticity.

In the case of a material which is not pure, the chemical composition of the boundary may be significantly different from the bulk, in part because if the size of the foreign atom is significantly different from the host material, the boundary may offer a location where its presence may induce less elastic strain. In a material exposed to elevated temperatures in service, the impurities may migrate to the grain boundaries over time, and this may result in a time-dependent change in the properties of the material. Many practical materials consist of more than one phase, having different crystal structures; and the boundaries between different phases present new possibilities. Once again, since the separation into the different phases is a matter than can be controlled by the treatment of the material, this presents a variable which can be used by the materials engineer to develop material systems optimized for the application; but carries the risk of time-dependent properties.

In this terminology, the outer surface of a material is an interface, and corrosion processes at a free surface by reaction with the environment can be regarded as an interface process. In high-temperature oxidation, the reaction product collects at the surface and protects the material from further attack. However, the properties of the system may then be determined by the oxide/metal interface, which itself may be subject to time dependent changes as a result of other impurities collecting at the interface.

Understanding the structure and properties of interfaces is thus a crucial matter for materials scientists; and in some cases examination with techniques having high spatial and chemical resolution is the only way to find out what is going on.

5.2.2 Past Highlights

In the opening presentations to the panel, the important issue of grain boundary networks in YBCO (yttrium-barium-copper-oxide, one of the earliest of the high-temperature perovskite superconductors) was discussed. The issue here is that the properties of these superconductors are highly anisotropic; and high-angle grain boundaries can act as Josephson-coupled weak links leading to a significant field-dependent suppression of the supercurrent across the boundary. Controlling the grain boundary misorientation distribution towards low angles is one of the keys to fabricating high critical current density materials. Using the SHaRE Philips XL30 FEG-SEM to generate electron backscatter Kikuchi patterns, investigators were able to show that in the case of a high critical current material the bulk of the film was percolatively connected within a 2^{o} misorientation.

An issue related to composition is presented by high-density recording media of the kind used for computer hard disks. CoCr(PtTa) thin films are one of the materials used for this purpose, but intergranular Cr segregation decouples the magnetic exchange between the small ferromagnetic grains. Cr depletion within the grains adversely affects the bulk magnetic anisotropy. The nanoscale structural and chemical details that are needed for modeling and material development were not well understood. Excellent characterization revealing these effects was achieved at SHaRE using the energy-filtered electron microscope.

Barrier-layer preferred orientation and its effect on interfacial integrity during thermal excursions is expected to become increasingly important as the dimensions of integrated circuits continues to decrease while the number of metallization layers increases. The Al/TiN metal/barrier couple has been the most widely used. In previous studies of the Al/TiN interface reactions, there have been no attempts to vary systematically the TiN film texture or properties. CMM staff worked with the IBM T. J. Watson research center on this, using a novel magnetron reactive sputter deposition system. This research is particularly interesting because it combines the use of synchrotron X-ray diffraction and XTEM. They showed clearly the growth of Al_3Ti, and were able also to show that dense highly (111)-oriented TiN had enhanced thermal stability.

SHaRE grew out of the ORNL Materials and Ceramics Division, and continues to support studies of irradiated structural materials. One of the effects of irradiation of austenitic stainless steels is to enhance stress corrosion cracking. Generally, stress corrosion cracking in stainless steels is a consequence of chromium depletion at the grain boundaries, and one of the possible reasons for irradiation enhanced stress corrosion cracking (IASCC) is radiation-induced segregation (RIS). At the temperatures experienced in light water reactors, RIS occurs over small distances, typically <5nm. Recent studies have used the CM200-FEG analytical electron microscope which enables X-ray microanalysis at a spatial resolution of under 2 nm. These studies showed that RIS enrichment of Ni and Si or P contributed to enhanced Cr depletion. This work has also given some indication of the beneficial effects of Mo in these systems. Pre-existing segregation of B, C and P atoms to the grain boundaries in Type 316 stainless steel has been shown in a three-dimensional atom probe reconstruction.

Understanding of superconducting properties of grain boundaries in YBCO has been significantly advanced by the microscopy of Miller and coworkers. Microstructures in bulk (using a new method of bicrystal growth) and thin film bicrystal boundaries are compared with critical current densities across the same [001] tilt boundaries as a function of misorientation angle (Todt, V. R., X. F. Zhang, D. J. Miller, M. St. Louis-Weber and V. P. Dravid, Appl. Phys. Lett. 69 (1996) 3746.). Enhanced pinning of Josephson vortices at the meandering boundaries in thin films is proposed to explain the much larger critical current density supported across these boundaries compared with more perfectly flat bulk-grown boundaries (Gray, K. E., Field, M. B. and Miller, D. J., Phys. Rev. B 58 (1998) 9543.).

A new wider range of grain boundary geometries has been accessed by the discovery of a templating technique for thin film growth of bicrystals (Merkle, K. L., and Thompson, L. J., Phys. Rev. Lett. 83 (1999) 556.), with which high-resolution imaging of atomic structures of twist and general grain boundaries are reported in gold.

Detailed analysis by diffraction and contrast analysis has shown that many precipitation systems exhibit irrational orientation relationships and interface geometries. (U. Dahmen, C. P. Luo, S. Q. Xiao and K. H. Westmacott). Often, such precipitates form as laths or plates with irrational habit planes or facets. Using the hypothesis of an invariant line transformation strain, irrational lath precipitate morphologies and interface structures

were recently explained in detail for Cr precipitates in Cu, Mo_3Si_5 laths in $MoSi_2$, and S-phase precipitates in Al-Cu-Mg alloys. Based on detailed microcharacterization and comparison with crystallographic predictions, the invariant line concept has now been validated as an underlying principle of morphology in solid state phase transformations. In the past several years, more than 70 publications in other groups have successfully applied this concept to the study of precipitation systems and heterophase interfaces. The most significant application of the concept has been to lath-shaped precipitates in metals and ceramic alloys and to metal-on-metal thin film growth.

5.2.3 Current Strengths/Highlights

Here we consider some of the outstanding work that is on-going at these labs and that was presented to us at our visits.

5.2.3.1 SHaRE/ORNL – There is a continued strong effort in examining the grain boundaries in structural materials. The work on irradiated samples continues and is outstanding. They also have a strong program on intermetallic compounds. They have also worked to develop creep-resistant stainless steels by using analytical electron microscopy to study the compositions of precipitates in the material.

5.2.3.2 MMC/UIUC – Most of the ongoing work at the University of Illinois is strongly connected to research projects of the individual PIs. An excellent example of this work is the activity of D.A. Payne. Ferroelectric material ($BaTiO_2$) was processed with different grain sizes and the dielectric constant was measured. The authors showed that for very small grain sizes a break down of the properties occurred. These studies are extremely important for a correlation between microstructure and properties of polycrystalline $BaTiO_2$. The TEM studies are crucial for the investigations, although only conventional TEM is required. However, the coupling with materials science is impressive. Similar conclusions can be drawn for other interesting and valuable investigations.

5.2.3.3 EMC/ANL – Excellent research is being done in the interfaces group of D. Wolf where stable atomistic structures are simulated by molecular dynamics calculations. Wolf is one of the leaders in that area. However, this work was not presented to the panel. Merkle is performing the experimental HREM studies of exactly the same boundaries that are calculated by Wolf. This comparison of theory and experiment gives insight into the true nature of grain boundaries. The HREM videos presented which show the movement of boundaries were outstanding and presented many new and unusual features of grain boundary motion. This work should be of great importance. Merkle and Thompson have also worked out a template technique to control grain boundary orientation in bi-crystal growth.

5.2.3.4 NCEM/LBNL – The National Center for Electron Microscopy (NCEM) concentrates on high-resolution TEM. After a period of stagnation, they have had recent growth. All instrumentation and computational techniques for HRTEM are available. Excellent research has been done on interfaces in different materials and strong efforts have been undertaken for quantification (including reliability) of all

experimental data acquired with HRTEM. Image simulation is done on a high level, although at a slow pace (O'Keefe). The comparison of experimental images to simulated micrographs is done on a now standardized way (Kilnas). It is important that the two groups be linked together with a clear vision.

The OÅM is now running well and programs developed by Thust are well incorporated and applied to a specified defect in GaN. Excellent work has been completed on interfaces of precipitates in aluminum, grain boundaries in rutile, defects in GaAs, and semiconductor heterostructures. It would be desirable that the lab have stronger links to other scientists either at LBNL, UC Berkeley, or at other institutions. Specifically, strong collaborations could be made with Wolf at Argonne, Vitek at Penn, Srolovitz at Princeton, who are doing theoretical modeling of interfaces and correlating these models with properties.

5.2.4 Future Perspectives

The ultimate goal of interface science will be the column-by-column analysis of atoms from the matrix up to the interface of interest. The analysis includes, as mentioned above, quantitative determination of structure, composition, and bonding. Besides phase contrast imaging, which is being performed at ANL and LBNL, STEM imaging (as done by Pennycook) should be done at the highest possible level. Compositional maps on a column-by-column basis should be acquired and at the same time ELNES spectra should be taken. The data have to be analyzed by comparing them to calculated ELNES spectra, a major commitment of a theoretician.

For the dynamic behavior of interfaces under load and high temperature, in-situ experiments have to be performed in a manner similar to that performed by Merkle. The possibility of observing segregation is particularly exciting and would be a great addition to the many studies in the past in which heat treatments were first performed and the segregated boundary examined.

It would be interesting if dynamic experiments could be performed in an environmental cell. Those studies are relevant to many different materials and materials components. This work would include studies of interfaces during phase transformations (e.g. in Al_2O_3) oxidation processes, chemical reactions at heterophase boundaries (e.g. metal/ceramic boundaries in thermal barrier coatings) and studies of specific interfaces in materials of interest for energy generating systems.

5.3 Phase Transformations and Alloy Design

5.3.1 Introduction

The subject of phase transformations plays an important role in establishing the fundamental principles of the dynamic evolution of the microstructure of materials at different scale levels (from atomic to macroscopic) that are key components in the integrative process of alloy (materials) design. This topic is also of central importance to the current DOE-OBES interest in materials as complex adaptive matter. Much of the theory of the mechanisms of first-order solid-state phase transformations was established

before the capability had been developed to directly observe them. In the last thirty years high resolution microscopy and microanalysis have emerged as the principal tools for the study of this important topic. The science of alloy design is based entirely on the intelligent and controlled application of phase transformations to obtain tailor-made microstructures. For example, dispersion hardened alloys that find applications ranging from airplanes to soda cans rely on an even distribution of strengthening precipitates to impede dislocation motion on a microscopic scale. The crystal structure, shape, size and distribution of the strengthening precipitates, and with them the strength of the alloy, are the direct result of the nucleation and growth of a new phase. Similarly, high strength steels derive many of their properties from a diffusionless (martensitic) transformation. Even the apparent paradox of tough ceramics has become possible by designing microstructures that utilize martensitic transformations for transformation toughening.

The ability to identify phases by crystal structure and composition is central to all experimental research in materials science. Electron beam micro-characterization has been the major tool to help develop our current understanding of microstructure-property relationships. Electron beams are the foremost probe for identification of finely distributed phases whenever spatial resolution is of the essence.

Two of the four centers (SHaRE/ORNL and NCEM/LBNL) have active programs in phase transformations and alloy design.

