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HIGH TEMPERATURE MATERIALS LABORATORY FIFTH ANNUAL REPORT (OCTOBER 1991 THROUGH SEPTEMBER 1992)

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HIGH TEMPERATURE MATERIALS LABORATORY FIFTH ANNUAL REPORT* (OCTOBER 1991 THROUGH SEPTEMBER 1992)

V. J. Tennery and F. M. Foust

ABSTRACT

The High Temperature Materials Laboratory (HTML) has completed its fifth year of operation as a designated Department of Energy (DOE) User Facility at the Oak Ridge National Laboratory (ORNL). Growth of the User Program is evidenced by the number of outside institutions executing user agreements since the facility began operation in 1987. A total of 145 nonproprietary agreements (77 university and 68 industry) and 30 proprietary agreements (2 university, 28 industry) are now in effect. Five other government facilities have also participated in the User Program. Thirty-six states are represented by these interactions.

Eighty-one nonproprietary research proposals (44 from university, 36 from industry, and 1 other government facility) and six proprietary proposals were considered during this reporting period. Research projects active in FY 1992 are summarized.

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1. INTRODUCTION

The High Temperature Materials Laboratory (HTML) is a modern research facility that houses an array of special instruments used to meet research needs for advanced high-temperature materials, including structural ceramics and alloys. The research instruments in the six HTML User Centers provide a comprehensive set of tools for performing state-of-the-art determination of the structure and properties of solids. A key part of the HTML concept includes a staff of highly trained technical personnel who interact with industrial and university researchers in this Department of Energy (DOE)-designated National User Facility. The User Centers are organized to provide materials characterization support to appropriate university and industrial users and to research programs throughout the local DOE facilities. Support includes a wide range of involvements with research personnel such as (1) conducting research relating materials properties to structure, (2) characterization of one-of-a-kind specimens, and (3) training qualified users and then providing them access to equipment to perform their own materials research.

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User Agreements were developed which establish the intellectual property and liability rights of the user institution and Martin Marietta Energy Systems, Inc. The first official User Agreement was signed on July 15, 1987. Since that time, 175 agreements have been executed. The first users from the university and industrial community started their research projects in August of 1987.

2. COMMITTEE ACTIVITIES

Three advisory committees assist in the successful operation of the HTML User Program. These committees are listed below with a brief description of their organization, function, and activities during the past year. A listing of members of these committees is included in Appendix A.

Advisory Committee. This senior committee has the responsibility of advising the HTML Director on policy for operation of the User Centers. It is composed of five members who represent the industrial and academic communities. The committee meets annually. A formal report containing conclusions resulting from a meeting of the committee is submitted to the Associate Director for Physical Sciences and Advanced Materials of Oak Ridge National Laboratory (ORNL).

User Advisory Committee. The responsibility of this committee is to review nonproprietary research proposals and make recommendations to the HTML Director as to their acceptability. It is composed of six members: two from industry, one from a university, one from DOE, one from the Metals and Ceramics (M&C) Division staff, and the HTML Director, who serves as the permanent chairman. The committee meetings are normally held on a quarterly basis (March, June, September, and December) each year.

HTML User Exchange Group. On September 25, 1992, the third meeting of this group was held. Present, past, and potential HTML users are invited to these meetings, which provide participants an opportunity to give advice on how the User Program can be improved and made more effective. The agenda of the 1992 meeting is included in Appendix B.

3. USER AGREEMENTS

Two types standard agreements are utilized in the HTML User Program: the "Non-proprietary Agreement" for research projects whose results are reported in the open literature within six months of the completion of the project in the HTML and the "Proprietary Agreement" for all projects in which the user desires that the data and results be proprietary. Table 1 is a listing of institutions executing nonproprietary user agreements to date.

Table 1. Standard User Agreements executed July 15, 1987, through September 30, 1992

UNIVERSITIES - 77

Alfred University
Auburn University
Berea College
Brown University
Clemson University
Cornell University
Dartmouth College
Florida International University
Florida State University
Georgia Institute of Technology
Harvard University
Illinois Institute of Technology
Iowa State University
Johns Hopkins University
Kent State University
Lehigh University
Louisiana State University
Marquette University
Massachusetts Institute of Technology
Michigan State University
Michigan Technological University
Mount Holyoke College
New Mexico Tech
North Carolina State University
North Carolina State A&T University
Northwestern University
Ohio State University
Oklahoma State University
Pennsylvania State University
Rensselaer Polytechnic Institute
Rice University
Rochester Institute of Technology
Rutgers University
South Dakota State University
Southern Illinois University
Southern University
Stanford University
Stevens Institute of Technology
Tennessee Technological University
Tuskegee University
University of Akron
University of Alabama
University of Arizona
University of California/Los Angeles
University of California/San Diego
University of California/Santa Cruz
University of Cincinnati
University of Connecticut
University of Dayton
University of Delaware
University of Denver
University of Florida
University of Houston
University of Illinois
University of Kentucky
University of Maryland
University of Massachusetts
University of Michigan
University of Minnesota
University of Missouri-Colombia
University of Missouri-Rolla
University of New Mexico
University of North Carolina/Chapel Hill
University of Notre Dame
University of Pennsylvania
University of Pittsburgh
University of Puerto Rico
University of Southern California
University of South Carolina
University of Tennessee
University of Utah
University of Wisconsin
Vanderbilt University
VPI & State University
Washington State University
Washington University
Wright State University

Table 1. Continued

INDUSTRIES - 68

Alcoa Technology Center	Great Lakes Research
Allied-Signal	GTE Laboratories, Inc.
Allison Gas Turbine Div./GM	IBM Almaden Research Center
Alzeda Corporation	ImTech Company
Americom Inc.	INRAD, Inc.
American Matrix, Inc.	Institute for Defense Analyses
American Superconductor Corporation	Ionic Atlanta, Inc.
Aluminum Co. of America	Litton Industries
AT&T Bell Laboratories	McDonnell Douglas Corporation
Babcock & Wilcox	Miniature Precision Bearings
CarboMedics, Inc.	Monarch Tile, Inc.
Carborundum Company	Norton Company
Ceramics Process Systems Corp.	Norton/TRW Ceramics
Certainteed Corporation	Nuclear & Aerospace Materials Corporation
Chrysler Corporation	Proctor & Gamble Co.
Church & Dwight Company, Inc.	Quadrax Corporation
Concurrent Technologies	Refractory Test Associates
Coors Ceramics Company	ReMaxCo Technologies, Inc.
Cummins Engine Company	SB&TD Business System
Detroit Diesel Corporation	Selee Corporation
DG Trim Products	Solar Turbines, Inc.
Dow Chemical Company	Southwest Research Institute
Dow Corning Corp./Midland	Sullivan Mining Corporation
E. I. du Pont de Nemours	Sundstrand Power Systems
E. I. du Pont de Nemours (Fluorochems)	Teledyne Allvac
Eastman Chemical Company	Tennessee Center for R&D
Eaton Corporation	Textron Specialty Materials
Energy Conversion Devices, Inc.	Thermacore, Inc.
Engelhard Corporation	Third Millennium Technologies, Inc.
FMC Naval Systems Division	Torrington Company
Ford Motor Company	Tosoh SMD, Inc.
Foster-Miller, Inc.	United Technologies Corporation
General Electric Aircraft Engines	Universal Energy Systems, Inc.
Goodyear Tire & Rubber Company	Westinghouse Electric Corporation

Due to the sensitive nature of many of the proprietary research activities, the names of the user institutions for proprietary agreements are not listed. In addition, researchers from five other government facilities have participated in the HTML User Program to date.

4. USERS

A cumulative summary of user activity since the start of the User Program is included as Appendix C.

The category of users and number of user days accumulated during this fifth year of HTML operation are shown in Table 2.

Table 2. HTML FY 1992 user experience

Type of user	Number of Institutions	Number of Individuals	User days
Industry	24	42	3692
University	20	27	880
Local Oak Ridge users	1	124	4661
Totals:	45	193	9233

Figure 1 shows the total number of 8-h user days per quarter during FY 1992. The industrial user days varied from 756 to 1229 during a particular quarter, university user days ranged from 123 to 334, and local user days ranged from 977 to 1379. Figure 2 shows the number of user days for all industrial and university users in the HTML for each quarter of FY 1992.

To date, 331 research proposals have been submitted to the User Program. A breakdown by user category of these proposals is given in Table 3. Figure 3 is a graph indicating proposals received by fiscal year of operation. Access clearances have been initiated on 452 individual researchers who were listed as principal investigators on these proposals.

The capabilities of each User Center are described, and a short summary of the research performed by nonproprietary users in that center is given for each user organization. A listing of publications and presentations resulting from user research projects is given in Appendix D.

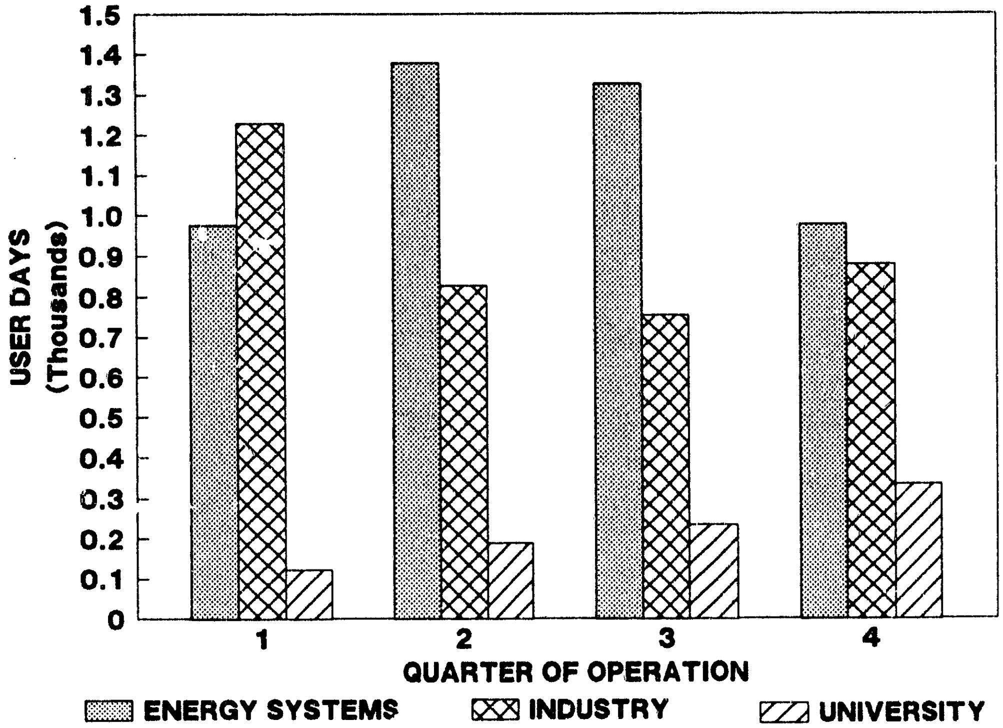

Fig. 1. Total user days in the HTML for FY 1992.