5.3.2 SHaRE/ORNL

5.3.2.1 Past Highlights - The SHaRE/ORNL Center has a strong tradition in the study of phase transformations coupled with alloy development. Much of this work has been driven by the demands of the electrical utility companies for enhanced performance from conventional metallic alloys used as key components in power generation, as well as the development of new materials. This includes the design of creep-resistant stainless steels, the development of welding procedures and new weld metal formulations for pressurized components through phase identification and control of transformations, and the development of structural intermetallic compounds for high temperature applications. The panel was impressed by the strong industrial interactions that have developed through the SHaRE program. While these interactions are pervasive, the quality of the science is high, the research is fundamental, and the outcome of the work has been far reaching.

5.3.2.2 Current Strengths/Highlights - The SHaRE/ORNL Center has several unique capabilities. As noted elsewhere in this report, they are one of a handful of laboratories in the world developing the techniques of APFIM for studying the first stages of phase transformations in metallic systems. The APFIM work on high temperature Ni-based alloys and multiphase aluminides has led to an in-depth understanding of phase development in these complex, multi-component alloys which cannot be achieved by any other method. Although the technique of ALCHEMI was developed elsewhere, SHaRE/ORNL has refined and applied ALCHEMI in a number of elegant studies of intermetallic compounds and superalloys. They have also

developed the techniques of energy filtered imaging in the TEM, using characteristic energy losses in the electron energy loss spectrum, to study problems of grain boundary segregation (sensitization) in stainless steels. This research is a particularly good example of the synergy that exists between the different techniques at the disposal of the microscopist today: one that the SHaRE/ORNL group have used to good effect in solving "real" engineering problems in a collaborative environment.

5.3.2.3 Future Perspectives

The acquisition and continued development of the Kindbrisk ECOPoSAP is a major advance in capability that will clearly impact phase transformation and alloy design research. In particular the instrument should yield statistically significant data, allowing the quantification of phase composition, morphology and interphase boundary composition in the first stages of phase transformations. If the development of an aberration free, low-voltage FEG-SEM, coupled with a new X-ray detection system (e.g. the bolometer design), proceeds, a number of unique opportunities will arise for microstructural characterization of phase transformations in bulk samples with a resolution approaching that currently achieved in TEMs. In parallel with SEM developments, continued research and application of filtered imaging in the transmission electron microscope would also have a high impact. In general, the development of these techniques should lead to a more quantitative understanding of phase transformations, especially at the atomic level.

We also recognize the importance of continuing international and local collaboration in these developments. We conclude that the developments and applications at SHaRE represent the best example of strong links with materials technology development evident at the four centers reviewed.

5.3.3 NCEM, LBNL

5.3.3.1 Past Highlights – NCEM/LBNL has a strong tradition of fundamental phase transformation research and alloy development with TEM, both of which are excellent. Much of this work has relied in the past on the strong links that were developed between the NCEM and the Department of Materials Science and Engineering at the University of California (Berkeley). This collaboration has led to the development of high strength steels, new magnetic materials and ceramic alloys.

5.3.3.2 Current Strengths/Highlights – The NCEM/LBNL has continued the tradition of high quality microscopy of atomic/nanoscale mechanisms of solid-state phase transformations, established in the 80's under the leadership of Gareth Thomas. Some recent noteworthy examples of phase transformations research are (a) the analysis of strain-induced martensite formation in Si at twin intersections induced by hot deformation; (b) the complete characterization of the Al_2CuMg (S) phase and its precipitation behavior in Al alloys, and (c) the development of general invariant line concepts in solid state precipitation. However, claims of a strong connection between this work and the concepts of alloy design (as the review panel understands this term) were not supported by the information provided.

5.3.3.3 Future Perspectives

Future plans for NCEM/LBNL emphasized the further development of *in situ* capabilities and more quantitative nanoscale analysis of multi-component alloys. These objectives are well supported by the further application of the TopCon and JEOL 200CX systems. The proposed HVEM instrument ARM III (discussed elsewhere in this report) would support *in situ* studies in thicker specimens, important to the study of phase transformation phenomena in metals, ceramics, glasses and semiconductors. On the other hand the proposed NTEAM instrument would provide an enhanced capability for spectroscopic studies, but would be restricted to the study of thinner specimens. If a choice had to be made, the panel believes that the ARM III would have the greater impact in the context of phase transformation studies. A number of unique opportunities arise to capitalize on the strengths of NCEM/LBNL if one or both of these instruments were to be obtained. These include automated structure refinement from nanophase components in microstructures, nanoscale compositional analysis with single atom detection, the direct observation of the mechanisms and dynamics of solid-solid and solid-liquid phase transformations, and the atomic structure of glasses.

However the panel was concerned that the phase transformation work at NCEM/LBNL appeared to be poorly linked to alloy design. We propose that better scientific and technological impact in this area might be fostered by a stronger connection to groups working in alloy design, and by a closer collaboration with theorists working in the area of phase transformations.

5.4 Defects, Deformation and Radiation Effects

5.4.1 Introduction

Defects control the properties of most of the materials made by man. The ultimate strength, for instance, of a material depends on defects. The strongest materials either have no defects or have so many defects that their mutual interaction controls the mechanical behavior. To make materials without defects or with controlled defect populations requires a deep fundamental understanding of defect structure and behavior. Understanding the relationships between defects and properties of materials is an important goal of the materials scientist, one which enables new processing techniques to enhance material strength, to reduce property degradation from exposure to various environments, and to improve magnetic and electronic device performance down to a nanometer scale.

In the same way, point defects and defect clusters are of great importance for the properties of materials. In thermal equilibrium they play a central role in phase transformations, kinetics and materials processing. When produced by radiation, they are of critical importance for nuclear reactor materials, long term storage of nuclear waste and the modification and processing of materials with ion beams. Electron beam microcharacterization continues to play a principal role in research on radiation effects in materials.

The most powerful tool to study defects in solids is the electron microscope. Electron beam microcharacterization has been the key to developing our current understanding of dislocations because virtually all our current experimental knowledge on dislocation reactions and behavior has come from analysis using electron microscopy.

Defects formed by irradiation of metals, semiconductors and superconductors have been most effectively characterized by TEM techniques. *In situ* irradiation and straining experiments have revealed defect formation and interaction mechanisms that cannot be discovered in any other way.

The characterization and analysis of defects, deformation behavior and radiation damage in materials is a particularly significant area to the Department of Energy. These analyses provide fundamental understanding of material behavior and strength, and are also important for the development of new materials. Thus, maintaining expertise in these areas is critical for understanding materials behavior. The panel recognizes the need for this type of research within the national centers.

5.4.2 Past Highlights

Both ANL and ORNL have a long and distinguished history of radiation damage research and defect analysis. The Electron Beam Microcharacterization capabilities at these laboratories have played a significant role in resolving such technologically important problems as irradiation embrittlement, fuel swelling and irradiation assisted stress corrosion cracking. In particular, ORNL has successfully applied AEM and high spatial resolution microanalysis to the characterization of radiation-induced segregation in ion-irradiated and neutron-irradiated materials. The HVEM at EMC/ANL has provided in situ straining capabilities for studying the development of dislocation structures in metals. Irradiation and environmental effects have been studied extensively at EMC/ANL and CMM/UIUC. HREM and modeling efforts at NCEM/LBNL have provided atomic resolution images of defects.

5.4.3 Current Strengths/Highlights

The IVEM and HVEM Tandem Accelator facilities at EMC/ANL are one-of-a-kind facilities for in situ irradiation damage studies. This is corroborated by the substantial list of current collaborative and user programs for the EMC/ANL, including international users. Significant applications include studies of: radiation stability of waste glass; Zr_2Cr amorphization in Zr-4; the discovery of pinning vortices in high T_c superconductors; novel in situ fracture experiments and mechanistic interpretation; cascade production and analysis leading to the formation of voids, and basic TEM studies of defect formation in ion-irradiated pure metals.

At SHaRE/ORNL, the FEG-AEM studies of intergranular segregation in steels (Irradiation-Assisted Stress Corrosion Cracking) and the APFIM characterization of irradiation-induced solute clusters in reactor pressure vessel steels and welds (to explain irradiation embrittlement), are both examples of excellent microcharacterization studies. Other important work includes the AEM studies of creep resistant steels for power generation applications.

CMM/UIUC: The 400 kV environmental microscope has enabled unique high quality in situ studies of dislocation motion during straining in hydrogen environments in order to assess the mechanism of hydrogen embrittlement in various alloy systems.

5.4.4 Future Perspectives

EMC/ANL: For current and future research efforts, it is very important that EDS capability is added to the IVEM to permit complementary microchemical analysis. The IVEM/Tandem Accelator facility is expected to continue to provide unique *in situ* experimentation opportunities in the development of waste glasses, as well as for the assessment of phase stability in structural materials for nuclear power applications, and for radiation-induced defects production and amorphization in semiconductors. However, it is important for the center to expand its focus to include nanophase and soft materials while maintaining the IVEM/Tandem capability.

SHaRE/ORNL: The recent acquisition of the 3D-AP (Kindbrisk ECOPoSAP) will enable improved 3D morphological and microchemical characterization of the complex solute-enriched clusters associated with hardening in irradiated materials. Additional improvements in the FEG-AEM evaluation of radiation-induced segregation could be achieved through the incorporation of a microcalorimeter/bolometer for high energy resolution x-ray microanalysis.

NCEM/LBNL: The acquisition of ARM III coupled with advanced modeling and simulation will permit the more detailed characterization of defects in a broader range of materials. The present state of the 1.5 MeV HVEM (the highest voltage electron microscope outside of Japan) requires evaluation. It remains an effective tool for in situ electron irradiation experiments, and is recognized as such internationally.

Significant challenges exist in both defect physics in materials and in the microscopy of defects. The exact structure of some defects has been determined in just a few materials, mainly metals, but remains largely unknown in most materials. The interaction forces between dislocations and pinning defects in metals, and between magnetic vortices and pinning defects in superconductors, are unmeasured experimentally. The electrical and magnetic nature of defects in electronic materials has not been measured locally. The strain fields around dislocations or precipitates need to be measured precisely to understand and quantify their function in the microstructure.

These challenges can be met by recent and expected advances in microscope design and technique. Increases in atomic resolution will advance our ability to determine defect structure. However, advances in computer modeling and image simulation of defects with comparison to quantitative recording of image contrast will provide the best opportunity to determine defect structure in any material including glasses. Chromatic and spherical aberration correction will present a tremendous opportunity for more quantitative *in situ* experimentation, including electric field, magnetic field, and strain field distributions around defects, and their dynamic interactions among themselves, dislocations and magnetic vortices.

The expected advances will allow opportunities for studies of: the imaging of individual point defects and their clusters; the quantitative correlation of the nucleation, motion and interaction of dislocations with local stresses; the imaging of dislocation core structure in metals, alloys semiconductors and ceramics at sub-Ångstrom resolution; and the high-resolution, high-precision mapping of localized strains in materials.

5.5 Nanostructured Materials

5.5.1 Introduction

The behavior of solids in the nanometer size regime, as their dimensions approach the atomic scale, is of increasing fundamental and applied interest in materials research. Electronic, optical, magnetic, mechanical or thermodynamic properties all may depend on the size and shape of the solid. As a result, in the nanoscale regime, size and shape may be used as design variables to tailor a material's properties such as giant magnetoresistance in multilayer films, or the optical properties in semiconductor nanocrystals.