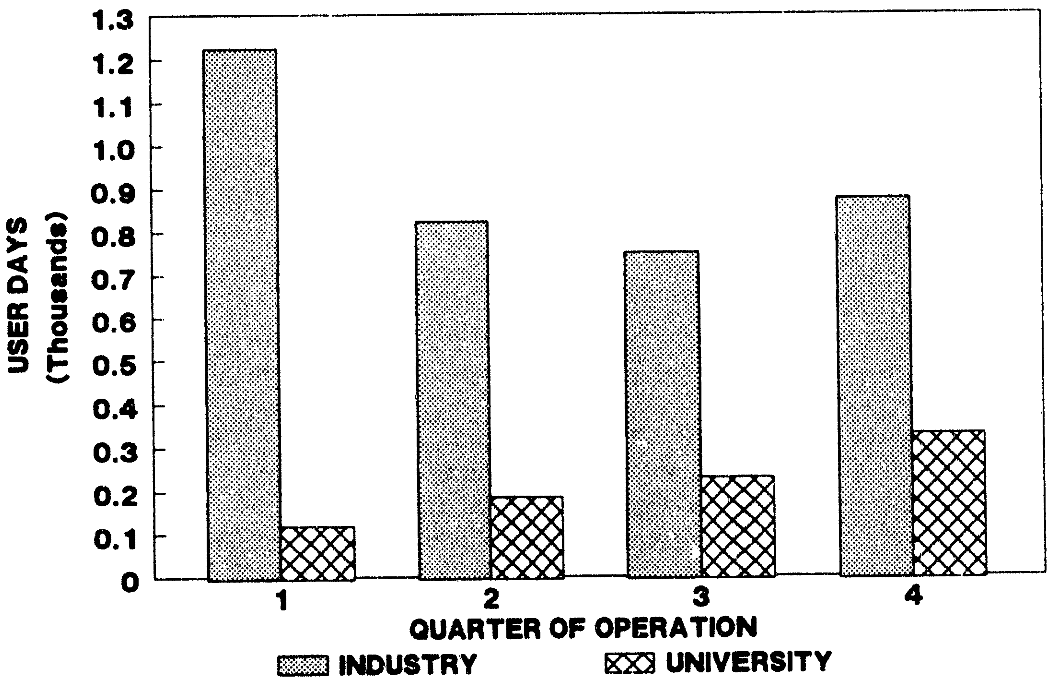

Fig. 2. Total industrial and university user days in the HTML for FY 1992.

Table 3. Research proposals submitted July 1987 through September 1992

User type	Research proposals submitted
Industry	126
University	177
Other Government	5
Total Nonproprietary	308
Proprietary	23
Grand Totals	331

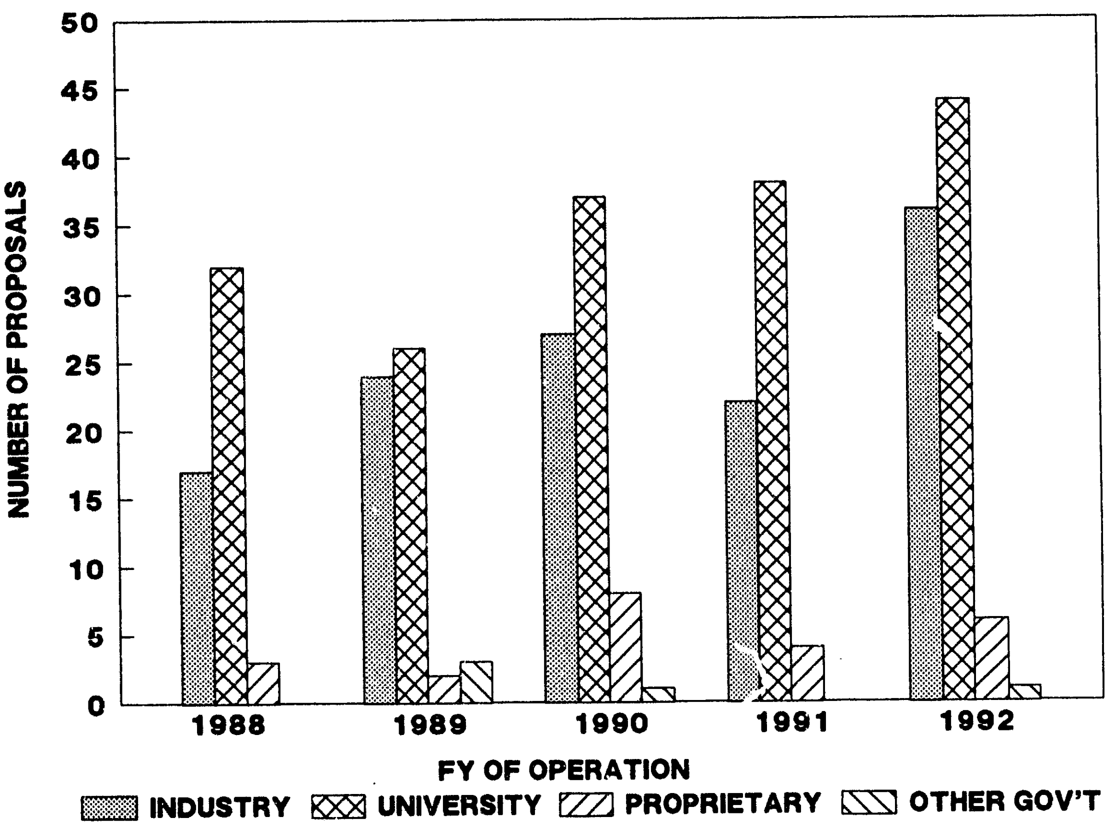

Fig. 3. Total number of research proposals submitted for consideration since the HTML User Program began in July 1987.

5. USER CENTER CAPABILITIES, INSTRUMENTS, AND RESEARCH PROJECT SUMMARIES

A description of the capabilities and function of each User Center is given here. Following the description of the User Center are brief summaries of those research projects that had significant activity during this report period. Many projects were active in more than one User Center as indicated in the projects summary (see page i).

5.1 MATERIALS ANALYSIS USER CENTER (MAUC) - T. A. NOLAN

The MAUC utilizes electron microscopy and surface chemical analysis techniques to characterize the structure and chemistry of advanced structural materials. The information obtained from these characterizations is used to elucidate the mechanisms that control materials performance.

Additional staff members in the MAUC are listed with the instruments for which they have primary operating responsibility:

Dr. L. F. Allard	Hitachi HF-2000 FE-TEM, JEOL 4000EX TEM
Mr. D. N. Braski	PHI 660 SAM and Topometrix SPM
Ms. D. W. Coffey	Hitachi S-800 FE-SEM and specimen preparation
Mr. T. J. Henson	JEOL 733 Electron Microprobe
Dr. K. L. More	JEOL 2000FX AEM and 4000EX TEM
Ms. A. D. Underwood	Specimen preparation and SPM
Ms. R. W. Shupe	Administrative support

Several important new instruments have been added to the suite of User Center instruments. A scanning probe microscope (SPM) incorporating both scanning tunneling and atomic force microscopy (AFM) modes is operational. This instrument has interchangeable heads that allow the surfaces of large items, such as ceramic tensile specimens and metallographically mounted specimens, to be imaged with near-atomic resolution. Another major new addition is a field emission gun-transmission electron microscope (FEG-TEM). The Hitachi HF-2000 200-kV FEG-TEM began beneficial operation in June 1992 (two months ahead of schedule). This instrument adds two major new capabilities. On specimens having ideal geometry, it provides the highest lateral resolution presently attainable for X-ray elemental analysis; elemental composition of regions as small as 1 nm can be determined, thus greatly enhancing our abilities to analyze ceramic grain-boundary compositions. The field emission source illuminates the specimen coherently, thus allowing electron holography to be performed. Electron holograms preserve image phase information (lost in conventional TEM). Utilizing the additional phase information, lens aberration corrections can be made that should result in greatly improved resolution (possibly reaching the 0.1-nm level). Also, magnetic flux quanta can be imaged, and specimen thickness variations of less than 0.05 nm can be determined. The first year of a three-year Director's Funds initiative to develop electron holography has been completed, and we are now routinely taking high-resolution electron holograms on the HF-2000. Dr. E. Völkl, an international leader in electron holography, has joined our staff as a postdoctoral fellow to assist in the holography project.

Both internal and external user research projects have produced significant results during the past year. Analytical and high-resolution TEM have been used to determine mechanisms of creep and fatigue in Si_3N_4 structural ceramics. These studies contributed to the reformulation of a manufacturer's Si_3N_4 ceramic, which greatly improved the high-temperature properties. During this year, we have continued to develop an understanding of the specific structural and chemical differences that result in the improved high-temperature behavior. The HF-2000 has been used in this endeavor to provide high-spatial-resolution elemental analyses of grain boundaries. Both high-resolution electron microscopy (HREM) [using the 4000EX] and high-spatial-resolution elemental analysis (using the HF-2000) have been employed to characterize the microstructure of the ytterbia-fluxed Kyocera SN-260 prior to and after high-temperature fatigue testing. This ceramic provides insight into the effects of using a "non-traditional" sintering aid on properties. High-resolution transmission and scanning electron microscopy (SEM) techniques have been used to characterize multi-ion-beam reactive sputtered lead lanthanum titanate thin films. Changes in electrical properties have been related to the microstructural development as a function of processing conditions. AFM has been used to measure topographical details of small indentions in ceramic materials that will be related to the mechanical properties of the ceramics. The technique has also revealed interesting growth morphologies of chemically vapor-deposited (CVD) SiC in SiC/SiC composites, provided information on the quality of machined silicon nitride surfaces, and shown minute features created on silicon nitride wear (tribology) samples.

Allied-Signal Aerospace Company User Center: MAUC, MPUC

Project Title: "Microstructural Evaluation of Time-Dependent Failure of NT 154 Silicon Nitride," *M. N. Menon*

Within the framework of this user project in the MAUC, 29 specimens were examined, using the SEM, for cavitation. Cavities were observed on grain boundaries transverse to the stress. Results indicated that cavity size increased with increase in temperature and time in the specimens tested in creep between 1204 and 1400°C.

GTE Laboratories, Inc. User Center: MAUC

Project Title: "Electron Holography of Multilayers in Strained-Layer Quantum Well Structures," *C. Sung*

Ultra-high-resolution TEM examination of Quantum wells (QW) in InGaAs/InP materials was carried out in order to obtain information regarding interfacial structures on an atomic scale. Two samples, C-59 (having 5 QWs and 20/20 interrupt times) and C-43 (having 15 QWs and 0/20 interrupt times), were examined using the JEOL 4000EX high-resolution microscope. Atomic images of the interfaces indicated that the use of a delay time (20 s) provided a sharper interface with less interaction of the As into the InP lattice. An atomic step was observed at the interface as a result of processing using 3° off InP substrate orientation. This type of detailed atomic data cannot be obtained using any other type of analytical instrumentation.

A second set of InGaAs/InP samples contains QWs that range in thickness from approximately 15 to 100 Å. The exact thicknesses were measured directly from the ultra-high-resolution TEM images and correlated to the known deposition times (in seconds). Initial review of the images obtained seems to indicate some differences in the interfaces in regard to group V substitution of As-P, depending upon the processing conditions. One sample of InGaAs on InP substrate was analyzed. Images obtained on this specimen showed the interface between the InP substrate and Zn-doped InGaAs to be highly strained. Many defects, such as dislocations, were observed on an atomic scale in the deposited layer with some of these dislocations observed to propagate into the InP substrate.

<u>Quadrax Corporation</u> User Center: MAUC, MPUC

<u>Project Title</u>: **"Elevated-Temperature Characterization of Three Dimensionally Braided Ceramic Composites,"** *M. D. Mello*

Research efforts have been directed at the preliminary characterization of three dimensionally braided continuous fiber ceramic composite (CFCC) materials. The composite constituents are Nicalon™ fiber and silicon carbide produced by chemical vapor infiltration and polymer precursor impregnation for the matrix.

Auger electron spectroscopy (AES) was used to perform an elemental analysis of the matrix composition and fiber interface. Foreign elements were identified in the matrix materials and were concluded to be by-products of the matrix-forming process.

SEM provided the opportunity to examine the integrity of the fiber/matrix composition. High magnification revealed the presence of porosity in the matrix, and variations in the fiber cross-section geometry, and also verified fiber coating thickness and matrix morphology.

<u>Georgia Institute of Technology</u> User Center: MAUC

<u>Project Title</u>: **"Identification and Structure of Phases in the CVD Si-B-Ti-C System,"** *J. A. Hanigofsky*

Work on the identification of Si-C-B phases deposited using CVD was initiated in June 1992. Drs. More and Freeman determined that the film thickness was too thin to successfully complete TEM sample preparation. Deposition work at Georgia Tech is being completed at this time for further microscopic evaluation.

<u>Pennsylvania State University</u> User Center: MAUC

<u>Project Title</u>: **"High-Resolution Electron Microscopy Study of the Chemical Order Domains in Complex Lead Perovskites, $Pb(Mg_{1/3}Nb_{2/3})O_3$:$PbTiO_3$,"** *C. A. Randall*

HREM studies have been completed on the $Pb(Mg_{1/3}Nb_{2/3})O_3$:$PbTiO_3$ solid-solution system. The role of intermediate scale ordering is strongly correlated to the dielectric properties within the lead-based perovskites. A detailed analysis of the fine structure of the order is still under study using computer simulations. Based on our modeling so far, Fm3m

$2a_o$ x $2a_o$ x $2a_o$ unit cell is the ordered structure. However, the sensitivity of the image simulation to various occupancy models is proving to be difficult.

Pennsylvania State University User Center: MAUC

Project Title: "Ferroelectric Phase Transitions in the High-Resolution Electron Microscope," *C. A. Randall*

Submicron $BaTiO_3$ crystals were aligned in a polymer under electric fields using dielectrophoretic forces. The alignment was characterized on microtomed sections. Dielectric properties were related to the connectivity of the $BaTiO_3$ alignment. Dielectric anisotropies correspond to 90% alignment along the field direction. Unfortunately, the dielectric properties could not be used to assess the nature of the phase in the submicron particles.

Pennsylvania State University User Center: MAUC

Project Title: "Nature of the Ferroelectric Curie Point Transition in Barium Titanate," *C. A. Randall*

A high-temperature analytical TEM study has been completed on $BaTiO_3$ crystals in the paraelectric phase. Zone-axis electron diffraction patterns were taken along [001] directions to reveal diffuse scattering planes along <100> directions. The diffuse scattering was found to be independent of the processing of the $BaTiO_3$ crystal and hence believed to be an intrinsic feature of the structure. The temperature dependence of the diffuse scattering did not correspond to second-harmonic generation studies, leading us to the conclusion that the origin of these two phenomena is not related as previously anticipated in the literature.

Pennsylvania State University User Center: MAUC

Project Title: "Microstructure and Interface Studies of Lead-Lanthanum-Titanate Thin Films," *S. B. Krupanidhi*

Ferroelectric thin films deposited by multi-ion-beam reactive sputter deposition were analyzed with high-resolution SEM and HREM. The relationships among composition, crystal structure, microstructure, and electrical properties were determined for thin films of the lead-lanthanum-titanate ternary system. The influence of the $Pb_{1-1.5x}La_x$ (PL) substrate on $Pb_{1-1.5x}La_xTiO_3$ (PLT) microstructure formation and the interface between the PL and PLT were also observed.

University of Arizona User Center: MAUC

Project Title: "Chemical Analysis of Laser-Processed Ceramic Coatings," *B. D. Fabes*

The research being done under this proposal involves the use of the scanning Auger microscope (SAM) at the HTML to analyze the near-surface composition of borosilicate glass (BSG) films. The thin films are prepared from the sol-gel method at a specific composition of boron. When these films are traditionally fired in a furnace to densify them, much of the boron is lost because it is easily volatilized. By densifying these BSG films with a laser, the heating time is much shorter, milliseconds as opposed to minutes in the furnace. This very short firing time may not allow much volatilization of the boron from the BSG coating. Some samples were made by laser firing and some by furnace firing. The boron content of the films was measured with the SAM.

The first experiments were preliminary and proved that there is more work to be done and that our methods are appropriate. More laser-densified films will need to be measured, as well as a bulk sample with known boron content to calibrate the system, before conclusions can be drawn.

University of New Mexico User Center: MAUC

Project Title: **"Characterization of Polymer-derived Ceramic Materials,"** *A. K. Dayte*

During this period, we have continued our work on the oxidation stability of BN thin films on oxide substrates. We found that BN films are lost during oxidation on substrates such as MgO and Al_2O_3. The loss occurs due to reaction of BN with the oxide to form orthoborates. This does not occur on the BN/TiO_2 system where the BN phase segregates but survives intact up to 1000°C.

University of Tennessee User Center: MAUC

Project Title: **"Study of the Fracture Surfaces of Samples of a High-Temperature Flange Bolt Material,"** *C. R. Brooks*

In secondary ion mass spectroscopy (SIMS) of steels containing carbon, it is necessary to know what mass peak to use to represent carbon. Although mass 12 can be used, multiples of 12 may also be present in the gas to be analyzed by the mass spectrometer due to carbon combination. To establish which peak best represents carbon, spectra were obtained from a 0.4% carbon-annealed steel, consisting of primary ferrite (of very low carbon content) and of pearlite (containing 12% of Fe_3C). Thus, in this sample, the location of carbon was known. It was determined that the mass 24 peak was best to use for carbon detection. A SIMS analysis was then conducted of a 12% Cr steel, high-temperature flange bolt in an attempt to identify elements in matrix and grain-boundary particles. These particles were found to be rich in C and Cr. Attempts to locate other particles (e.g., Laves) known to be in the sample were not successful.

University of Tennessee User Center: MAUC

Project Title: **"Measurement of the Boron Content of Intergranular Fracture Facets of a Boron-Doped Ni_3Al Alloy,"** *C. R. Brooks*

The purpose of this project was to see if there is a correlation between the boron content of grain boundaries in Ni_3Al and the grain-boundary structure. Special samples were prepared so that the crystallographic orientation of grains on a metallographic surface was determined using electron channeling patterns obtained on an SEM at the University of

Tennessee. These samples were fractured in the Auger electron microscope (AEM), and the boron content of the exposed grain boundaries was determined. It appears that the high-angle boundaries have the highest B content. This work was the basis of the Ph.D. dissertation of Y. C. Lin, who received his degree in March 1992.

University of Tennessee User Center: MAUC

Project Title: **"Study of Thin Films of 316L Stainless Steel Exposed to Microbial Activity,"** *A. J. Pedraza*

Sputter-deposited 316L stainless steel films deposited on various substrates were characterized using TEM and X-ray diffractometry. The deposits were found to be fine grained, and the phases present in the films depended on the nature of the substrate. Films of various thicknesses deposited on microscope slides or oxidized stainless steel substrates contained a mixture of two phases: a body-centered cubic (bcc) and a modified hexagonal ϵ-phase. The hexagonal phase appeared to be an ordered phase, as suggested by the a_o value of the structure, which is twice that for the ϵ-martensite found in many deformed stainless steels. Films deposited on oxide-free austenitic stainless steel substrates, on the other hand, were mostly bcc and exhibited a dominant <200> texture.

University of Tennessee User Center: MAUC

Project Title: **"Ordering Reaction in Ni_4Mo,"** *C. R. Brooks*

The purpose of this study is to examine the fine structure of the alloy Ni_4Mo while the ordering reaction proceeds from a cold-worked, disordered condition. TEM samples have been prepared and are ready for examination in the JEOL 4000EX TEM in order to obtain lattice image micrographs of the interface between the cold-worked and the recrystallized regions.

University of Tennessee User Center: MAUC

Project Title: **"Hot-Cracking in Welds,"** *C. R. Brooks*

Hot-cracking in welds is a common problem, but establishing the causes has proven elusive. However, using a Gleeble, small samples have been prepared which have internal cracks caused by hot-cracking. This project deals with attempts to fracture these samples in the Auger microscope in order to obtain the chemistry of the hot-cracked surfaces. Problems have been encountered in being able to fracture the samples in the Auger microscope. Samples broken in air, then transferred quickly to the Auger microscope, were too contaminated to give useful results. It is now believed that the device designed to cool the sample in the Auger microscope to near-liquid-nitrogen temperature to allow easy and brittle fracture in order to expose the internal cracks was not cooling properly, and the solution to this is now being examined.

University of Tennessee User Center: MAUC, MPUC

Project Title: "Study of Aluminum/Metal and Metal/Molybdenum Carbide Interfaces,"
A. J. Pedraza

Laser-treated substrate-grade alumina was investigated under TEM (JEOL 2000FX) for composition and microstructure. Surfaces of substrate-grade alumina were laser irradiated with xenon-chloride pulsed excimer laser under $Ar+H_2$ atmosphere with fluences of 3, 4, and 5 J/cm^2. Laser-treated and virgin alumina samples were back-thinned and characterized under the TEM for changes in crystal and grain structure and in chemical composition, if any. Only the grain size showed increase after laser treatment, whereas crystal structure and composition remained unchanged.

5.2 MECHANICAL PROPERTIES USER CENTER (MPUC) - M. K. FERBER

The MPUC is dedicated to the study of the mechanical performance of high-temperature materials. Other staff and their areas of expertise are as follows:

Dr. M. G. Jenkins	Tensile creep/fatigue of structural ceramics
Dr. A. A. Wereszczak	Fracture toughness testing and evaluation of interfacial properties in fiber-reinforced ceramic composites
Dr. C. K. Lin	Cyclic fatigue of structural ceramics
Dr. E. Lara-Curzio	Fiber strength evaluation
Ms. L. Riester	Indentation and flexure testing
Ms. P. Humphreys	Administrative support

A major thrust of the MPUC is to examine the influence of temperature, time, and applied stress level upon properties such as strength, toughness, fatigue, and creep resistance. The major research facilities include: (1) a Flexure Test Facility (FTF) comprising six high-temperature flexure load frames; (2) a Tensile Test Facility (TTF) consisting of eight high-temperature tensile testing load frames, a fiber test machine, a composites test machine, and a servohydraulic universal test machine equipped with tension/compression grips; (3) a general-purpose testing lab comprising two universal test machines and a room-temperature tensile tester; and (4) a mechanical properties microprobe (MPM) [Nanoindenter]. Currently, one of the universal test machines is equipped with a ceramic retort so that the high-temperature mechanical properties can be evaluated in inert environments or in vacuum. In the paragraphs that follow, detailed descriptions of flexure, tension, and indentation studies conducted in FY 1992, using these facilities, are provided.

5.2.1 Flexure Testing

During FY 1992, extensive flexure (and c-ring) testing was conducted using the FTF, a universal test machine equipped with a high-temperature furnace (designated as UTM-A), and a universal test machine equipped with a high-temperature furnace and ceramic retort for environmental testing (designated as UTM-E). The FTF is dedicated to high-temperature fatigue studies of structural ceramic materials and consists of six test frames, each having the capability of loading three flexure samples. For a given load frame, the specimen loading can be specified as a function of time. This feature permits the user to implement a number of standard fatigue tests including (1) static fatigue (time to failure measured as a function of static stress), (2) dynamic fatigue (fracture stress measured as a function of loading rate), and (3) cyclic fatigue (cycles to failure measured as a function of cyclic stress). Fast-fracture testing is also possible.

The UTM-A electromechanical tester is an Instron Model 6027 instrument with a load capacity of 200 kN (45 kip). The test machine is currently configured to apply loads up to 10 kN (2245 lb) at test speeds ranging from 1 μm/min to 1000 mm/min. The instrument is controlled using an electronic console consisting of a microprocessor and keyboard. Application programs are entered into the microprocessor memory via floppy disks. Data generated during testing may be displayed on an x-y recorder and/or transferred directly to a personal computer. A high-temperature clamshell furnace capable of generating temperatures (in air) to 1600°C is currently mounted on the 6027 test frame. This instrument is used to measure (1) creep rate as a function of stress for both flexure and compression specimens and (2) flexure and compression strength (including load versus displacement) as a function of temperature. Low-frequency cyclic testing is also possible with this instrument.

The UTM-E electromechanical tester is an ATS Model 1220 instrument with a load capacity of 89 kN (20 kip). The test machine is currently configured to apply loads up to 20 kN (4490 lb) at test speeds ranging from 50 μm/min to 50 mm/min. The instrument is capable of operating in displacement, load, or strain control. A built-in function generator provides for simple trapezoidal waveforms to control the displacement, load, or strain as a function of time. More complicated control waveforms can be generated by a computer equipped with a digital-to-analog converter. Data generated during testing are transferred directly to the computer using an external data acquisition system (DAS). The test frame also includes a high-temperature clamshell furnace equipped with a ceramic retort. Both compression and flexure tests may be conducted in air, inert gas, or vacuum to temperatures up to 1500°C.