Although in most cases the size dependence of properties is not well understood, nanophase materials hold great promise for breakthrough advances in materials science. In support of these advances, accurate nanoscale characterization is extremely important. In this area transmission electron microscopy plays a critical role, particularly in cases of buried nanophase structures such as small inclusions, thin film multilayers, quantum wires or nanotubes and other types of fullerenes.

5.5.2 Past Highlights

The field of nanostructured materials is relatively new but it is clear that for their experimental study electron beam microcharacterization with high spatial resolution is essential. For example, the existence of nanotubes was discovered by electron microscopy, their elastic modulus was derived from electron optical experiments, and the first observations of diamond formation inside a bucky onion were made with high voltage electron microscopy.

5.5.3 Current Strengths/Highlights

At NCEM/LBNL the direct observation of tunneling effects from STM tips and other nano-electronic effects (e.g. quantized resistance) using *in situ* HREM techniques has developed rapidly over the last decade. Many of these developments have occurred in Japan. If low temperatures can also be used, the panel sees considerable opportunities for the growth of this area of *in situ* mesoscopic physics, and we therefore encourage a tolerant attitude amongst NCEM/LBNL staff for the instrumental modifications needed to do this work. In particular, Alex Zettle's student's work on the observation of nanotube contacts by in-situ TEM impressed the panel as having exciting possibilities. Secondly, the work on melting of nano-scale lead particles in alloys and on magic number size distributions in similar particles, and study of its energetics was an important highlight. Finally, the work on patterned Co/Pt multilayers by Lorentz microscopy (and correlated Kerr microscopy) impressed the panel with its ability to relate magnetic, crystallographic and morphological properties. Notable work by external users included that on

interstellar diamond in meteorites, and the work on magnetite/calcite, relevant to the issue of life-forms on mars.

At CMM/UIUC strong support of local academic research and ideas was evident. Several highlights impressed the panel including David Payne's group work on grain-size effects in ferroelectric and dielectric nanostructures, work on bimetallic Pt-Ru nanoparticles, work on Ge/Si quantum dots by TEM, and studies of polycrystalline Si films by magnetron sputtering together with low energy particle beams. The panel was also excited by the promising results from studies of GaInAsP quantum wire heterostructures. The work on in-situ sintering by UHV TEM was particularly impressive, and the impressive research on catalysts might also be considered "nanostructural". The panel was also most impressed by the Erhadt/Nuzzo/Jeon/Whitesides collaborations and the exciting possibilities for contact printing lithography using SAMs on gold. The new method for mapping strains around Ge quantum dots by Miller et al, which addresses an old and important problem, was noted.

At EMC/ANL nanostructural studies have not been traditional areas of concentrated effort. However we were impressed by the work on diamond nanocrystals formed as a result of irradiation, and its implications for fission processes in geology rather than meteoritic impacts. In addition the use of speckle microscopy for the study of radiation damage appears to have exciting possibilities, and is enthusiastically endorsed.

At SHaRE/ORNL the Vanderbilt work on magnetic storage media based on nanostructured materials addresses an important industrial need, which requires the unique capabilities of the ORNL instrumentation and staff (energy filter, etc), and is a fine example of the spirit of the highly successful SHaRE program.

5.5.4 Future Perspectives

Future developments of nanophase materials will depend to an extreme degree on the ability to characterize their structure, composition, bonding and behavior with atomic resolution. In the more distant future the NTEAM environmental microscope, with its larger space in the pole-piece, could greatly facilitate this work.

Future opportunities will include: direct tests of theory as experimentally observable and computationally accessible nanoscale systems scales converge; *in situ* measurement of electrical and mechanical properties of individual nanotubes and the direct observation of Fullerene formation; grain boundary structure in nanocrystalline solids and the size- or shape-dependence of phase transformations in the nanoscale regime; nanophase structure determination and refinement; understanding of interface structure in nanocrystalline solids and size- or shape-dependence of phase transformations in the nanoscale regime.

5.6 Thin Films and Surfaces

5.6.1 Introduction

With miniaturization of components and devices, surfaces and interfaces will assume even greater role in controlling and dictating materials performance. Thin films and

surfaces present daunting challenges to materials scientists owing to both spatial and dimensional constraints imposed in these materials systems. EM in many forms has played a vital role in analysis and will no doubt continue play even more decisive role in imaging and analysis. The advent of (UHV) LEEM and SPLEEM is providing surface scientists with exciting and unique techniques for forefront analysis of statics and dynamics of surfaces and surface mediated phenomena. All EBMC centers are capitalizing on instrumentation and technique development to probe details of surface and thin film phenomena.

5.6.2 Past and Current Strengths/Highlights

Excellent work is being done in the following four areas: refractory metal molecular beam epitaxy thin films (CMM/UIUC synthesis of thin film grain boundary bicrystals (ANL); magnetic imaging via SPLEEM of early stage of thin film growth (LBNL); and the collaborative research on polycrystalline magnetic thin films (SHaRE/Vanderbilt University).

The group at UIUC headed by Peter Flynn is doing world class work utilizing LEEM to understand the growth and dynamics of thin films of BCC metals prepared *in situ* in LEEM by MBE. This work represents a unique combination of advanced technique (LEEM), *in situ* thin film growth (MBE in LEEM) and dynamics (LEEM at elevated temperature) coupled with extensive expertise in thin film phenomena at UIUC (Peter Flynn, Joe Greene groups). The work highlights the role of interface strain, substrate constraint, crystallography and surface steps on growth and dynamics of BCC metal thin films.

At EMC/ANL, Karl Merkle has utilized an elegant method of preparation of thin film textured and bicrystal thin films as model system for understanding atomic structure of interfaces. The unique feature of this work is not only the elegant synthesis of "controlled" thin film bicrystals but the combination of experimental analysis of grain boundary atomic structure coupled with atomistic simulations of grain boundary structure (D. Wolf) – in the spirit of combinatorial analysis of thin film nanostructures.

At NCEM/LBNL H. Poppa has shown that the SPLEEM instrument to be a highly innovative instrument in the hands of a research team which, in collaboration with Professor E. Bauer, holds a position of international leadership in low energy electron-optical design. The instrument is thus capable of putting the lab in a position of international leadership in the area of thin film magnetism. Comparisons with the PEEM instrument on the ALS will inevitably be made – each has strengths and weaknesses. In particular, by comparison with the PEEM, the SPLEEM allows the incident beam polarization direction to be rotated, allows fast imaging (movies), provides better resolution, low temperature operation, and can provide atomic resolution in the direction normal to the surface. The instrument allows three-dimensional mapping of local magnetization. It lacks, however, the element-specific imaging capability of the PEEM resulting from the tunability of the ALS. The panel sees many exciting opportunities for the application of this instrument to the important and rapidly growing field of micro-

magnetics and the study of the relationship between magnetic properties, defects, and crystal growth.

The SHaRE program at ORNL provided an excellent example of collaborative research in true spirit of the program with Prof. Jim Wittig of Vanderbilt University. The nature and role of GB segregation of Cr was elucidated with the combination of imaging filter (which provided a pictorial and quantitative view of the grain boundary segregation of Cr in grain boundaries of polycrystalline magnetic films for storage media) and magnetic measurements (especially noise) on the same films. The results highlight the role of Cr segregation in "ferromagnetic decoupling" of the magnetic grains and provided technological useful results in designing of magnetic storage media. The work involved industrial participation and subsequently industrial support for the work (Komag, Inc.).

Other high quality studies include:

The work at EMC/ANL in collaboration with a small industry (Conductus), on thin films with the extensive microstructure characterization of grain boundary junctions in high T_c SQUIDS. These technologically important thin film structures were carefully prepared for x-TEM analysis which requires skillful specimen preparation. The TEM work complemented transport and related measurements of the superconducting properties of the SQUID, and contributed to the understanding of growth and microstructural issues in SQUID devices.

The work at CMM/UIUC utilizing unique surface science instrumentation and techniques to resolve important thin films issues of growth, stability and dynamics of TiN films. Joe Greene and his group have provided an excellent account of Ostwald ripening in thin film nanostructures via *in situ* dynamic STM at elevated temperature. The results are significant in terms of fundamental science of thin film growth/stability which has significant technological overtones.

The work at NCEM/LBNL studying magnetic thin films and multilayers (GMRs) which has involved considerable technique development; the Lorentz microscopy studies, the DPC mode, and the role of interface strain and growth parameters); the GaN thin film work – quantitative CBED/polarity analysis, role of dopant distribution, internal potential imaging via e-holography; and the work with the OÅM that is beginning to contribute significantly, especially in imaging light elements and determination of interface structures in thin films and multilayers.

5.6.3 Future Perspectives

At ANL the new leadership in MSD has brought a renewed vigor and sense of optimism for the role of advanced EM in thin film research. The planned acquisition of an FEG TEM/STEM should revitalize work in area of thin film in grain boundaries, high-temperature superconductor films, and assembled nanostructure thin films (Jeff Eastman).

At SHaRE/ORNL the new TEM/STEM will provide additional opportunities for thin film studies.

At NCEM/LBNL the proposed in-situ magnetic field SPLEEM should yield interesting and exciting results in dynamics of magnetic phenomena in ultra-thin films. With the OÅM on-line and the development of specialized techniques with FEG TEM, work on GMR/magnetic thin films should continue to yield useful and interesting results.

The present collaboration with local industries is likely to reach new heights with establishment of new techniques of holography, DPC and atomic resolution imaging.

At CMM/UIUC the LEEM will no doubt continue to provide exciting results on surface and thin film phenomena. The VT STM would likely continue to play a significant role in dynamics of thin film processes. The new FEG TEM/STEM should revitalize advanced research in semiconductor thin films and assembled nanostructures.

5.7 Microelectronics Materials and Devices

5.7.1 Introduction

The electronic and photonic revolution, which has occurred in the past fifty years, has had a profound impact on our way of life and national economy. Central to this process has been the continual development of the materials and processing technologies necessary for the production of devices. Electron microscopy has been crucial to these endeavors. The microelectronics revolution is driving the Si-based devices to smaller and smaller dimensions and electron-optical imaging is the only technique able to resolve the structures that are produced. In particular, gate oxide widths are now too small to be seen by SEM so that new STEM or TEM modes must be used. Moreover, the thicknesses of the SiO_2 gate oxides in current devices are now in the tens of Ångstroms and limiting thicknesses are being approached. Understanding the limitations of these oxides and, for example, the structure of the crystal/amorphous interface is a key challenge for the industry.

Important challenges for materials science are also posed, for example, in the world of high temperature electronics where materials such as SiC or diamond are being developed. Understanding local defect structures and interfacial properties is essential for their successful development and again electron microscopy IS the key analytical technique. Active and passive photonic devices are now central to the telecommunications and information industries and electron microscopy is essential for the advancement of the complex materials and structures in these devices. It is difficult, for example, to imagine the fabrication of quantum well devices without high resolution TEM.

5.7.2 Past and Current Perspectives

The charter of the DOE labs has not naturally encompassed the study of electronic and photonic materials. This posture is reflected in the EMC/ANL and SHaRE/ORNL centers where the scientific drivers have been predominantly the study of metals and

ceramics with a natural emphasis on radiation damage. It should be noted, however, that at ORNL there is outstanding STEM research on semiconductor interfaces but this effort is not part of the center activities. The scenario is different at the FS-MRL/UIUC labs where the synergy between materials science and physics has led to outstanding examples of semiconductor research. For example, the ultra-high B doping of Si by gas source MBE is at the cutting edge of Si research but electron microscopy is not central to this work. In contrast the lateral composition modulations in AlAs/InAs superlattices could only be observed by high resolution STEM as nicely demonstrated in the CMM/UIUC studies. A potentially important development from the CMM/UIUC is that of TEM speckle analysis to observe medium-range order in amorphous materials. Amorphous materials are ubiquitous in electronic and photonic devices and a new probe of order could have widespread application. The strengths of LBNL in high-resolution microscopy have produced some seminal observations of semiconductor structures. In particular the One-Ångstrom Microscope has been employed to produce atomic images of both N and Ga in GaN; a photonic and electronic material of ever increasing importance where understanding of defect structures at the atomic level is crucial.

What has been the impact of these centers on the science and development of electronic materials and concomitant processing technologies? It is fair to say that the impact on the industry has been limited, at best. The economic strength and importance of the industry and its recognition of the importance of electron microscopy has resulted in the semiconductor research labs and manufacturing plants being equipped with state of the art scanning and transmission electron microscopes. In this milieu there is little need for routine service work or collaboration with the national labs centers. While there have been some seminal observations or discoveries from the centers, their impact is diminished when one consider the totality of the scientific challenges facing the industry. The impact of the centers is much more substantial in term of collaboration with the universities that cannot afford the state-of-the-art instruments.

5.7.3 Future Perspectives

The demand for materials research in electronic and photonic materials has never been greater. The inexorable shrinkage of dimensions in Si-based microelectronics has pushed electron microscopy to the forefront as the only analytical technique capable of tackling the atomic size dimensions. The need for photonic devices is also great requiring, for example, research on atomic defect structures in compound semiconductors where, again, electron microscopy is the essential analytical tool. The key question then is what role can the centers play with the corollary that if they are not active participants they will miss some of the most important materials research of the new millennium. Industry is central to the answer. At the present, industry can afford the investment in cutting edge microscopes and the scientific expertise to run them. The next generation machines, with their improved resolution and correspondingly severe siting limitations, could conceivably be beyond even the resources of the semiconductor industry. (It has become clear that, because of the extreme requirements for mechanical and electronic stability needed to obtain sub-Ångstrom spatial resolution, the highest quality data will only be obtained from machines at the quietest sites. A recent large-scale European sub-Ångstrom TEM instrument, for example, has been sited in an isolated field). We

therefore expect that this research will be "site limited". If these machines do exceed the resources of the industry, there would be a natural role for the national labs to house and staff these machines in the centers. The proposed NTEAM machines might fit this role. In particular, the use of energy-loss spectra in the STEM mode, which can now be obtained from individual columns of atoms near, for example the Si-SiO_2 interface, the use of electron holography to map out dopant concentrations, and the use of dual-beam focussed ion and electron beams for in-situ failure analysis by atomic-resolution imaging and spectroscopy are all cutting edge techniques now available in a few specialized labs which can be expected to become essential to the semiconductor industry. The obvious analogue is the importance of the national synchrotron labs to the pharmaceutical industry and life sciences for large molecule analysis. Such research now occupies a dominant role on these machines. The next generation electron microscopes could play a similar role at the national labs for the semiconductor industry and materials research.

6.0 DISCUSSION

Electron Beam Microcharacterization is an essential tool for modern Materials Science, which is concerned with determining the relationships between structure and properties, in the sense that both of these terms have been defined in the report. In particular (and this is made clear in the body of the report) in many ways one can regard the most important element of the structure in determining the properties of materials as the nature and distribution of 'defects' of various kinds. EBMC offers the possibility of defining the materials structure, including the nature of the defects, over the full range of length scales that are relevant to the problem. A high proportion of all materials science papers contain information derived from electron beam techniques. All four of the centers we reviewed have good experienced and competent staff, good facilities, which appear to be developing, and are doing a considerable amount of good work supporting modern aspects of materials science.

The centers are all fairly small, typically having 5-8 FTE technical staff, 2 – 3 state-of-the-art instruments, and 3 – 4 'core' instruments. The level of support staff is uneven, but generally less than we believe it should be. Funding is of the order of $2M per annum. The special skills of the centers are different, as indeed are their state-of-the-art instruments. From the point of view of OBES, they could be regarded as an "extended center", which would make clear the value of the total contribution, in the sense that the catalogue of instruments and skills would be seen as more comprehensive and substantial. However, we did not discuss this concept with them, and they are certainly not managed in this way at the moment.

There are a number of 'leading edge' instruments, including:

- HVEM/IVEM Tandem (EMC/ANL)
- OÅM High resolution electron microscope (NCEM/LBNL)
- SPLEEM (NCEM/LBNL)
- 400kV TEM with environmental cell (CMM/UIUC)
- LEEM with MBE (CMM/UIUC)
- VG FIM 100 energy-compensated APFIM (SHaRE/ORNL)
- Kindbrisk ECOPoSAP (SHaRE/ORNL)

There are plans for the addition of other leading-edge instruments as funding becomes available, including

- A 200 or 300kV FEG-TEM/STEM with EDXS, energy-filtered imaging and diffraction, high-angle annular dark-field detector, and holography capabilities (EMC/ANL)
- ARM III, a High-Voltage High Resolution TEM, with considerable capabilities (NCEM/LBNL)
- LV-EPMA (Low-Voltage Electron Probe Microanalyzer) including a bolometer EDS detector with better than 5eV energy resolution (SHaRE/ORNL)
- SAP/LEAP (SHaRE/ORNL)

The panel strongly supports these additional facilities. With them, and the addition of appropriate staff and support, the value to the materials community of the centers would become much clearer, providing an impetus for expansion of the user base.

The majority of important advances, however, (except for HREM itself) have arisen over the last three decades as a result of additional capabilities that can be added to an existing instrument. A few examples: EDS, EELS, PEELS, Imaging energy filters, ALCHEMI, Cathodoluminescence in STEM at helium temperatures, the CCD camera for TEM, Video recording, Field-Emission Guns (FEG), Bolometers, Nanodiffraction using a FEG, Z-contrast detectors, LaB_6 sources, and EBSD (there are several other examples). It is striking that most of these (including FEG, EELS, Video, CCD and Nanodiffraction) resulted from University-based research, with a few from national labs and companies (notably EDS). The importance of adequate funding for instrument development, which has recently become very difficult to obtain, is therefore clear. It is important that the centers are able to adopt and develop innovations of this kind in a timely manner; it is clear that they are at least as important as new major instruments, particularly from a user's point of view.

Specimen preparation techniques are of critical importance in EBMC. This often does not receive the attention – or publicity – of the development of a new detector or imaging system, but you can't do good science with poorly-prepared specimens no matter how sophisticated the instrument. Specimen preparation techniques in the centers should be at least state-of-the-art, but (particularly in the case of high-resolution studies) it should be leading edge. Staff specializing in specimen preparation is also of critical importance.

The panel felt that both from the point of view of equipment and of staff, the centers need additional help in this area. In particular, staff in the specimen preparation area have left and not been replaced. Other expert staff in this area will soon retire, and it is not clear if the budgets will support a replacement. This is a very serious issue, and should be addressed.

One of the centers remarked that a state-of-the-art instrument can remain such with good maintenance and appropriate addition of equipment upgrades for ten years. Some of our panel thought that this was perhaps optimistic, although clearly there examples where this is true. After this time, they have a further life as a 'core' instrument. Good maintenance is absolutely critical, and for new instruments, this is best done by a service contract with the manufacturer, backed up by highly-skilled maintenance staff resident in the centers.

Care and maintenance of the high-level technical staff is at least as important as that of the physical equipment! It is vital to have people combining a high level of technical competence, and skill in supporting users even if there is no collaborative element in the support. Maintaining technical competence, in the panel's view, requires active involvement in individual research, and staff time must be made available for this. It is obviously best, from the point of view of maximizing the capability of the center if most of this research uses the center equipment, and addresses those areas in which the center specializes, and which are relevant to the DOE mission; but it is also beneficial in the long run if the staff member has the possibility of working with leading-edge equipment which is not yet part of the center. This ability to perform individual research will be very important if good staff members are to be hired into the centers. It appeared that in the centers the staff were spending perhaps 40% of their time on individual research, and this did not seem to the panel to be out of line. Planning requires the recognition of the need for adding new staff as the capabilities change, and replacing retiring staff. For some of the centers, this is a current issue, and arises at a time when the staffing pressures on the laboratories are considerable.

All of the centers are presently recovering to a greater or lesser degree from gaps in their development in the past, as a result of renewed support from OBES. If they are to be vital user centers in the future, some long range planning is required to give some assurance that they will be able to continue to achieve a high level of up-to-date capability. Clearly the centers themselves need to have some long-range plans, and it appeared to us that their planning was largely incremental in character. In particular, they did not appear to have plans relating to the expansion of their user bases, for example. However, it also seemed to us that OBES also needs to have a plan concerning the future roles and development of the centers, and this too was not apparent. In the absence of such a plan, and in view of the limited budgets and in one or two cases a lack of a firm commitment for their futures, the limited long-range planning in the centers is scarcely surprising. This planning must relate also to the wider field of the role of DOE in the future developments of EBMC techniques in the U.S., and the part that the centers may be asked to play in this.

Three of the four centers were originally formed to support the Materials Research entities in their host sites, and a significant amount of their activities continues to be of this character. The panel did not regard this as unexpected, and also felt generally that a high proportion of activity of this kind was appropriate. The exception to this is the NCEM at LBNL, which was established principally to conduct research into the development of high voltage high resolution electron microscopy. Again, this historical role continues to be clearly evident. Some of the panel considered that more effort to become involved with the neighboring materials departments at LBNL and the University of California in Berkeley might be helpful.

The users of the EBMCC are different in character to those in, for example, the synchrotron radiation light sources. There, research teams are formed that contract for a beam line – the CATs – who perform their own research, with the role of the facility being, by and large, to provide the beams. The scientific staff of the facility have access to the source to perform their own research, and of course in some cases may collaborate with a team; but by and large the CATs are independent. For this reason, there is a very careful assessment of proposals by prospective users to conduct research at these large facilities. At the EBMCCs the collaborative aspect of the research is much more evident, because of course a state-of-the-art microscope, for example is one of a kind, and has to be shared by the external researchers using it and by the staff of the center performing their personal research. One of the centers in their statements to us distinguished between assistance to external users as being either collaborative, in which case the papers published would have a center co-author; or supportive, in which case they would not. For the reasons given above, most of the users come from the historically-supported materials departments, and much of this work is collaborative in character. Again, the panel did not see this as necessarily a fault; although if the very large part of the work done was either by the staff or the local materials staff in a collaborative mode, one might have to reexamine the concept of a user center. It appeared that the off-site users probably represented perhaps 10% of the total, although this varied from center to center, and depended a little on definitions.