Studies involving flexure (and c-ring) testing have focused upon (1) the measurement of cyclic fatigue behavior of silicon nitride ceramics as a function of temperature, (2) the effect of microwave annealing of silicon nitride upon the creep and fatigue resistance, (3) the relationship between fracture toughness of whisker-reinforced alumina and crack/whisker orientation, (4) the evaluation of the strength of SiC-SiC ceramic composites, (5) the effect of environment upon the fatigue resistance and retained strength of silicon nitride, and (6) the correlation of flexural creep data with tensile creep data generated for a high-performance silicon nitride. Specific examples are provided below.

University of Delaware User Center: MPUC, MAUC

Project Title: "Evaluation of Toughness of SiC Whisker-Reinforced Alumina Composites Fractured as a Function of Temperature and Loading Rate," *A. A. Wereszczak*

A hot-pressed 30 vol % silicon carbide whisker-reinforced alumina (SiC_w/Al_2O_3) composite (Product CC7000, ACMC) was chosen to investigate effects of fracture temperature (25, 1000, 1200, and 1400°C) and loading rate (displacement-controlled cross-head speed) on the K_{Ic} toughness. Flexure bars were chevron-notched and four-point loaded to induce a crack plane and direction that were parallel and perpendicular to the hot-press axis, respectively. Resulting toughness values (~ 5.5 MPa$\sqrt{m}$) remained relatively constant through 1200°C and decreased to about 4 MPa$\sqrt{m}$ at 1400°C. For all test temperatures, it appeared that as long as stable crack propagation was induced, the toughness seemed to be independent of the test cross-head speed (2, 5, 13, 32, and 79 μm/min). Fractography was used to understand the fracture mechanisms involved.

Silicon carbide whisker-reinforced alumina (SiC_w/Al_2O_3) composite interfaces were examined using HREM. HREM specimens were prepared from the bulk of samples that were previously tested for toughness at 25, 1000, 1200, or 1400°C in ambient air. The test temperature history served as the independent variable. It was found that the examined as-received material possessed a negligible interface thickness and that the test temperature history (which included a 30°C/min heating and cooling rate, a 30-min soak prior to specimen loading, and a typical test duration of 5 to 10 min) did not appreciably change the interface thickness at any of the elevated test temperatures.

<u>Allied-Signal Aerospace Company</u> User Center: MPUC

<u>Project Title</u>: **"Cyclic Fatigue Properties of Two Sintered Silicon Nitride Materials,"** *R. R. Rateick*

The fatigue resistance of Allied-Signal AS44B silicon nitride has been measured in four-point flexure. Tests were performed in both static and cyclic (0.33 Hz) modes at room temperature and 900°C. Slow crack growth tests were also performed at 900°C at strain rates from 10^{-8} to 10^{-4} s^{-1}. Crack growth rate exponents n of about 14 have been observed for the high-temperature measurements. Cyclic and static behavior did not appear to be differentiated at 900°C. Analysis of room-temperature data is in progress. Future work will investigate the existence of a fatigue threshold at 900°C.

<u>Ceramic Process Systems Corporation</u> User Center: MAUC, MPUC, XRDUC

<u>Project Title</u>: **"Duophase Sialon for High-Temperature Application,"** *R. R. Lee*

Duophase (α'/β) sialon is being developed for ceramic engine applications by using the Quickset™ injection-molding process, followed by pressureless sintering and a thermal treatment. The sialon had an average four-point flexural strength of 670 MPa at room temperature and 490 MPa at 1370°C. It survived the flexural stress-rupture test at 1300°C and 340 MPa for 190 h. X-ray diffraction (XRD) and TEM characterization showed that crystallization of the grain-boundary phase improved the high-temperature flexural strength of this sialon material. The creep behavior was also found to be affected by the crystallized grain-boundary phases. The formation of an yttrium aluminum garnet (YAG) phase and elongated grains yielded better creep resistance. The correlation between mechanical properties and microstructure is analyzed.

Solar Turbines, Inc. User Center: MPUC, MAUC

Project Title: "Effects of Hazardous Waste Incinerator Environments on the Strength of a SiC-SiC Ceramic Composite Material," *B. D. Harkins* and *V. M. Parthasarathy*

Solar Turbines' high-pressure heat exchanger design includes the option of a ceramic fiber composite header. One of the composites under consideration, AMERCOM's SiC-SiC composite, was evaluated to see if it would meet design requirements. Composite specimens, with and without a protective coating, were c-ring strength tested at temperatures ranging from room temperature to 1000°C. The protective coating prevented oxidation of the carbon coating at the fiber-matrix interface. However, under a load that resulted in microcracking, the strengths were reduced considerably at temperatures >500°C. The c-ring strength at temperatures >500°C was reduced to almost one-third of its room-temperature strength. At the elevated temperatures, the effects of varying cross-head speeds were very noticeable, suggesting a possible stress-corrosion effect. Auger microprobe and electron microprobe analyses revealed an absence of carbon at elevated temperatures, which appears to cause degradation in the strength of the composites.

Norton Company User Center: MPUC

Project Title: "High-Temperature Deformation Behavior of NT 154 Silicon Nitride," *R. Yeckley*

In this project, the fatigue and creep properties of a commercially mature hot-isostatically pressed (HIPed) silicon nitride (Norton's NT 154) were examined at 1370°C in both air and nitrogen environments. The fatigue behavior was determined from dynamic fatigue tests in which strength was measured as a function of stressing rate. Preliminary results showed that the fatigue resistance is substantially higher in nitrogen. The presence of nitrogen slows the oxidation process, which has been shown to be responsible for the accumulation of creep damage in similar HIPed materials. These trends were verified from dynamic fatigue tests of a similar HIPed silicon nitride conducted in air, nitrogen, and argon. For the air environment, a significant dynamic fatigue effect was observed (i.e., the strength decreased as the stressing rate was lowered). In the case of the argon and nitrogen environments, there was no dynamic fatigue. These results were again explained by the reduced oxidation rates in the argon and nitrogen environments.

Allison Gas Turbines-GMC User Center: MPUC

Project Title: "Effect of Inert Environment (Ar) Exposure on the Retained Flexure Strength of PY6 Silicon Nitride," *P. Khandelwal*

The retained room-temperature strength of a HIPed silicon nitride (GTE's PY6) was measured after exposure to thermal anneals in argon. To date, the retained strength has been measured after 1, 10, and 100 h at 1000, 1200, and 1400°C. Results suggest that the room-temperature strength is not significantly affected by these thermal treatments. To further identify possible failure mechanisms in inert environments, dynamic fatigue tests at 1000, 1200, and 1400°C are currently being conducted in air and argon environments. The data collected in air will serve as a baseline.

5.2.2 Tensile Testing

Proper tensile testing of brittle materials in tension requires the minimization of bending and/or torsional components so as to create a uniform, uniaxial, tensile stress in the gage section of the test specimen. Two methods are available in the TTF for achieving this uniform tensile stress state. The first is a passive system that employs a "free-free" tension-only grip system (Instron Supergrip) utilizing hydraulic couplers in the loading train. These couplers can automatically reduce the bending and torsional components during testing such that about 1 to 2% bending occurs at loads of 2250 N or greater. The second method is an active system (requiring user interaction) that employs a "fixed-fixed" tension-compression grip system utilizing an adjustable grip at one end of the loading train.

Eight electromechanical tensile test machines (Instron Model 1380) in the TTF are equipped with the Supergrip hydraulic couplers. The operation of all test machines is controlled with integral, electronic load controllers and function generators which allow three principal test modes: ramp at a controlled rate, ramp and hold at a constant load, and tension-tension cyclic loading. All machines are also equipped with short (100-mm) resistance-heated furnaces capable of 1600° C maximum temperature or 1500° C for sustained testing in ambient air. Six machines are equipped with contacting, capacitive extensometers that have resolutions of approximately 0.1 μm at room temperature and ~0.5 μm at 1500° C. A Keithley 500 DAS and IBM-compatible computer are used to monitor or control up to four test stations simultaneously.

A servohydraulic test machine (Instron Model 1332 with 8500 Series electronics) is equipped with the tension-compression grip system. A unique feature of the servohydraulic test machine is the state-of-the-art digital control system that allows either direct control (load or displacement) over the testing or remote control of testing by an IBM-compatible computer and custom software via a general-purpose interface bus (GPIB). Reversed cyclic loading can be accomplished at frequencies up to 25 Hz depending upon the maximum displacement.

The test machines described above are designed for testing primarily cylindrical, button-head specimens. Two additional electromechanical tensile test machines (Instron Model 1380) in the TTF provide for the evaluation of the tensile mechanical properties of fibers and flat composite specimens. Fiber testing is achieved through the use of a pneumatically actuated, kinematic fiber grip system. The water-cooled fiber grips are equipped with flat, titanium grip faces between which the fiber is squeezed without slippage or grip-related damage. The gripping force is adjustable through changes in the applied pneumatic pressure. Fiber gage length can be varied from 25 to 200 mm for room-temperature testing. Gage lengths of 155 to 200 mm are possible for high-temperature testing using a resistance-heated furnace capable of temperatures up to 1400 °C for sustained testing in ambient air.

The second electromechanical tensile test machine is equipped with a hydraulically actuated, wedge-loaded grip system. The gripping force applied to a flat composite specimen is adjustable through changes in the applied hydraulic pressure. Specimen lengths can be varied from 175 to 250 mm for room- and high-temperature testing. The resistance-heated furnace is capable of temperatures up to 1500°C for sustained testing in ambient air.

Extensive studies of the strength, creep, and fatigue behavior of silicon nitride buttonhead specimens were conducted at temperatures in the range of 900 to 1400°C. A major objective of these studies was to measure the temperature and stress sensitivities of the dominant failure mechanisms and then compare the resulting experimental data to model predictions. A major finding from this work was that when failure was controlled by creep-damage generation and accumulation, the fatigue life was uniquely determined by the steady-state creep rate (i.e., Monkman-Grant behavior). A second objective was to verify the expected improvements in creep and fatigue resistance of a HIPed silicon nitride, which resulted from processing modifications to the intergranular phase. Specific examples of these research activities are provided below.

Norton Company User Center: MPUC

Project Title: **"Characterization of Microstructural Changes Occurring During High-Temperature Deformation of NT 154 Silicon Nitride,"** *R. Yeckley*

Research activities have focused on the measurement of the tensile fatigue and creep properties of both NT 154, a mature HIPed silicon nitride, and modified NT 154 material designated as NTX 164. Baseline data generated for the NT 154 at 1260 and 1370°C indicated that fatigue was controlled by growth and coalescence of creep damage in the form of lenticular cavities formed between two-grain faces. In this temperature regime, the fatigue life was uniquely determined by steady-state creep rate in accordance with the Monkman-Grant relationship. These cavities were absent in the NTX 164 material. At a given stress and temperature, the creep rate was a factor of 2 to 4 lower than for the NT 154, while the fatigue life was a factor of 2 to 5 longer. These creep rate and fatigue life data fell along the same Monkman-Grant curve as for the NT 154 material. These results have been instrumental to the introduction of the NTX 164 silicon nitride as a commercial material.

Allied-Signal Aerospace Company User Center: MPUC

Project Title: **"Microstructural Evaluation of Time-Dependent Failure of NT 154 Silicon Nitride,"** *M. N. Menon*

In this project the creep/fatigue behavior of a HIPed silicon nitride (Norton's NT 154) was evaluated at temperatures of 982, 1149, 1204, 1260, 1315, 1371, and 1400°C. The resulting data have been used to verify several models for predicting both primary and secondary creep rates, and the stress-rupture life. The parameters used for relating failure time to stress at a given temperature were based on those developed previously for metals. In this work, the Dorn parameter was used for modeling of stress rupture because of its closeness in formulation to the creep rate model. However, it was further modified to accommodate an apparent change in mechanism observed both in the creep rate and stress-rupture behavior of NT 154. The applicability of a modified Monkman-Grant expression for describing the stress-rupture life was also examined. These phenomenlogical models are expected to be applicable to other silicon nitrides of similar class or chemical composition.