One of the concerns was the very low usage by industry. In part, this is because of the interest in industry in conducting research that is proprietary; although mechanisms exist in the National Laboratories for such work to be conducted on a full cost recovery basis, and these costs are not large enough to put off most industrial researchers. In part it may be because up to now an industry interested in microcharacterization was concerned with length scales that can be handled by relatively unsophisticated instruments, which are relatively inexpensive and require little in the way of specialist support. As a result, it made more sense to purchase the instruments themselves, or use the facilities of local vendors or universities. With the development of challenges at much smaller length scales, as in modern integrated devices, MEMs, and nanostructural materials applications, it might be expected that the interest of industry in the facilities available at the centers would have increased. Although in the literature supplied by NCEM there was a list of 20 industrial interactions largely with Silicon Valley enterprises, we saw little evidence in our visit or in any other literature supplied to us of strong industrial interactions. We

found this surprising and believe that all the centers should spend some time in researching the needs of industry in the area of EBMC to develop this market segment.

Without exception, the users we spoke to at the four centers spoke very highly of their experience; of the support they received from all levels of the staff, and of the quality of the instruments that they worked with. Their only adverse comments related to financial issues: the problems associated with travelling to the centers, and the cost of accommodation close by. The panel believes that this issue does need to be addressed.

Concerning the contributions of the centers to advances in materials science and technology the panel believes that the contributions have been valuable and of good quality, but they were less convinced that any of the major recent advances in materials science have emerged from them. There are some reasons for this related to the support of DOE/OBES for electron beam microcharacterization techniques. The centers by no means represent all of the Office's support, and in particular a number of leading edge instruments have been located with specialist researchers or research teams with no obligations to support users. In one or two cases, these have been in the National Laboratories which also house the user centers. In addition, there are other groupings of EBMC capabilities in other National Laboratories and Universities, again with no responsibility to support users. Under these circumstances, it is scarcely surprising that major advances have frequently taken place elsewhere. Nevertheless, there are some unique contributions:

- The ability of the HVEM/IVEM-Tandem at EMC to observe the effects of energetic ion impacts;
- The lattice imaging capabilities of the OÅM at NCEM;
- The capabilities of the SHaRE Atom Probes to show the compositional variations in structural materials at a very fine scale.

Since these build on the unique instruments at these locations, they can be expected to continue.

We were asked about the interaction between the electron beam techniques and those using neutrons or photons, and in particular about interactions with the DOE/OBES centers in these techniques. It is easy to catalogue the complementary nature of the information developed by the techniques, and we have done that in the body of this report. However, we saw very little sign of any actual interactive research of this type being conducted in the EBMC centers. The example most often quoted is the information developed by the three different techniques relating to the high T_c perovskite superconductors, but at the moment these are more parallel efforts than interactive research.

In addition to the equipment planned for by the centers for the near future which is described above, we looked at two more innovative developments. The first of these is the Materials Microcharacterization Collaboratory which is a development to allow researchers to access the EBMC equipment from remote locations. This involves all the

centers, and we saw examples of some of the early developments during our tour. The panel overall welcomed this development, and believes that it will lead to an expansion in the users of the centers. It can, for example, reduce the financial barrier to participation that the users we met talked about. A cautionary note was expressed that there would be some loss in the personal contact between the users and the center experts. It will be a considerable time before this is a substantial issue, however.

The second innovative suggestion was for the development of a new microscope. We saw a preproposal document relating to this, which is called the National Transmission Electron Achromatic Microscope, NTEAM. The general idea is to improve the optics of the microscope, and in particular to reduce both the spherical aberration and the chromatic aberration. It is generally believed that this can be done; however the tradeoffs between intensity loss and contrast gain by monochromation and intensity and contrast gain by aberration correction have yet to be studied. The optimum arrangement of monochromator, corrector and imaging filter has also yet to be decided for TEM/STEM/Microdiffraction configurations. A major benefit of improving the optics is that the working distance of the lenses can be increased, so the accessible volume around the specimen will be much greater. This will allow many in situ experiments to be performed at high resolution, for example. We did not review this preproposal in great detail, largely because it arrived too late for us to consider it in relation to the BESAC charge. However, we believe that it correctly points to the next major advance in high-resolution electron microscopy, and if it were to be produced in the U.S. it would do a lot to reestablish U.S. leadership in this research field. We recommend that OBES gives favorable consideration to the development of an instrument of this type for the near future.

Finally, we were asked to contrast and compare the four centers. There were considerable differences, but this is to be expected, and is desirable for the reasons discussed above. Very briefly: NCEM is primarily concerned with high-resolution electron microscopy; SHaRE is primarily concerned with compositional characterization at the highest possible resolution in structural materials; EMC is really in two parts: the HVEM/IVEM-Tandem, which is the User Center so far as ANL is concerned, and the balance of the ECM which primarily supports the materials research at ANL; and MMC, which has been traditionally associated with *in situ* environmental microscopy and support of the materials research in the Frederick Seitz Materials Research Laboratory.

The panel felt that the MMC was so different to the other three centers that it was very difficult to assess it on the same criteria. It is an integral fully embedded part of the FS-MRL, and it would make much more sense to assess the MRL as a whole for its contribution to materials science and the contributions of the MMC in this context. There is a considerable tradition of very good work there, and the contributions to understanding the effects of hydrogen on cracking (for example) are well-known to all materials scientists. The role that they play in the training of electron microscopists is also important and valuable to the community. However, the users of the MMC were nearly all derived from UIUC one way or another.

The research at NCEM was believed by the members of the panel with skills in electron microscopy to be the best among the centers. The recovery from the low point of a few years ago is well under way, and we all felt that it was a vigorous and motivated group. The hoped-for addition of the ARM-III will cap their reestablishment as leaders in the HREM field, certainly in the U.S. We were also impressed with the development of the SPLEEM. The materials scientists in the panel felt that the links of the excellent work with the development of materials science was less clear, and urged closer links with the materials science community, specifically those in the LBNL itself, and in the University of California. The panel as a whole was surprised at the relatively limited contacts with the industries of Silicon Valley evidenced in the material we were shown, and suggested that outreach to potential users needed to be improved.

The situation at EMC was less clear to the panel. The HVEM/IVEM-Tandem facility continues to be a unique capability, and is designated by ANL as a user facility. The need for this kind of research will increase in the future because of the increasing concern about the storage of radioactive wastes, and the need for information on the behavior of containment materials over very long times. In addition, the development of first wall materials for fusion energy systems continues to be a major thrust for DOE, and for ANL. The HVEM is old, and the control electronics are deteriorating; by modern standards the resolution is not very good. However, it is still capable of showing effects in relatively thick films, which is very important in this context. The panel believes that this facility should certainly continue to be supported. The staffing level is critically low. The center state that “the remaining microscopes are primarily (though not exclusively) associated with use by the Materials Science Division and other scientific divisions at Argonne”. Clearly, the EMC is entering a transition phase, and the panel welcomes the signs of a redirection. They believe that the program should be looked at again in perhaps two years to see how the changes are working out.

SHaRE is, in some ways, the opposite end of the spectrum from NCEM, in that its primary concern is with the support of materials science. Its research is directed at the problems of real structural materials, and particularly those for which ORNL has a strong background. Their special area of expertise is in compositional characterization at very high resolution, and they have a suite of world-leading instruments, particularly the atom probes, to enable this. The involvement of the senior laboratory management, and the professionalism of their potential customer interaction procedures, impressed the panel as the best of all the centers. We strongly support their request for additional leading-edge instrumentation. However, as with all the centers, the industrial involvement is much less than one would hope, and in this case expect. The users are from other ORNL Divisions, the associated Universities, and other National Laboratories. They operate mainly in a collaborative mode at the moment, which is scarcely surprising considering the unique instrumentation.

Overall, the panel believes that the EBMCC need to be looking much more actively at the possibilities for application of their capabilities in the rapidly developing area of nanotechnology, including the new developments in microelectronics. This should certainly not replace the traditional areas of materials science for them, but it does appear to open an area for which EBMC techniques are uniquely suited, and where, because of the expense and

sophistication of the techniques, the developing industries will be more prepared to look to federal user facilities for help.

Electron Beam Microcharacterization is a vital tool in the development of materials science. The Office of Basic Energy Sciences has recognized this for many years, and has been (and continues to be) a major supporter in the development of these techniques. The identification of the four User Centers represented a further step in the evolution of their portfolio in this area, and the panel feels that this has been generally successful. With some of the developments we have discussed above and in the body of the report, we feel that the centers can continue to expand their role in supporting the general materials research community, offering access to advanced instruments and technical support beyond the capabilities of individual materials research departments. The rapid expansion of the interest in engineering and science at the nanotechnology level makes this a very important opportunity, and not one that the nation can afford to miss.

7.0 CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusions

Electron Beam Microcharacterization is a tool whose importance to modern Materials Science and Engineering is very great.

It has been said that “a material is a substance which can serve some useful purpose”, and that includes gases, liquids, and solids: but the large part of materials science is concerned with solid materials. A material is useful because it has properties which enable it to perform some function. These properties include mechanical properties, such as strength, ductility, rigidity, toughness, hardness; physical properties, such as electrical conductivity, thermal conductivity, magnetic properties; chemical properties, such as corrosion resistance, reactivity, stability; and surface properties, such as catalytic activity, friction, wear resistance, and so forth. Often, the material’s usefulness will depend on a combination of several properties. A material may be a single crystal of a very pure element. More often it is polycrystalline, composed of an assembly of individual crystals, usually called grains, bonded together with boundaries between them. The strength of the bonding is a further property. Again, this may be a pure elementary material. It can also be chemical compound, or a metallic alloy of different elements. In these cases, there may be a single phase present, or a physical mixture of two or more phases. The properties of the boundaries between different phases (‘heterophase’ boundaries my be important in determining the properties of the overall material. Within the individual phases, or in the boundaries between two grains of the same phase or heterophase boundaries there may be local variations in chemical composition, or departures from regularity in the crystal array: missing atoms (‘vacancies’) additional atoms interpolated within the structure (‘interstitials’) or, in compounds, atoms on the wrong sublattice. These are point defects. One may have line defects such as dislocations, or planar defects, such as stacking faults. The material may be wholly or partly amorphous, with phases that do not have a regular crystal structure: glasses are

commonly of this type. All of these aspects (and many more of a similar kind) together are referred to as the structure of the material.

The basic aim of materials science is to discover the relationships between the properties of a material, since this is what we want; and the structure of a material. This is because the structure of a material is something over which we have at least the possibility of control.

Electron beam microcharacterization offers us the most powerful tools for measuring the structure. It can be seen that the scale over which we are concerned ranges from the macroscopic, as in the grains in a large steel casting, or the fibers in wood, or the aggregate in concrete, to the microscopic, as in the size and shape of grains, or the geometrical aspects of phase distributions, to the submicroscopic, as in dislocation structures in solids, or chemical segregation of species to phase boundaries. Optical inspection, including optical microscopes, can provide information at the larger size end of this distribution; but the electron beam techniques can extend this down to the atomic scale. In addition, in the same machine one can derive crystallographic information from individual phases and chemical compositions again to the limit of identifying individual atoms. No other technique is capable of providing this information.

The importance of electron beam microcharacterization is well-recognized by the Office of Basic Energy Sciences, and the Office has supported the development and application of the techniques for many years. One can distinguish three levels for this.

(1) The provision of EBMC instruments to individual university materials science and engineering departments. It is difficult to imagine a modern materials department without at least one transmission electron microscope and a couple of scanning electron microscopes with analytical capabilities.
(2) The provision of advanced instruments to individuals or research groups who wish to either develop a new instrument capable of doing new science, or apply a very advanced tool to the solution of a specific problem.
(3) The provision of a group of instruments and specialist staff to provide capabilities for researchers who are either determining whether an advanced instrument can indeed offer the possibility of addressing their problem (in which case they will attempt to buy one for their own laboratory) or wishing to use an advanced instrument to study one aspect of a problem which they are otherwise addressing by different techniques.