<u>Carborundum Company</u> User Center: MPUC

<u>Project Title</u>: **"High-Temperature SiC Development Program,"** *G. V. Srinivasan*

Mechanical properties of a developmental high-strength and high-toughness SiC, Generation I (SX-G1), were evaluated under a DOE/ORNL subcontract. The mechanical properties determined included flexural strength, tensile strength, and fracture toughness at room and elevated temperatures. Stress rupture, dynamic fatigue, and creep at elevated temperatures were also evaluated. The strength-limiting factors were identified at room and elevated temperatures. In terms of representative results, a mean flexural strength value of 780 MPa was measured at room temperature. The strength decreased at elevated temperatures, primarily due to a reduction of K_{IC}. The SX-G1 material appeared to be susceptible to slow crack growth at 1370°C. Finally, the SX-G1 SiC possessed good creep resistance, despite an oxide second phase in the material.

<u>Quadrax Corporation</u> User Center: MPUC, MAUC

<u>Project Title</u>: **"Characterization of Three Dimensionally Reinforced Ceramic Composites Formed by Various Matrix-Processing Techniques,"** *M. D. Mello*

Over 25 room-temperature tests of ceramic composites reinforced with 2- and 4-ply braided Nicalon fibers were evaluated in this project. Fibers were coated with 0.4-μm pyrolytic carbon to provide the fiber/matrix interface. Three types of matrices were employed: CVI SiC, polymer-infiltrated precursor (PIP) and pyrolized SiC, and a slurry-infiltrated Al_2O_3. All straight-sided test pieces (nominally 100 × 10 × 7 mm) were measured for the density of the composite and then excited using an impulse resonance technique to obtain the elastic modulus of the composite in the longitudinal (fiber) direction. Selected pieces were tested in uniaxial tension at room temperature and 0.1 mm/s displacement rate to obtain the complete stress-strain history, the matrix-cracking stress, the ultimate tensile strength, and total strain to failure. Agreement was found between the elastic modulus measured using the impulse resonance method and the mechanical behavior exhibited in the stress-strain curve, thus providing encouragement for use of the impulse resonance model as a material screening tool. In addition, good agreement was found between predicted and measured values of matrix-cracking stress, thus lending credibilty to heretofore unsubstantiated analytical predictions of this critical material parameter.

5.2.3 Mechanical Properties Microprobe

The MPM (the Nanoindenter), a major instrument in the MPUC, is a special microhardness tester capable of operating at loads in the microgram range (0 to 20 mN). A high-load range (0 to 120 mN) is also available. Unlike conventional hardness testers, it is not necessary to determine the area of an indent optically in order to calculate hardness. Instead, the height of the indenter relative to the surface of the specimen is constantly monitored with a sensitivity capacitance gage, thus allowing the depth of an indent to be determined. The unique feature of the Nanoindenter is its ability to measure indent depths to ± 0.2 nm. The area of the indent is then calculated from a knowledge of the geometry of the tip of the diamond indenter. The load is also constantly monitored, with the result that hardness is reported as a function of displacement. Measurements of sample stiffness from unloading data permit a separation of the plastic and elastic components of displacement, and the projected areas for indents can be calculated on the basis of the plastic depth of the indents. The elastic moduli of samples can also be estimated from stiffness data.

Motion of the specimen stage in the x-y plane is also precisely controlled. The indenter can be positioned within 2 μm of any chosen point on the specimen, and a series of indents, separated by steps as small as 0.1 μm, may be made in any geometrical pattern. The entire operation of the system is computer controlled, and one or several series of indents may be specified and carried out without further operator intervention.

During FY 1992, the Nanoindenter was used to (1) evaluate the plastic and elastic properties of thin films, ion-implanted surfaces, and laser-annealed surfaces; (2) generate load-displacement curves for silicon microbeams; and (3) measure the interfacial properties of fiber-reinforced ceramic-ceramic composites. Examples of these research activities are provided below.

Massachusetts Institute of Technology User Center: MPUC

Project Title: **"Fatigue of Microchemical Devices,"** *S. Brown*

The Nanoindenter was used to generate precracks in a single-crystal silicon micromechanical device developed to evaluate the fracture toughness as well as the static and dynamic fatigue properties. The structure of this device consisted of a cantilever with a large end plate and gold mass. Torquing and sensing electrodes, which extended over the plate, were used to drive (with associated electronics) the structure at resonance. Fatigue-crack propagation was measured by detecting shifts in the natural frequency caused by the extension of a crack from an initiation site near the base.

Stanford University User Center: MPUC

Project Title: **"Hardness and Elastic Modulus Measurements of InGaAs/InP and GeSi/Si Superlattices,"** *D. A. Stevenson and Jane Farthing*

This project, summarized in the HTML Fourth Annual Report (ORNL/TM-12023), was continued to allow for more measurements for comparison purposes. The Nanoindenter was used to measure the hardness of two sets of samples: (1) $In_1Ga_{1-x}As/InP$ superlattices with varying x values and (2) Ge_1Si_{1-x}/Si superlattices with varying x values. Elastic modulus values were also calculated from the elastic portion of the unloading curve. This work is continuing at Stanford through the development of models and by using TEM to study the dislocation structure of the superlattices.

<u>**Institute of Mining and Technology (New Mexico)**</u> User Center: MPUC

<u>**Project Title**</u>: **"Effect of Tin Dioxide Interlayer in Alumina/Glass Composites,"**
Krishan Chawla

Oxide fiber/oxide matrix composites, such as alumina fiberglass matrix, represent an important class of ceramic matrix composites because of their inherent stability in air at high temperatures. Alumina and glass, however, form a very strong chemical bond, which is undesirable from a toughness point of view. We present an interface engineering approach, which involves the incorporation and interface between the matrix and the fiber, in order to obtain energy-dissipating processes such as interface debonding, crack deflection, and fiber pullout in this system. We first examined the efficacy of tin dioxide as a barrier coating between alumina and glass bars. We confirmed by microprobe analysis that alumina and tin dioxide were mutually insoluble, but there was some solubility between silica and tin dioxide. This was followed by coating continuous PRD-166 (alumina + 15 wt % zirconia) fiber with SnO_2 and analyzing the microstructure and mechanical behavior of coated fiber composites. We observed that although the SnO_2 coating provided the intended diffusion barrier and the thermal stress distribution was of the desirable kind, a neat and clean fiber pullout was absent because of the roughness of the PRD-16/SnO_2 interface. Some fiber/matrix debonding, crack deflection, and crack bridging were operating. The roughness-induced radial clamping stress was too large to allow fiber pullout. To reduce this radial clamping effect, we then used a relatively smooth fiber, viz., Saphikon, a single crystal-alumina fiber. As expected, SnO_2 coated Saphikon fiberglass composite showed a much larger fiber pullout length than the coated PRD-166 fiberglass composite. Fiber push-through tests on this composite are under way.

<u>**Dow Corning Corporation**</u> User Center: MPUC

<u>**Project Title**</u>: **"Characterization of a-SiC:H Films Using Micro-Indentation Techniques,"**
M. Loboda

Amorphous hydrogenated silicon carbide (a-SiC:H) films were grown from two different precursor gases, a methane/silane mixture, and silacyclobutane (SiC_3H_8). Plasma-enhanced CVD was used to deposit a-SiC:H films at temperatures of 175 and 600°C. The a-SiC:H films were characterized using the Nanoindenter and by scratch testing. A simplified approach has been used to calculate the average micro-indentation hardness of the samples. Measurements on silicon show good agreement with that previously reported. The calculated a-SiC:H film hardness is shown to depend on the precursor gas at 175°C, while the film elastic modulus will vary with precursor gas, composition, and density, as determined by the plasma source deposition power. The micro-indentation and scratch test data show similar correlations to plasma source power and film structure and composition. A strong correlation is observed between the relative coefficient of friction of th film surface and the elastic modulus. Although small indentations (<100 nm) were performed, the data and estimation of the spatial extent of the plastic stress zone under the indenter suggest that for the modulus-to-hardness ratio range of the films examined ($5< E/H <9$), the indentation measurements were not influenced by the substrate.

Rutgers University User Center: MPUC

Project Title: "Hardness of Wear-Resistant Silicon Nitride," *J. B. Wachtman and A. Zutshi*

The Nanoindenter was used to evaluate the hardness of grains in a silicon nitride hot pressed with aluminum oxide. An attempt was made to correlate changes in grain hardness with the pickup of the aluminum. Although an indentation size effect (ISE) was observed in these materials, the specimen-to-specimen hardness variations were statistically insignificant.

Aluminum Company of America User Center: MPUC

Project Title: "Interfacial Properties Characterization in Nicalon SiC Fiber/Alumina-Based Composites," *Felix Wu*

Interfacial mechanical properties of both Nicalon SiC/aluminum borate and Nicalon SiC/aluminum phosphate with various fiber coatings and heat treatments were evaluated using a push-in test conducted with the Nanoindenter. Varying degrees of fiber/matrix bonding and sliding were measured depending upon the nature of the coatings and thermal treatments. An analytical model based on the work of Marshall and Oliver was found to provide reasonable predictions of the experimental stress-displacement curves.

Coors Electronic Package Company User Center: MPUC, MAUC

Project Title: "Effect of Impurities on Crack-Propagation Modes in Aluminum Nitride," *W. T. Minehan and L. Dolhert*

The Nanoindenter was used to measure hardness and elastic modulus on AlN samples containing thin Ni coatings. An attempt was made to correlate hardness and elastic modulus variations with distance from the coated surface. Originally, it was anticipated that variations in these properties would occur as a result of residual surface stresses and/or changes in the microchemistry in the surface regions. However, due to the load limitation of the MPM, the indent size was always significantly smaller than the grain size, such that the hardness and elastic modulus values were representative of the single crystal values. Tests performed with a high-load (1000-g) MPM, also available at the HTML, revealed a transition in hardness as the indent load was increased to 500 g. This transition was attributed to a change in deformation resistance as the indent size exceeded the grain size. The crack patterns emerging from the indenter corners provided some evidence of residual stresses in the surface regions adjacent to the coating. Work on this project is continuing.

Church and Dwight Company, Inc. User Center: MAUC, MPUC, RSUC

Project Title: "Surface and Near-Surface Damage on Aircraft Structural Alloys due to Impact of Very Soft Abrasives," *M. C. Rao*

The effect of particle shape and size on material removal and near-surface damage during aircraft depainting by soft abrasives was examined. Strips of Al 7075-T6 and Al 7075-T6 Al clad ("bare" and "clad" 7075) were impacted with flake- and cube-shaped NaCl abrasive particles in three particles sizes (graded fine, medium, and coarse) between 215 to 653 μm. Damage characterization was done by SEM (surface damage), Nanoindenter (subsurface work hardening), and XRD (residual stress).

Impact damage was more in the "clad" specimens than in the "bare" specimens, which showed minimal surface damage and no measurable workhardening. However, no significant abrasive shape or size effect was observed. A small, compressive biaxial residual stress was measured in the "clad" specimens, but large potential errors masked any existing trends. The residual stress could not be measured in the "bare" specimens due to excessively distorted diffraction peaks, probably from overly large grains.

Observations of the particles showed that the three grades of cube-shaped particles were somewhat similar in size and that the flake-shaped particles were agglomerations of smaller cube-shaped particles, probably breaking down on impact into the cube-shaped constituents. These two factors were likely the main reasons for the observed lack of a size or shape effect. The residual stress results were encouraging and may have been better if the dedicated residual stress diffractometer had been available at the time. The overall results could have been more conclusive if better characterized abrasives had been used.

5.3 PHYSICAL PROPERTIES USER CENTER (PPUC) - C. R. HUBBARD

The PPUC is dedicated to measurement of physical properties as a function of temperature and correlation of the thermophysical properties with processing, microstructure, and performance. Current facilities include the following instruments:

- Stanton Redcroft STA1500 Simultaneous Thermal Analyzer (STA)
  - Differential thermal analysis (DTA)
  - Thermogravimetry (TG)
  - Evolved gas analysis (EGA)
- Stanton Redcroft DSC1500 Differential Scanning Calorimeter (DSC)
- Theta Dual Push Rod Dilatometer
- Holometrix Laser Flash Thermal Diffusivity System
- Xenon Flash Thermal Diffusivity System
- Longitudinal Bar Thermal Conductivity System

The last two instruments are being built by the staff to meet specific measurement needs where no commercial instruments exist.

Members of the Diffraction and Thermophysical Properties Group with prime responsibilities for these instruments include:

Mr. W. D. Porter	Thermal analysis
Dr. R. B. Dinwiddie	Thermal transport
Dr. S. Beecher	Thermal transport
Ms. A. Abeel	Co-op student
Ms. J. Kilroy	Administrative support

Dr. Beecher recently joined the group as a postdoctoral fellow and is assisting with the development of the longitudinal bar and xenon flash systems. Ms. Abeel is an undergraduate co-op student from Virginia Polytechnic Institute who assists in development of instrumentations and in measurement and interpretation of thermal transport data.

The Stanton Redcroft thermal analysis instruments (STA1500 and DSC1500) can both make measurements up to 1500°C under a variety of atmospheres. A flexible stainless steel manifold system permits rapid exchange of gases and limits the oxygen partial pressure for studies of oxygen-sensitive materials. The dilatometer works with a helium atmosphere up to 1500°C for determination of bulk thermal expansion and determination of transition temperatures when volume changes occur. The laser-flash thermal diffusivity system operates in vacuum up to 2000°C for determination of thermal diffusivity as a function of temperature. The xenon-flash system is optimized for room-temperature measurements of thermal diffusivity and is used to screen samples prior to measurement in the laser-flash system. The longitudinal bar system is nearing completion of its development. It will be used to determine thermal conductivity from near-liquid-nitrogen temperature to 200°C. Combining data from the three thermal transport instruments will permit extensive modeling of the causes of thermal resistance in structural ceramics, high-temperature alloys, and composite materials. Besides a wide array of ORNL programs supported by the staff and facilities in the PPUC, the following user projects were supported.

<u>American Superconductor Corporation</u> User Center: PPUC, XRDUC

<u>Project Title</u>: **"Phase Stability of $Bi_2Sr_2Ca_1Cu_2O_x$ Superconductor at High Temperatures,"** *H. S. Hsu*

Final tests to fill in the experimental matrix were completed during the period. The STA has now been used to perform studies in 0.001, 0.10, 1.0, 7.6, 21, and 100% O_2/Ar atmospheres for thick films of $Bi_2Sr_2Ca_1Cu_2O_x$ supported on silver, and the results complement and extend those using high-temperature X-ray diffraction (HTXRD) as reported in the HTML Fourth Annual Report (ORNL/TM-12023). Results indicated that exothermic events on cooling at 10°C/min from a partially liquid state vary with oxygen partial pressure and can be grouped into three distinct sets. Set I is prominent for 0.001 and 0.1% O_2 in the range of 740 to 775°C and is believed to be associated with the crystallization of a Cu-free ~$Bi_5Sr_3Ca_1$ oxide phase. Set II results from the crystallization of 2212; it is observed for $P(O_2) \geq 1.0\%$ in the temperature range of 800 to 870°C. Set III appears for 21 and 100% O_2 in the temperature range of 880 to 910°C, and its origin is not clear from the results of this study. Subsequent room-temperature XRD from these samples suggests that, in general, high-oxygen partial pressures (100% O_2) tend to favor the formation of $Bi_2Sr_2CuO_6$ (2201), whereas low-oxygen partial pressures (0.001 to 0.1% O_2) lead to the formation of a Cu-free, Bi-Sr-Ca oxide phase. The 2212 phase forms at this cooling rate predominantly for intermediate oxygen partial pressures (7.6 to 21% O_2).

HTXRD during cooling (2°C/h) from the partially liquid state shows a pronounced dependence of the order of evolution of crystalline 2212 and 2201 phases on $P(O_2)$. For an oxygen partial pressure of 1.0%, the formation of 2212 precedes that of 2201, whereas for 0.01% O_2, 2201 crystallizes at a higher temperature than 2212.

The results of this study led to a better understanding of the role of kinetics and oxygen partial pressure in the development of phases in the $Bi_2Sr_2Ca_1Cu_2O_x$ system. Proper choice of processing conditions may lead to control of the phases present as well as the amount of each phase. Optimization of these parameters will lead to improved performance of Bi-based high-temperature superconductors. All results have now been analyzed and presented in both a poster presentation and a journal article.

Coors Electronic Package Company User Center: PPUC

Project Title: **"Thermal Conductivity of Alumina Nitride,"** *W. T. Minehan*

Aluminum nitride is used for high-performance electronic packages that require superior thermal dissipation characteristics. For example, the thermal conductivity of a typical alumina package is 25 W/mK, whereas aluminum nitride packages can be made with thermal conductivities greater than 200 W/mK. Hot-pressed technology for aluminum nitride is a unique and innovative method of co-firing aluminum nitride electronic packages with many advantages such as tighter dimensional tolerances.

Characterization of thermal conductivity as a function of material properties of hot-pressed aluminum nitride was performed. The major material characteristics of interest are oxygen content, residual calcia content, grain size, surface finish, and sample thickness. It is proposed to hot press aluminum nitride under a range of conditions, analyze samples for thermal conductivity at ORNL, and then characterize the samples. Characterization will include oxygen content by neutron activation, calcia content by X-ray fluorescence spectroscopy, and grain size by SEM. Data will be correlated to thermal conductivity using statistical analysis.

Coors Electronic Package Company User Center: PPUC

Project Title: **"Pyrolysis of Polyvinyl Butyral and Acrylic Binders from Ceramic Tape,"** *D. Horn*

Initial experiments were performed using the dilatometer and the STA1500S simultaneous thermal analysis system on a green alumina + glass + binder composition to determine the feasibility of the project. Evolution of a large amount of tarlike material during the dilatometer run proved to be a problem, and indicated pretreatment of the samples to burn out most of the binder will be required.

General Electric Aircraft Engines User Center: PPUC

Project Title: **"Thermal Conductivity of Vapor-Deposited Thermal Barrier Coatings,"** *B. A. Nagaraj*

We have modified the software to measure the thermal diffusivities of two phase-layered specimens (coating + substrate) at room temperature. Work is in progress to measure the thermal diffusivities of the specimens at high temperatures. Specimens have been prepared with physically vapor deposited (PVD) thermal barrier coatings (TBCs). The specimens are being subjected to high-temperature exposure.

Georgia Institute of Technology User Center: PPUC

Project Title: "Study of Polysilazane Pyrolysis by Simultaneous Thermal Analysis and Mass Spectroscopy," *P. Desai and D. Mohr*

The effect of pretreatment type on two polyorganosilazane preceramic binder materials was investigated using the STA/mass spectrometry (MS) system. Samples pretreated in air or nitrogen were heated at 10°/min to 1200°C in flowing argon. Weight losses and thermal events, as well as evolved gas species, were monitored. It was found that pretreatment in air resulted in formation of siloxane cross links between the polyorganosilazane molecules, which reduced evolution of low-molecular-weight silazane species during pyrolysis. Polysilazane materials are of interest because they can potentially be used as precursors to ceramic fibers, monolithic, or composite matrix materials. As binders, they may enhance processibility or density of pyrolyzed products.

McDonnell Douglas Missile Systems Company User Center: PPUC

Project Title: "Thermal Conductivity of Various Carbon Fibers," *J. W. Sapp*

McDonnell Douglas Missile Systems Company research at the HTML in 1992 continued to focus on development of high-thermal-conductivity carbon-carbon (C-C) composite materials. Four, one-dimensional C-Cs from different suppliers were tested in the laser-flash facility to define effects of fiber type and composite fabrication process on C-C diffusivity (conductivity). Room-temperature conductivity as high as 850 W/m K was measured for one material.

Rensselear Polytechnic Institute User Center: PPUC, XRDUC

Project Title: "High-Temperature X-ray Diffraction Analysis of Composite CVD SiC Fibers," *S. S. Sternstein and Edgar Lara-Curzio*

The structural stability of the SCS-6 filament was investigated in the temperature interval of 1000 to 1600°C by differential scanning calorimetry (DSC) and in situ XRD techniques. It was found that this filament is composed mostly of the cubic β polytype of SiC and graphite and that these phases are stable in the temperature interval of study. The lattice parameter for β-SiC was determined between room temperature and 1600°C, and the calculated average linear thermal expansion of the lattice in this temperature interval ($5.2 \times 10^{-6}C^{-1}$) is in agreement with macroscopic thermal expansion measurements on single filaments. Thermal events observed by DSC occur at 1380°C on heating and 1300°C on cooling and are suggested as being related to the melting and solidification, respectively, of excess silicon. These results are analyzed in relation to the anomalous thermal expansion behavior exhibited by this filament.

Teledyne Allvac User Center: PPUC

Project Title: **"Study of Major Alloying Elements and Morphology of Microstructural Components in Superalloys,"** *W. D. Cao*

Minor elements are present in small quantities, mostly less than 0.1%, in superalloys; however, little work has been reported on the effect of minor elements on phase segregation in these important materials. According to our theoretical analysis, minor elements may have significant influence on phase segregation. Segregation is one of the most important concerns of superalloy quality. During the report period, three visits were made to the HTML in which DSC was used to study the effect of minor alloying elements on the melting behavior of experimental heats of Inconel 718 produced by Teledyne Allvac. The effect of parts per million (ppm) levels of phosphorus and yttrium have been investigated. Changes in the melting behavior of the γ/Laves phase eutectic in the region of 1140 to 1200°C were noted.

Tosoh SMD, Inc. User Center: PPUC, XRDUC, MAUC

Project Title: **"Physical Property and Phase Equilibria Study of the Co-Cr-Pt Systems,"** *P. Frausto*

During this report period, the thermophysical properties and phase relationships of nine Co-Cr-Pt alloys were characterized using every instrument in the PPUC and XRDUC. Alloy compositions ranged from 11 to 30 at. % Cr and 2 to 18 at. % Pt. Properties determined included melting point, phase content and transformation temperatures, specific heat, thermal diffusivity, thermal conductivity, thermal expansion coefficient, and curie point. These properties are important in modeling the behavior of the alloys during casting and heat treating in preparation for their use as sputtering targets for magnetic media coatings (such as those used in computer hard disks) and potential use as corrosion-resistant coatings.

University of Tennessee User Center: PPUC, XRDUC

Project Title **"Analysis of High-Temperature Phase Transformation in Inconel 718,"** *M. H. McCay*

The phase-transformation behavior of Inconel 718, in as-cast, wrought, and wrought-grain-grown conditions, was examined using a DTA technique. The redistribution of alloying elements in wrought and wrought-grain-grown alloys slightly lowered the liquidus and solidus temperatures compared to the corresponding temperatures for as-cast alloy. The high-temperature (>1000°C) transformation events associated with the dissolution of $Ni_3Nb(\delta)$, γ/Laves and γ/NbC were present for as-cast alloy both during heating and cooling through the melting range, while they were absent for wrought and wrought-grain-grown alloys during heating. To the contrary, events such as the dissolution of γ/Laves (at ~1152°C) and $Ni_3Nb(\delta)$ (at ~1098°C) for wrought alloy and γ/Laves (at ~1100°C) for wrought-grain-grown alloy occurred only during cooling. During heating at lower temperatures (<1000°C), all three alloys exhibited the formation and growth of γ' and γ" phases from the supersaturated γ solid solution.

During this period, continued contact with the staff of the PPUC and XRDUC resulted in the submission of a journal article presenting the results of this study.