It is this third aspect which the User Centers are intended to fulfill; and this gives us a set of criteria to judge them against.

This is a dynamic situation. For example, in the earlier stages of microelectronics, the length scales were accessible using relatively simple microscopes, which were not very expensive and were relatively easy to maintain and to operate. A company might well choose to purchase one for its own research, or it could arrange for help from a local consultant or university. However, the advanced devices now being produced are at a length scale inaccessible to those instruments. The more advanced instruments are too

expensive, both to purchase and maintain, and require specialist staff which it would be difficult to justify. It is logical to suppose that these industries would now find a National Center attractive.

It follows that to be successful, the centers must have as complete a range of advanced instruments as possible. We distinguish three levels of instruments:

- Leading edge instruments, which are those which generally are used by the second group above. Generally, it is difficult to imagine that they would be appropriate for the users of a center of the sort considered here, but as experience is developed and operational difficulties solved, they may eventually be suitable for this purpose.
- State-of-the-art instruments. Typically, these are the highest level of instruments that can be purchased from a manufacturer, and can be supported by service contracts.
- Core instruments, which are those that are appropriate for general users: the top end of instruments in Materials Science departments in Universities for general graduate student use will be at this level.

On this basis, the 'advanced instruments' above will be state-of-the-art; and these will be backed up by core instruments which will be available to users for preliminary examinations of specimens, and for training purposes.

It is very important that all instruments at the centers should be maintained to the highest level of reliability and performance. For the state-of-the-art instruments, the panel believes that this means they should all be covered by service contracts from the suppliers; but it is also necessary to have a top-class team of technical staff resident in the center.

The major advances in capabilities over the last three decades (with the exception of High Resolution Electron Microscopes) have been achieved by additional capabilities that can be added to an existing instrument, mostly detectors or imaging systems. Examples of these are given in the report. The panel sees the provision of funding for this type of upgrade as a very high priority, which will extend the life of existing instruments as state-of-the-art.

Nevertheless, the lifetime of an instrument is finite, and for a center to remain effective the acquisition of new state-of-the-art instruments in a timely fashion must be planned. It is evident that this has not been done in the past, as our report makes clear. The panel recommends that the centers develop long-range plans for the maintenance of their capabilities, and that OBES should also have a plan for the centers, to the extent that this is possible.

Specimen preparation is a critical issue. A state-of-the-art instrument must have state-of-the-art specimens; and for high resolution studies it should be even better than this. The panel believes that at the moment the centers fall short of this standard, in part because of

equipment deficiencies, but largely because skilled support staff are retiring and not being replaced. The panel strongly recommends that this issue must be addressed by the centers, and by the establishments within which they are located.

Having first class instruments in a center means nothing if the high level technical staff are not also first class. A particular type of individual is required by a user center of this class, because he or she must at once be at the top of the profession, but at the same time prepared to contribute significant time to the support and training of users. There are excellent people in the existing centers at the moment who do indeed combine these abilities, but a number of them are approaching the ends of their distinguished careers. The panel recommends that plans for their eventual successors are developed in good time. It is important to remember that appointments have to be made well before individuals retire, to permit the transfer of knowledge.

To appoint a first-class person is not enough. It must be possible for them to maintain and develop their own skills while in service, and this means that the ability to perform individual research at the highest level must be provided, and this includes research directed toward developing the techniques of electron beam microcharacterization. We note that a similar point was made in the 1987 report from the Council on Material Science.

It is not necessary that the state-of-the-art instruments are duplicated at the centers. In fact, this would be an inefficient method of operation. In the Charge to our panel this is embedded in the question regarding the Visions of the centers, but this is only effective if there is a high level interactive planning between the centers which includes a consideration of the distribution of capabilities.

There has been a significant advance in the equipment available in the centers over the last few years, and this is encouraging. It is, however, uneven; and there are still very significant gaps arising from inadequate funding for new instrumentation in the past. It is also clear that several formerly leading-edge instruments are reaching the end of their lives. The centers are relatively small: the annual budget is typically between $2M and $2.5M, of which approximately half is for staffing. The panel felt that the staffing at all levels was less than would be required for the most effective operation, and rough estimates of the number of instruments required and a steady-state upgrading and replacement policy suggest that some increase in funding for the centers would be very effective. However, this point really depends on the results of the development of a proper plan, which was outside our charge.

It will be clear from the above material that the panel believes, and recommends that plans for the operation and development of the centers are essential, and that the planning must involve all the centers and the Office of Basic Energy Sciences. It is probable that involving external advisors familiar with the field would also be desirable. This planning must relate also to the wider field of the role of DOE in the future developments of EBMC techniques in the U.S., and the part that the centers may be asked to play in this.

We were presented with some proposals for additional instrumentation in the immediate future. These were:

- A 200 or 300kV FEG-TEM/STEM with EDXS, energy-filtered imaging and diffraction, high-angle annular dark-field detector, and holography capabilities (EMC/ANL)
- ARM III, a High-Voltage High Resolution TEM, with considerable capabilities (NCEM/LBNL)
- LV-EPMA (Low-Voltage Electron Probe Microanalyzer) including a bolometer EDS detector with better than 5eV energy resolution (SHaRE/ORNL)
- SAP/LEAP (SHaRE/ORNL)

The panel strongly recommends support for these additional facilities. With them, and the addition of appropriate staff and support, the value to the materials community of the centers would become much clearer, providing an impetus for expansion of the user base.

There is a good involvement of users of the facilities. However, the majority of the current users are from materials departments in the host sites. There are good historical reasons for this, but the justification for centers of the sort that are described above must involve making the facilities more available to a wider group of users from the national materials science researchers. SHaRE have a very positive program to make the availability of their facilities known to University departments. The panel recommends that all the centers make similar efforts.

The panel was impressed by the strong industrial interactions that have developed through the SHaRE program. While these interactions are pervasive, the quality of the science is high, the research is fundamental, and the outcome of the work has been far reaching. However, in general the panel noted the low usage of the centers by U.S. industry. The increase in the importance of nanotechnologies would appear to present an opportunity for advanced EBMC techniques, as has been argued in this report. Although in the literature supplied by NCEM there was a list of 20 industrial interactions largely with Silicon Valley enterprises, we saw little evidence in our visit or in any other literature supplied to us of strong industrial interactions. We found this surprising and recommend that all the centers should make a positive effort to determine the needs of industry in this area, and develop a strategy for expanding this part of the user base.

The users we met were all very positive about their experience at the centers. The panel welcome this, and congratulate the centers on their excellent efforts. The only problem that was brought to our attention was the travel and accommodation costs to research students of using the centers. The panel recommends that OBES discusses with the centers ways of addressing this issue.

We were asked to compare the contributions of the four facilities. We were very impressed by SHaRE; it appeared to be doing extremely good work, addressing materials science issues, and generally behaving as we expected a DOE/OBES User Center should. It was also clear to us that it had very complete support from the senior management at

ORNL. We were also impressed with NCEM; it was doing good science, and had a clear vision for its future and its new instrumental needs. It also had very good support from the Laboratory management. Although they showed us a very supportive set of letters from materials scientists in the adjacent departments, our materials scientists felt that their involvement with materials science could be strengthened; their involvement with the nanotechnology industries of Silicon Valley were less than we would have expected. ECM presented some problems for us. The HVEM/IVEM-Tandem User Facility continues to satisfy all our criteria: the staffing issues need to be considered as a matter of urgency. The remaining part of the center clearly stated that their principal objective was to support the ANL materials programs; and their equipment needs urgent upgrading if they are to be truly a user center. However, it is clear to us that the management at ANL recognize these issues, and are committed to addressing them; we support their current plans, and we recommend that their progress is reviewed in two years time.

MMC presented us with a problem. They are embedded in the Frederick Seitz Materials Research Laboratory, and their users are almost entirely connected with UIUC. They are well equipped, and the staff members are enthusiastic and talented. In particular, they have a remarkable role in the training of electron microscopists. We believe that they are doing an excellent job; but it is not what we expect from a DOE/OBES users center, and it is very different from the other three centers. Our view was that they should be reviewed as a component of the MRL. We recommend that OBES studies the role of MMC within the mix of EBMC user centers, to see whether it satisfies their requirements for this role. However, we support their continued funding as an EBMC within their present context.

Finally, we addressed two proposals for innovative initiatives. The first of these is the Materials Microcharacterization Collaboratory which is a development to allow researchers to access the EBMC equipment from remote locations. This involves all the centers, and we saw examples of some of the early developments during our tour. The panel overall welcomed this development, and believes that it will lead to an expansion in the users of the centers. It can, for example, reduce the financial barrier to participation that the users we met talked about. We also welcomed it as a clear sign of the centers' collaboration. A cautionary note was expressed that there would be some loss in the personal contact between the users and the center experts. It will be a considerable time before this is a substantial issue, however. The panel recommends that this experiment is continued.

The second innovative proposal is the National Transmission Electron Achromatic Microscope, NTEAM. We did not receive this proposal in time to review it, but it was discussed informally during our meetings. It is our opinion that it offers an accurate view of the direction for the next major development in electron microscopy, and we recommend that OBES gives favorable consideration to the development of an instrument of this type for the near future. This will involve creating a review committee drawn from the electron microscope community in the U.S. to assess the proposal, and to discuss the role that the EBMCC might play in this development. However, we are

anxious that involvement with this development should not deflect their interest from the user functions we have discussed in this report.

7.2 Recommendations

(1) The panel believes that the concept of Electron Beam Microcharacterization User centers is very valuable to the Materials Science community, and strongly recommends that funding for them should continue to be a high priority.

(2) The panel recommends that plans for the operation and development of the centers are essential, and that the planning must involve all the centers and the Office of Basic Energy Sciences. It is probable that involving external advisors familiar with the field would also be desirable. This planning must relate also to the wider field of the role of DOE in the future developments of EBMC techniques in the U.S., and the part that the centers may be asked to play in this.

(3) The panel recommends that the centers develop long-range plans for the maintenance of their capabilities, and that OBES should also have a plan for the centers, to the extent that this is possible.

(4) Having first class instruments in a center means nothing if the high level technical staff are not also first class. There are excellent people in the existing centers at the moment, but a number of them are approaching the ends of their distinguished careers. The panel recommends that plans for their eventual successors are developed in good time. It is important to remember that appointments have to be made well before individuals retire, to permit the transfer of knowledge.

(5) The panel strongly recommends that the critical issue of specimen preparation must be addressed by the centers, and by the establishments within which they are located. The panel believes that at the moment the centers fall short of the standards required, in part because of equipment deficiencies, but largely because skilled support staff are retiring and not being replaced.

(6) The panel believes that the levels of equipment and staffing in the centers are somewhat low, and in connection with the planning recommended above, we recommend that the appropriate size and funding levels appropriate for the centers should be carefully reviewed.

(7) The panel strongly recommends support for the additional facilities listed below. With them, and the addition of appropriate staff and support, the value to the materials community of the centers would become much clearer, providing an impetus for expansion of the user base.

- A 200 or 300kV FEG-TEM/STEM with EDXS, energy-filtered imaging and diffraction, high-angle annular dark-field detector, and holography capabilities (EMC/ANL)

- ARM III, a High-Voltage High Resolution TEM, with considerable capabilities (NCEM/LBNL)
- LV-EPMA (Low-Voltage Electron Probe Microanalyzer) including a bolometer EDS detector with better than 5eV energy resolution (SHaRE/ORNL)
- SAP/LEAP (Scanning Atom Probe/Local Electrode Atom Probe) (SHaRE/ORNL)

(8) The panel recommends that all the centers make similar efforts to those that SHaRE has undertaken to make the availability of their facilities known to University departments.

(9) The panel recommends that the centers make a positive effort to determine the needs of industry in the area of nanotechnologies, since this would appear to present an opportunity for the application of advanced EBMC techniques, and develop a strategy for expanding this part of the user base

(10) The panel recommends that OBES discusses with the centers ways of addressing the issue of the travel and accomodation costs for research students using the centers.

(11) EMC has issues concerning renewal of infrastructure and personnel that concern us (see below). However, it is clear to us that the management at ANL recognizes these issues, and is committed to addressing them; we recommend that ANL's efforts to solve these problems should be supported, with a review of progress in three years time.

(12) CMM also presented us with a problem, which is also described above. We recommend that OBES studies the role of CMM within the mix of EBMC user centers, to see whether it satisfies their requirements for this role. However, we support their continued funding as an EBMC within their present context.

(13) The panel recommends that the Materials Microcharacterization Collaboratory experiment is continued. The panel overall welcomed this development, and believes that it will lead to an expansion in the users of the centers. It can, for example, reduce the financial barrier to participation that the users we met talked about. We also welcomed it as a clear sign of the centers' collaboration. A cautionary note was expressed that there would be some loss in the personal contact between the users and the center experts.

(14) The panel recommends that OBES gives favorable consideration to the development of an instrument similar to that described in the National Transmission Electron Achromatic Microscope (NTEAM) preproposal. It is our opinion that this preproposal (which we were unable to discuss in depth) offers an accurate view of the direction for the next major development in electron microscopy. We suggest that consideration of this will involve creating a review committee drawn from the electron microscope community in the U.S. to assess

the proposal, and to discuss the role that the EBMCCs might play in the development. However, we are anxious that involvement with this should not deflect their interest from the user functions we have discussed in this report.

APPENDIX A: Charge from DOE to BESAC

APPENDIX B: Charge from BESAC to Subpanel

Charge Letter to John Stringer
From Professor Geraldine Richmond, Chair
Basic Energy Sciences Advisory Committee

Dear John,
The Basic Energy Sciences Advisory Committee has been asked by Dr. Martha Krebs to help in assessing the scientific impact of and the Nation's need for the electron beam microcharacterization centers operated by the Basic Energy Sciences program. To this end she has asked us to assemble an expert balanced panel to present a report to BESAC at its summer meeting in 1999, and I am delighted that you have accepted the task of convening and chairing it.
The four centers to be considered in this review are the Electron Microscopy Center for Materials Research at Argonne National Laboratory; the National Center for Electron Microscopy at Lawrence Berkeley National Laboratory; the Center for Microanalysis of Materials at the University of Illinois Frederick Seitz Materials Research Laboratory; and the Shared Research Equipment Program at Oak Ridge National Laboratory. As part of the panel's work, it would be desirable to visit each of the four centers, and meet with the members of the management, staff, and user communities. Prior to those visits, it would be desirable to convene a meeting at which each of the centers could present an overview of their individual contributions to the panel.
We would specifically like the panel to address the following issues and questions:

1. What has been the scientific and technological impact of the microcharacterization centers during the past decade, and what is it expected to be during the coming decade? In particular, what scientific studies are enabled by the centers that could not otherwise be done?
2. What are the user groups served by each of the centers? How do they differ? What is the user demand at each of the centers, and how is it expected to change?
3. What special needs do each of the centers serve, and how do the centers complement one another?
4. What is the vision of each center? Are the visions appropriate? How do the visions complement one another? Is there anything missing in the set of visions for the future?
5. How does the use of electron beams for characterizing materials complement the use of photons and neutrons?
6. What are the opportunities for improving the techniques to maintain the facilities at the forefront?

The centers differ from Basic Energy Sciences major user facilities such as the synchrotron radiation light sources or the neutron sources in that they do not have distinct "operating budgets"; they are supported as part the Materials Science Division research budget. Furthermore, each of them can be regarded as a suite of instruments aimed at using electron beams to characterize materials with high resolution, both structurally and chemically. The fifth charge above addresses the ways in which the information that can

be developed using electron beam instruments complements that that can be derived using the photon or neutron beams available at the major user facilities.

The electron beam microcharacterization centers have a large user base, and the combination of their suites of leading-edge instruments and the highly-talented scientific staff available to the users makes them of considerable value to the study of the structure and behavior of materials. Recent improvements in techniques, and in particular the ability to characterize materials at a resolution approaching 0.1 nm, can be expected to increase this value still further in the near future. In this context, it is important that your panel assesses the degree to which the user community at each center is being served.

The differences in the aims and objectives of these four centers are probably greater than is the case for the four synchrotron light centers, for example; and I suggest that your panel take this into account in your assessment.

It is probably best for your panel to be balanced between members familiar with the electron beam microcharacterization techniques and members familiar with the scientific areas that the centers support or enable.

Once again, I am grateful for your help, and I look forward to your panel's report.

Sincerely,

Geraldine Richmond
Chair, Basic Energy Sciences Advisory Committee

APPENDIX C: Panel Membership List

INITIAL MEETING OF E-BEAM SUBPANEL
August 13, 1999

Gaithersburg Marriott Washingtonian Center

7:30am	Continental Breakfast
8:30-8:45	Welcome and Introductions John Stringer
8:45-9:15	Panel Charge and proposed organization John Stringer; Geraldine Richmond (BESAC Chair); Patricia Dehmer (OBES Associate Director); Iran Thomas (OBES Deputy Associate Director; Director of Materials Science Division).
9:15-10:00	"Role of TEM Microcharacterization to Materials Science" Manfred Rühle (Max Planck Institute, Stuttgart).
10:00-10:45	"Electron Beam Microcharacterization Facilities: Opportunities and Needs" J. Murray Gibson (ANL MSD Division Director).
10:45-11:00	Coffee Break
11:00-11:30	"The Role of Microanalysis in Phase Transformations and Interface Science" James Bentley (ORNL)
11:30-12:00	"Atomic Resolution Imaging of Defects in Interfaces and Nanostructures" Ulrich Dahmen (LBNL)
12:00-1:00	Lunch (The Panel will take a private working lunch to discuss the morning's presentations).
1:00-1:30	"In-Situ Experiments in the Electron Microscope" Mark Kirk (ANL)
1:30-2:00	"Thin Films and Surface Science" Ivan Petrov (UIUC
2:00-2:15	"The Materials Microcharacterization Collaboratory: Web-Based Access to the Centers" Michael O'Keefe (LBNL)
2:15-2:45	"Outlook and Future Research Challenges" Ulrich Dahmen (LBNL)
2:45-3:00	"Opportunities for Future Instrumentation and Technique Development" James Bentley (ORNL).
3:00-3:30	Questions and comments from the Panel on the Center presentations.
3:30 – 4:30	Private Panel discussions on the day's presentations and the implications. Identification of additional material (if any) to be supplied to the Panel by the Centers or by OBES prior to the site visits. Preliminary discussion of the structure of the final Panel report and assignment of section preparation responsibilities. Dates for the site visits.
4:30	Adjourn.

APPENDIX E: Agenda of the 12/6-10/99 Review of Centers

APPENDIX F:

"Electron- Beam Microcharacterization Centers: A National Resource" (June 1999)

APPENDIX G:

"Contributions, Challenges, and Opportunities in the Core Scientific Fields of the Four Electron Beam Microcharacterization Centers" (August 1999)

EQUIPMENT LIST (Instruments and Key Features)

Electron Microscopy Center for Materials Research
Argonne National Laboratory

INSTRUMENTS	KEY FEATURES
High-Voltage Electron Microscope Kratos/AEI EM7 (1.2MeV)	Resolution 0.9 nm pt-pt Continuous voltage selection Current density 15A/cm^2 High-vacuum specimen chamber Electron and ion dosimetry systems Video recording system Ion-beam interface Specimen stages 10-1300 K Straining and environmental stages
Transmission Electron Microscope Hitachi H-9000 NAR (300keV)	Resolution 0.25 nm pt-pt Ion-beam interface Specimen holders 15-1200 K
Transmission Electron Microscope JEOL 100 CX (100keV)	Resolution 0.7 nm pt-pt Equipped with STEM, XEDS Specimen stages 85-900 K
Transmission Electron Microscope Phillips EM 420 (120keV)	Resolution 0.45 nm pt-pt Equipped with EELS, XEDS Specimen stages 30-1300 K
Transmission Electron Microscope Phillips CM 30 (300keV)	Resolution 0.25 nm pt-pt Equipped with PEELS, XEDS, video Specimen stages 30-1300 K
High Resolution Electron Microscope JEOL 4000 EX II (400keV)	Resolution 0.165 nm pt-pt Specimen stages RT
Analytical Electron Microscope VG603Z (300keV)	Resolution 0.28 nm pt-pt Ultra-high vacuum, Field Emission Gun Equipped with EELS, XEDS, AES, SIMS, LEED, etc.Specimen stages 85-1300 K

ACCELERATORS

NEC Model 2 UDHS Tandem	Terminal voltage 2 MV Energy stability ± 250 eV Current density: H^+, 10 μA/cm^2 (typical) Ni^+, 3 μA/cm^2
NEC Model 650kV Injector	Terminal voltage 650 kV Energy stability ± 60 eV Current density: He^+, 100 μA/cm^2 (typical) Ar^+, 10 μA/cm^2

Center for Microanalysis of Materials
University of Illinois at Urbana-Champaign

INSTRUMENTS	KEY FEATURES
Imaging Secondary Ion Microprobe Cameca IMS 5f (SIMS)	Dual Ion Sources (Cs^+, O_2^+) 1 μm resolution
Scanning Auger Microprobe Physical Electronics 660	Resolution: SEM 25 nm Auger 60 nm
X-ray Photoelectron Spectrometer Physical 5400 (XPS)	Resolution: 50 meV, 180^o spherical analyzer, Mg/Al and Mg/Ag anodes
X-ray Photoelectron Spectrometer Surface Science (XPS)	Spherical analyzer, small spot size, gas doping, high temperature
Transmission Electron Microscope Phillips EM 420 (120keV)	EDS, EELS, STEM, Cathodoluminescence, Stage 30 K
Transmission Electron Microscope Phillips EM400T (120keV)	EDS, Heating, cooling stages
Transmission Electron Microscope Phillips CM 12 (120keV)	EDS, STEM, Heating, cooling stages
Transmission Electron Microscope JEOL 4000 EX (400keV)	For environmental cell use. Straining stages, heating stages
Transmission Electron Microscope Hitachi 9000 (300keV)	Resolution 0.19 nm Atomic imaging