Vanderbilt University — User Center: PPUC

Project Title: **"Containerless Processing of Oxide Superconductor-Materials Characterization,"** *W. Hofmeister*

One visit to the HTML was made during the year in which two samples of drop-tube, containerless, processed YBaCuO superconductor were analyzed using the STA1500.

5.4 X-RAY DIFFRACTION USER CENTER (XRDUC) - C. R. HUBBARD

The XRDUC utilizes room- and high-temperature diffraction methods to characterize the phase(s) and stability of advanced structural materials. The data obtained individually, as a function of temperature, and/or in conjunction with data from thermal analysis or electron microscopy, are used to relate phase composition and stability with materials performance.

There are two major instruments in the XRDUC:

Scintag Θ-2Θ PAD V goniometer with l-N_2-cooled Ge detector
Scintag Θ-Θ PAD X goniometer with Buehler high-temperature stage

Members of the Diffraction and Thermophysical Properties Group with prime responsibilities for these instruments include:

Mr. O. B. Cavin	Mr. R. Simpson
Dr. T. Watkins	Mr. N. McAdams

Ms. J. Kilroy provides administrative support for this group also. Dr. Watkins recently joined the group as a postdoctoral fellow and is becoming familiar with the operation of these facilities. Two undergraduate co-op students, Messrs. Simpson and McAdams, also assist in measurement and interpretation of diffraction data.

The instruments are supported with a MicroVAX 3300 computer and Tektronix 4205 graphic terminals for data display and instrument control. The MicroVAX is one part of the overall HTML Local Area Network (ethernet) supporting both laboratory and office computers. The Buehler high-temperature stage is capable of heating specimens to 1500°C in oxidizing, reducing, or inert atmospheres, and to 2500°C in vacuum. Use of special insulation has permitted measurements in 1 atm N_2 to temperatures over 1800°C. Recent modifications permit multiple thermocouples to be placed on the sample and heater elements, providing greater knowledge of gradients and actual surface temperature of the specimen.

The room-temperature system is used in two modes. First, it supports many ORNL materials-related programs through phase identification, lattice parameter determination, assessment of preferred orientation, and quantitative analysis. Second, this system is used to characterize users' samples prior to and after measurement on the high-temperature system. The high-temperature system is widely used for recording powder diffraction data while simulating processing conditions; determination of phase equilibria; assessment of crystallographic thermal expansion; and characterizing thermal decomposition, solid-solid reactions, and even oxide-film-formation kinetics. Many projects in the XRDUC take advantage of the complimentary facilities in the PPUC.

Alzeta Corporation User Center: XRDUC

Project Title: **"Investigation of the Effect of High-Emissivity Agents on the Life of Fibrous Ceramic Infrared Burner Materials,"** *M. Carswell*

Lowering NOx emissions from burners is a major goal for combustion heater systems. One promising solution is to utilize ceramic fiber with high-emissivity coatings that reduce the flame temperature. Nextel ceramic fibers coated with NiO after 20 min and after 75 h at 1350°C were characterized by X-ray powder diffraction. The nickel was found to be present as a spinel-type $NiAl_2O_4$ phase. Other phases detected were mullite, alumina, and crystobalite. Major quantitative differences were detected with the spinel content decreasing and the mullite, alumina, and crystobalite contents increasing with length of exposure at temperature.

Clemson University User Center: XRDUC, MAUC

Project Title: **"Study of the Character of Stresses Developed in an Iron-Based Alloy Subjected to Sulfidizing Mixed Gases at Elevated Temperatures,"** *J. S. Wolf*

The primary objective of this research has been to provide information regarding the protective quality of scales formed in environments of interest at elevated temperatures. The crystalline nature of these scales and their microstructural evolution were studied for the case of type 310S stainless steel in oxidizing and sulfidizing atmospheres. Inferences were drawn with regard to stress generation during corrosion and other mechanistic details For these tasks, much of the data collection, processing, and analyses was done utilizing the XRD Facility of the HTML.

Clemson University User Center: XRDUC, MAUC

Project Title: **"Stress Generation in and Structure of Scales Developed on Unalloyed Nickel,"** *J. S. Wolf*

This research has been directed toward the characterization of ceramic scales naturally formed upon unalloyed nickel in oxygen at high temperatures. The primary objective has been to investigate methods for estimating the growth stresses that naturally occur in such oxides. The crystalline nature of these scales and the stresses generated during oxidation were the primary topics of investigation. For these tasks, much of the data collection and processing was done utilizing the high-temperature XRD Facility of the HTML.

The experimental portion of this program is essentially complete. Efforts are currently being focused upon data analyses, data interpretation, and report writing.

Clemson University User Center: XRDUC

Project Title: **"High-Temperature X-Ray Analysis of Titanium Aluminides,"** *H. J. Rack*

Research results on Ti-Al-Nb and Ti-Al-V alloys described in the HTML Fourth Annual Report (ORNL/TM-12023) have been published.

Clemson University User Center: XRDUC, PPUC

Project Title: **"The Effect of Initial Particle Size Distributions on the Melting Kinetics of Soda-Lime-Silica Glasses,"** *T. D. Taylor and D. K. Peeler*

Conventional glass-forming processes by high-temperature melting are usually divided into three interdependent stages: batch-free state, firing, and homogenization. In this research, the initial stage of melting has been evaluated, i.e., the batch-free process which involves the conversion of all crystalline raw materials into the liquid state. Effects of initial particle size distributions on the reaction kinetics of a typical soda-lime-silica glass system have been studied. HTXRD measurements provided a unique, dynamic analytical tool for monitoring the reaction pathways in situ.

Coors Electronic Package Company User Center: XRDUC

Project Title: **"Recrystallization in Alumina Ceramics,"** *J. Ghinazzi*

HTXRD was used to determine the phase composition as a function of temperature of green-state alumina. The results clearly reveal the decomposition temperatures of each of the starting minerals as well as solid-solid reactions which led to formation of intermediate crystalline phases. Research demonstrated the value of HTXRD for designing firing cycles for optimum processing.

Cornell University User Center: XRDUC, PPUC

Project Title: **"Calcining and Crystallization of Linear Organoelemental Polymer Precursors,"** *M. H. E. Martin*

Visits to the HTML during this period included experiments using the STA1500 and HTXRD to determine the crystallization behavior of both poly(methacrylate) and sol-gel precursors for forsterite and Cr-doped forsterites. Cr-doped forsterite is of increasing technological interest because of its potential use as a near infrared solid-state tunable laser. Results indicate a correlation between residual carbon in the precursors and their crystallization temperatures. Four presentations and five publications have resulted from this research.

Dow Chemical Company User Center: XRDUC

Project Title: "Glass Crystallization in Self-Reinforced Si_3N_4," *J. Hwang and R. Newman*

The purpose of our work at HTML was to (1) study the melt/recrystallization and (2) initial crystallization kinetics of the grain-boundary phase(s) in a silicon nitride formulation under development in Dow's Advanced Ceramics Laboratory. Understanding the crystallization/melt/recrystallization of the grain-boundary phases is important to engineering a better material for use at elevated temperatures.

Interfacing the Mbraun position-sensitive detector (PSD), which Dow Chemical supplied, to the HTML's high-temperature diffractometer enabled collection of XRD patterns in 3 to 5 min on average, much faster than what is possible with a scintillation counter. This data acquisition speed allowed observation of reaction kinetics during many of the experiments, and it also allowed completion of 28 high-temperature experiments during the 10-d period.

There were several important accomplishments during the 10 d of experimentation, which are summarized below:

1. Si_3N_4-based materials were examined at temperatures up to 1700°C, despite some complications due to the reactivity of Si_3N_4 at this temperature.
2. The actual specimen temperature was measured at the surface where XRD data are being collected by clamping a thermocouple in contact with the sample surface.
3. In later experiments, the thermocouple contacting the top surface was used as the control thermocouple, allowing us to directly control the temperature of the surface being analyzed.
4. With the PSD in place, we were able to successfully determine the kinetics of melting and crystallization of the grain-boundary phases in the Si_3N_4 formulation.
5. We were able to identify a previously unknown high-temperature intermediate grain boundary phase.
6. We observed the α-to-β phase conversion and the liquid-forming sequence during sintering of the multi-oxide-doped Si_3N_4 greenware.

Iowa State University User Center: XRDUC

Project Title: "Processing of Ti_5Si_3 and Cr_5Si_3 for Use as High-Temperature Materials," *M. Akinc*

The HTXRD facilities were used in this project to measure thermal expansion of Ti_5Si_3 and $Ti_5Si_3Co_{19}$. Time was not sufficient to allow Cr_5Si_3 to be measured. Publication efforts are under way.

Allison Gas Turbine-GMC User Center: XRDUC

Project Title: "Evaluation of the Effects of Cyclic Thermal Exposure on MAS Regenerator Materials," *J. Chang*

X-ray powder diffraction analysis of MAS ($2MgO:2Al_2O_3:5SiO_2$) samples exposed to cyclic thermal exposure in the Allison regenerator test rig was conducted to illucidate the mechanism responsible for strength reduction after cyclic thermal exposure. Phases found in all samples include $Mg_2Al_4Si_5O_{18}$ (Indialite) and $Al_6Si_2O_{13}$ (Mullite) with the Mullite phase better crystallized or in higher percentage in the exposed samples.

NASA Langley Research Center User Center: XRDUC

Project Title: "Hydrogen-Induced Phase Changes in Titanium-Based Alloys," *Ronald Clark*

To understand hydrogen transport/reactivity with titanium-based alloys, NASA Langley has an ongoing program that utilized the HTML's high-temperature diffractometer system for direct measurement of possible formation of hydrides. Ti-14Al-21Nb alloy was chosen for the study, and HTXRD data were collected at hydrogen pressures of 1, 5, and 50 torr as well as under vacuum. Evidence for probable hydride formation was observed.

University of Kentucky User Center: XRDUC, PPUC

Project Title: "Analytical Characterization of Ultrafine Particles," *J. Stencel*

During FY 1992, the research at the HTML was directed primarily at examining structural transformations of iron carbide particles by in situ HTXRD. These iron carbide particles, prepared by laser pyrolysis of C_2H_6/Fe (CO_5) precursors, contained particle sizes between 5 to 15 nm in their as-prepared state. Changes in the physical and chemical structures and size of these nanoparticles were followed by XRD under He and H_2 environments at temperatures as large as 600°C. Surface spectroscopy [X-ray photoelectron spectroscopy (XPS)] and thermal gravimetric analysis (TGA)/MS experiments were also performed.

University of Utah User Center: XRDUC, PPUC

Project Title: "High-Temperature X-Ray Analysis of the Gd_2O_3-Bi_2O_3 System," *Pomin Su*

Cubic solid solutions of Bi_2O_3-Gd_2O_3 are potential solid electrolytes in fuel cells and oxygen-separation systems. The addition of Gd_2O_3 is intended to stabilize the cubic phase. To further understand the role of Gd_2O_3 on the potential phase transitions, a series of compositions from 2 to 7 mole % Gd_2O_3 have been studied by high-temperature X-ray powder diffraction and from 2 to 25 mole % using DSC. Results are compared with quench studies and thermophysical property measurements to fully characterize the transition temperature, phase diagram, and properties. Future work will primarily focus on analyzing the results obtained.

5.5 CERAMIC SPECIMEN PREPARATION USER CENTER (CSPUC) - B. L. COX

During this report period, Mr. B. L. Cox assumed responsibility for the CSPUC. This center provides basic facilities for (1) investigating material removal processes associated with the machining of high-performance ceramics, (2) fabrication of new specimen test geometries required to evaluate the mechanical performance of structural ceramics, (3) dimensional inspection of machined specimens, (4) measurement of surface roughness and form, and (5) application of strain gages.

Specific equipment in the CSPUC includes:

Slicer/Grinder with Open-Loop Control
Computer Numerically Controlled (CNC) Slicer/Grinder
CNC Four-Axis Grinder
Optical Comparator
Computer Controlled Profilometer

Facilities for the application of strain gages are also available. Gage outputs are monitored during testing using a dedicated computer and DAS.