Scanning Transmission E. M. VG HB501 (100kV) (STEM)	0.5 nm probe, field emission gun EDS, EELS
Scanning Electron Microscope Hitachi S800 (SEM)	Field emission gun Resolution 2.0 nm, EDX
Scanning Electron Microscope Zeiss 960 (SEM)	Back Scattering (EBSP), EDX Cathodoluminescence, Helium stage
Scanning Tunnelling Microscope Omnicron (STM)	Variable temperature 30-1000 K Auger, gas dosing, ion cleaning
X-ray Equipment Enraf-Nonius 18kW source Elliott 14kW source Rigaku 12kW source Several conventional sources Rigaku D/Max-11B Computer-Controlled Powder Diffractometer	4-circle diffractometer Bede high-precision diffractometer 3-circle diffractometer Powder cameras, etc. High & low temperature stages Texture Analysis
Van de Graff Accelerator for RBS High Voltage Engineering 3MeV Also PIXE (Proton Induced X-ray Emission)	Rutherford Backscattering Ion irradiation & implantation
Tandem Accelerator General Ionex 1.7 MeV	
Low-Energy Electron Microscope 10 – 100eV, IBM (LEEM)	UHVsurface analysis,surface structure, Crystal growth, evaporation

National Center for Electron Microscopy
Lawrence Berkeley National Laboratory

INSTRUMENTS	KEY FEATURES
High-Voltage Electron Microscope Kratos (1.5 MeV)	Resolution 0.3 nm pt-pt Continuous voltage selection Max. Beam current 70A/cm^2 Environmental cell, hot & cold stage Straining and straining/heating stage CBED, video camera

Atomic-Resolution Electron Microscope JEOL 1-MeV	Resolution < 0.16 nm pt-pt over full voltage range. Ultra-high resolution goniometer stage ± 40^{o} biaxial tilt with height control
High-Resolution Electron Microscope	Dedicated high-resolution 0.24 nm pt-pt U.H. resolution goniometer stage only Microdiffraction, CBED, UTW
Analytical Electron Microscope JEOL 200CX	X-ray Detector, high angle X-ray detector, PEELS spectrometer
Transmission Electron Microscope JEOL 200CX	In-situ instrument with electrical biasing holder, heating stage, video camera
Transmission Electron Microscope Phillips CM200 FEG	Field emission, resolution 0.24 nm pt-pt, holography, energy filter, hot stage Video and CCD cameras
Transmission Electron Microscope Phillips CM300 FEG	Field emission, resolution 0.17 nm pt-pt, 0.1 nm information limit, holography, energy filter, Video and CCD cameras

Shared Research Equipment Program (SHaRE)
Oak Ridge National Laboratory

INSTRUMENTS	KEY FEATURES
Transmission Electron Microscope Phillips CM 12 AEM (120keV)	EDS, CBED, STEM, heating & cooling stages video camera
Transmission Electron Microscope Phillips CM 200 AEM (200keV)	EDS, CBED, (P)EELS, STEM, minimum probe ~ 1 nm heating & cooling stages spectrum imaging, video camera
Transmission Electron Microscope Phillips CM 30 AEM (300keV)	EDS, CBED, (P)EELS, STEM, energy filter, heating & cooling X

	PEELS spectrometer video camera
Scanning Electron Microscope Phillips XL 30/FEG (30 kV)	SEM, EDS (WDS), EBSP, minimum probe ~ 1.5 nm
Atom Probe Field Ion Microscopes	TOF atom probe, imaging atom Probe, FIM, pulsed. I.e. Atomic resolution imaging: single atom analysis, laser atom probe, elemental mapping
Scanning Auger Electron Spectroscopy PHI 590	200 nm beam, fracture stage, RGA, depth profiling, elemental mapping
Scanning Auger Electron Spectroscopy Varian	5 μm beam, hot-cold fracture stage, RGA, depth profiling, elemental mapping
Triple Ion Beam Accelerator Facility	400kV, 2.5 MV, 4MeV Van de Graff Accelerators. RBS, nuclear micro-analysis sputter profiling, elemental analysis
Mechanical Properties Microprobe - Nanoindenter	Computer controlled diamond indenter Resolution: 0.1 μm lateral, 0.01 nm depth. Cooling/heating capability, scratch test
Atomic Force Microscope Parl Autoprobe – XL	Optical-based position sensing, quantitative surface imaging, repulsive/attractive modes

ACRONYM LIST

Technical Acronyms

AEM	Analytic Electron Microscope
AES	Auger Electron Spectroscopy
ALCHEMI	Atom Location by Channeling-Enhanced Microanalysis
APFIM	Atom Probe Field Ion Microscope
APFIM	Atom Probe Field Ion Microscopy
ARM	Atomic Resolution Microscope
BSED	Back Scattered Electron Diffraction
CBED	Convergent Beam Electron Diffraction
CCD	Charge-Coupled Device
CL	Cathodoluminescence
CVD	Chemical Vapor Deposition
EBIC	Electron-Beam Induced Conductivity
EBSD	Electron Backscattered Diffraction
ECOPoSAP	Energy-Compensated Position-Sensitive Atom Probe
EDS	Energy Dispersive Spectroscopy
EDX or EDXS	Energy Dispersive X-ray Spectroscopy
EELS	Electron Energy Loss Spectrometry
ELNES	Energy Loss Near Edge Structure
ESCA	Electron Spectroscopy for Chemical Analysis
FEG	Field Emission Gun
FESEM	Field Emission Scanning Electron Microscope
FIM	Field Ion Microscopy
HREM	High Resolution Electron Microscopy
HRTEM	High Resolution Transmission Electron Microscopy
HVEM	High Voltage Electron Microscope
IASCC	Irradiation Assisted Stress Corrosion Cracking
IVEM	Intermediate Voltage Electron Microscope
LEED	Low Energy Electron Diffraction
LEEM	Low Energy Electron Microscope
LV-EPMA	Low-Voltage Electron Probe Microanalyzer
MBE	Molecular Beam Epitaxy
MEMS	Micro Electromechanical Systems
MOCVD	Metal Oxide Chemical Vapor Deposition
NTEAM	National Transmission Electron Achromatic Microscope
OIM	Orientational Imaging Microscopy
OPoSAP	Optical Position-Sensitive Atom Probe
PEELS	Parallel Collection Electron Energy Loss Spectrometry
PEEM	Photoemission Electron Microscopy
RBS	Rutherford Back Scattering
RHEED	Reflection High-Energy Electron Diffraction
RIS	Radiation-Induced Segregation
SAD	Selected Area Diffraction

SAP/LEAP	Scanning Atom Probe/Local Electrode Atom Probe
SEM	Scanning Electron Microscope
SIMS	Secondary Ion Mass Spectroscopy
SPLEEM	Spin Polarized Low Energy Electron Microscope
STEM	Scanning Transmission Electron Microscope
STM	Scanning Tunneling Microscopy
TAP	Tomographic Atom Probe
TEM	Transmission Electron Microscope
UHV	Ultra High Vacuum
WDS	Wavelength Dispersive X-Ray Spectroscopy
XAS	X-Ray Absorption Spectroscopy

Organizational Acronyms

ANL	Argonne National Laboratory
BES-MSD	Basic Energy Sciences - Materials Science Division
CMM	Center for the Microanalysis of Materials
EBMC	Electron Beam Microcharacterization
EBMCC	Electron Beam Microcharacterization Center
EMC	Electron Microscopy Center for Materials Research
FS-MRL	Frederick Seitz Materials Research Laboratory
LBNL	Lawrence Berkeley National Laboratory
MMC	Materials Microcharacterization Collaboratory
NCEM	National Center for Electron Microscopy
NIST	National Institute for Standards and Technology
OBES	Office of Basic Energy Sciences
ORNL	Oak Ridge National Laboratory
SFI	Scientific Facilities Initiative
SHaRE	Shared Research Equipment Program
UIUC	University of Illinois at Urbana-Champaign

Some Notes on Principles; Features; Spatial, Mass or Energy Resolutions; and Limitations of Some Important Techniques.

PEELS - parallel electron energy-loss spectroscopy. The electrons in the TEM pass through the specimen and scatter elastically and inelastically. A magnetic prism spectrometer disperses the electrons according to their energy; a YAG or phosphor scintillator coupled via fiber optics to a semiconductor photodiode array provides the parallel detection. Elemental and chemical bonding information as well as electronic structure information may be deciphered. Spatial resolution is determined by the electron probe size (can be <0.5nm) and beam spreading within the specimen (negligible in very thin specimens).

CL - cathodoluminescence. Recombination of electron-hole pairs to give off visible light. Good for semiconductors. The spectrum of light contains information about doping levels and band-gap changes. CL is done in a dedicated STEM or an SEM.

RBS - Rutherford back scattering. Incident ions [typically 1-3 MeV He+] penetrate to a significant depth into the specimen and back-scattered ions are collected both from surface and bulk scattering processes. The cross section for Rutherford back scattering varies with the square of the atomic number therefore RBS is most sensitive for higher atomic number components in or on a low atomic number matrix; sensitivity could be of the order of 10^{-2} - 10^{-3} monolayer. No chemical environment information is possible. Applications usually for elemental analysis as function of depth in a sample.

SIMS - secondary ion mass spectroscopy. Bombard the target specimen with an ion beam and mass analyze the ejected ions (ejected neutrals are not analyzed). A destructive technique. Can distinguish isotopes but no chemical environment information about the surface is possible. Sensitivity is about 10^{-6} monolayer. In static mode, 1-3 keV ions (focused to 0.1-0.5mm) remove only the outermost layer of the specimen. In dynamic mode, 15-20 keV ions (focused to 1 μm) produce a depth composition profile.

EDS - energy dispersive spectroscopy (or XEDS). High energy electron beam, 20-30 keV in SEM, 100-300 keV in TEM or STEM, generates x-rays characteristic of atomic elements in sample volume. Used in small spot mode, line profile mode, or 2D scan for elemental mapping. Spatial resolution depends on electron beam spot size, energy, and sample thickness (beam spreading). Best spatial resolution now achieved with 0.2 nm beam spot (field emission gun), at 200-300 keV and thin sample (say 10 nm). Energy resolution typically 140 eV, best about 120 eV for GeLi or Si detectors, however, for a wavelength dispersive spectrometer (WDS) the energy resolution is considerably better (10 eV?). Elemental sensitivity in typical experiments is around 1%, but with effort can be better. Precise quantification requires careful calibration with appropriate standards.

ALCHEMI - atom location by channeling-enhanced microanalysis. The technique uses EDS (or PEELS?) measurement with sample orientation in the electron beam exciting strong Bragg diffraction condition or planar channeling condition, such that specific Bloch waves are excited which are localized on known planes. If an impurity is located on such planes, get enhanced characteristic x-ray intensity, thus allowing impurity atom location to be determined.

STM - scanning tunneling microscopy. Surface electronic states probed by tunneling currents between sample and very small radius tip which is held at a constant distance (voltage?) from the sample surface. Spatial resolution depending on tip radius and distance from sample surface, usually atomic resolution. Recent application to magnetic vortex core imaging in high T_c superconductors.

FIM - field ion microscopy. Now the sample is a tip with very small radius, usually held at low temperature (to minimize thermal atomic motions?) and high voltage (10 kV?). Imaging gas ionized and accelerated from the tip from high points (atoms and edges of atomic planes), geometrical magnification and resolution of atom locations on exposed planes of tip. Pulsing (kV) evaporates planes on the tip to obtain 3D atomic data. Time of flight mass spectrometer then also becomes elemental identification, thus APFIM (atom probe FIM).

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