Members of the CSPUC with prime responsibilites for these instruments are:

Mr. V. T. Jenkins
Mr. R. Parten
Mr. E. Sheldon
Ms. P. Humphreys (provides administrative support to this group also)

During FY 1992, the CSPUC was utilized for several in-house programs. Silicon nitride compression specimens were machined for use in the Modular High-Temperature Gas-Cooled Reactor—New Production Reactor (MHTGR-NPR) Program. These specimens required very flat, parrallel ends and high concentricity. A machining plan was developed, and the specimens were machined on the Junger 4-axis grinder. The specimens were successfully tested in the MPUC. Also, a number of fiber-reinforced silicon carbide matrix composite tubes were machined in conjunction with mechanical tensile torsion testing at Virginia Polytechnic Institute and State University for use in the CFCC Program. These tubes had a 0.0002-in. tolerance on the OD surface, a length of 8 in., and basically represented an unknown material to machine. These factors contributed to the complexities of this project.

Several enhancements have been made to the equipment in the CSPUC. A Norton hydraulic wheel dresser was designed and installed as a permanent component inside the Junger 4-axis grinder. This has involved extensive planning in mounting the wheel dresser in the precise limited location for the work head to clear the wheel dresser during machining operations and still be able to reach the wheel dresser during specific operations. This is an innovative technique to automate the wheel-dressing operation in reducing time-consuming setups and give the CSPUC the capability of developing advanced wheel-dressing technology. Planned experiments on automated wheel dressing during machining will begin soon.

A special application to fit Harig wheel arbors onto the Junger 4-axis work head has been developed. It will allow generation of a concentric wheel with accurate form, straightness, or taper utilizing the CNC automated wheel-dressing operation on the Junger grinder. This would be very difficult to achieve on the Harig surface grinder alone. A unique arbor adapter for a 0.006-in.-wide diamond-plated slicing blade has been designed to mount on the Harig surface grinder instead of a Buehler cutoff saw. It was used to machine a 0.006-in.-wide slot, 0.?54 in. deep, to create a specially designed specimen for the use with the high-temperature Moiri interferometry system in the MPUC. This specimen was the object of study by a visiting professor during the summer.

A coolant overflow safety shut-off system has been designed and installed on the CNC grinders in the CSPUC. This has increased productivity by allowing safe, unmanned, after-hour usage of the equipment. Other enhancements include the addition of a Silicon Graphics crimson workstation with Pro/ENGINEER solid-modeling software, COSMOS/M finite element modeling software, CIMSTATION simulation software, and CARES brittle material life-prediction software. The workstation is installed in a computer laboratory and linked via ethernet to the CNC equipment in the CSPUC. The workstation will be used to model ceramic component geometry and optimize design parameters using the finite element and life-prediction software. It will also be used to program and simulate machine tool movement of the CNC equipment in CSPUC and a Coordinate Measuring Machine (CMM) being installed in the Ceramic Manufacturability Center. Actual inspection point information from the CMM will be displayed and evaluated on the workstation. A Macintosh-based machine tool programming system has also been purchased and installed. The system is very user friendly and will be used to program simple part geometries.

A presentation on the status and future direction of the CSPUC was made at the Workshop on Superabrasives and Grinding Wheel Technology for Machining Ceramics in Oak Ridge, Tennessee, on May 28, 1992. This presentation provided a brief description of the existing equipment and the user involvement, as well as a plan for future enhancement of existing equipment and expected research tasks. These plans include instrumentation of existing grinding equipment along with the addition of ultrasonic, hydroabrasive, and laser-machining equipment. Research tasks include basic machining studies, grinding wheel development, coolant evaluation, automated machine tool programming, and a closed-loop sensor feedback and control system.

5.6 RESIDUAL STRESS USER CENTER (RSUC) - C. R. HUBBARD

The RSUC has recently begun beneficial use following delivery of a unique, state-of-the-art instrument providing high intensity with very high precision in measurement. The system consists of two components:

Scintag PTS goniometer
MAC Science 18-kW rotating-anode generator

Members of the Diffraction and Thermophysical Properties Group with prime responsibilities for these instruments include:

Dr. C. R. Hubbard
Mr. O. B. Cavin
Dr. X.-L. Wang
Dr. T. Watkins

Both Mr. Cavin and Dr. Watkins, a postdoctoral fellow, have shared responsibilities between the XRDUC and RSUC. Dr. Wang is a postdoctoral fellow with primary responsibility for neutron diffraction measurements (discussed below). Ms. J. Kilroy provides administrative support for this group also.

The instrument is supported with the MicroVAX 3300 computer and Tektronix 4205 graphic terminals for data display and instrument control. The system was delivered in February 1992 and made operational in March. Considerable time has been spent in perfecting the operation and testing the software features. Recruitment for staff to provide additional technical support in this area is an ongoing effort now that the facilities are operational and user visits are being supported.

Mapping of residual stresses by XRD is being complemented by a joint project with the Neutron Scattering Group of the Solid State Division and the Welding Research Group of the M&C Division. This Laboratory-Directed Research and Development (R&D) Project has, as its main goal, to develop and demonstrate capabilities for nondestructively mapping residual stresses within engineering components such as welded plates or ceramic-to-metal joints. Facilities for X-Y-Z translation of specimens have been designed and automated at the High Flux Isotope Reactor (HFIR) and used for a series of demonstration projects.

The large number of proposals (12 in FY 1992) for use of the RSUC facility has been particularly encouraging. To date, however, due to limited instrument availability, only about one-third of the approved proposals have been scheduled and work initiated. Several of these proposals requested both X-ray and neutron residual stress mapping. The proposals where research has been initiated are summarized below.

<u>Concurrent Technologies Corporation</u> User Center: RSUC

<u>Project Title</u>: **"Vibratory Stress Relief-Process in Weldments,"** *Jinmyun Jo*

Experiment plans have been completed; sample preparation and preliminary characterization using XRD are continuing. Neutron experiments are pending scheduling.

Dr. Jinmyun Jo visited the HTML in July 1992 to discuss his research proposal with Drs. Hubbard and Wang. His proposal involves neutron residual stress mapping on vibratory stress-relieved welded steel plates. The discussion covered feasibility of the experiments as well as detailed plans for execution. Guided by Dr. Wang, Dr. Jo also toured the HTML's neutron mapping facility at the HFIR. A tentative schedule for the neutron experiment was set for November 1992.

A weld assembly, 1/2 x 12 x 12 in., was fabricated using HY-100 metal and MIL-100S-1 filler by gas-metal-arc weld technique. During welding, constraint was used on the weldment.

This assembly will be used for the preliminary measurements of residual stresses. A stress-free specimen, 1/8 x 1/8 x 4 in., was made from the parent HY-100 metal. This specimen will be used to determine the stress-free d-spacing, which will be the reference d-spacing value for determination of strain.

Cummins Engine Company, Inc. User Center: RSUC

Project Title: **"Residual Stress Measurements in Thermal Barrier Coatings,"** *T. Yonushonis*

The residual stresses were measured in plasma-sprayed zirconia TBCs on steel substrates before and after thermal cycling by X-ray and neutron diffraction method. The X-ray results indicate a low level of stress in the surface of the TBC (~3 mm above interface). Neutron diffraction measurements show maximum residual stress levels of ~100 MPa in the steel just below the interface. The results were compared with thermal imaging data to determine if "hot spots" in thermal images were correlated with poor adhesion of the TBCs. Preliminary results do not support the hypothesis. The results of this study are being used to guide the R&D effort at Cummins Engine Company to improve TBCs for diesel engines.

University of Florida User Center: RSUC

Project Title: **"Measurement of Residual Stresses in CVD Polycrystalline Diamond Films with X-Ray Diffraction,"** *J. J. Mecholsky, Jr., and L. Hehn*

Diamond film properties are dependent on residual stresses and texture within the film. Models based on thermal expansion differences between diamond and the substrate are believed to overestimate the resulting residual stress. Measurements at the HTML were conducted to determine the magnitude and sign of the residual stress in diamond films. Initial measurements revealed a small but measurable compressive stress and emphasized the critical importance for both alignment and for increasing rate of analysis, e.g., with use of a PSD. The study was interrupted by generator failure, and a new date will be scheduled during the next year for completion of this project.

University of Florida User Center: RSUC

Project Title: **"Measurement of Residual Stress in Ceramic/Metal Laminate Composites with X-Ray Diffraction,"** *J. J. Mecholsky, Jr., and L. Hehn*

Residual stresses in a ceramic/metal laminate composite result primarily from thermal expansion differences between the two phases. A laminated composite consisting of alternating layers of α-Al_2O_3 and Ni was prepared and subsequently aged at 700, 900, and 1000°C to test for stress relaxation. Triaxial stresses were observed in the Al_2O_3 layer instead of the predicted biaxial stress state. No change in stress was observed with samples aged at these temperatures. The stresses perpendicular to the laminate plane were determined to be due to interpenetration of the two phases at their interface. The residual stress in the plane was about 120 MPa, compressive, limited by the yield stress of Ni.

<u>University of Missouri-Columbia</u> User Center: RSUC

<u>Project Title</u>: **"Measurements of Residual Stress Distribution of Stainless Steel Workpiece Machined by CNC Turning Center,"** *D. Y. Jang*

The purpose of this experimental work is to determine the residual stresses resulting from various machining operations and to optimize the machining parameters to yield the desired surface finish and residual stress state. A number of samples of stainless steels were machined under variable conditions. X-ray residual stress measurements of the strain in the lattice have begun. From these data, we found large triaxial stresses that vary significantly with machine speed, depth of cut, type of tool, etc.

6. CERAMIC MANUFACTURABILITY CENTER (CMC) - T. O. MORRIS

A new center was created in the HTML Facility during this report period. This center will address the needs of the U.S. machine tool industry.

One of the three research groups that has occupied the HTML has been moved to another location at ORNL to provide working space for this new center. To date, several pieces of equipment from the Y-12 Facility have been moved into the HTML. These support instruments will be modified to address the research capabilities needed for projects that will be conducted in the CMC.

The CMC will be staffed by both Y-12 and ORNL employees. In addition to the Group Leader, the following staff have been identified to work with HTML Users and Cooperative Research and Development Agreement (CRADA) partners:

Mr. L. O'Rourke (Y-12)
Mr. R. Parten (also works in CSPUC)
Mr. E. Sheldon (also works in CSPUC)

The three most critical needs, as identified by a recent analysis conducted at ORNL with U.S. ceramic companies, are: (1) a more basic understanding of the grinding process and other high-speed materials removal processes, (2) automatic in-process inspection of both the work piece being machined and the cutting component (grinding wheel, cutting tool, abrasive jet, etc.) and feedback and control systems on the machine tool (which automatically brings the work piece to the required dimensions); and (3) overall system statistical process control.

To reduce the relatively high ceramic component machining costs quickly, it is necessary to create an environment in which a broad range of people skilled in ceramic manufacturing and in machining difficult materials, such as special metal alloys, can work together to specifically define the exact nature of the data and information needed to fulfill these industrial needs. The new CMC was created to provide this environment. This facility is being located in the HTML because of the close proximity of the CMC to the staff and the large array of materials characterization instruments located in the present six HTML

User Centers. The staff of the CMC will work with U.S. companies via special working arrangements called CRADAs and HTML User Agreements, to identify specific ceramic manufacturing problems of highest priority, and to mutually develop appropriate solutions to the problems. Most of this work will be specifically targeted at making major improvements in the ability to machine and dimensionally characterize structural ceramics. The full capabilities and expertise of the facilities at ORNL and Y-12 are available to use within this framework, but the actual R&D work required for the solutions to the identified ceramic problems will be conducted in the CMC.

The CMC will be equipped with specialized state-of-the-art material removal equipment and with dimensional characterization instruments. This equipment complements the existing materials characterization and surface characterization instruments already available in the HTML.

The HTML was honored with a visit from President George Bush in February 1992. The President witnessed the signing of a CRADA between Martin Marietta Energy Systems, Inc., and Coors Ceramics Company. The research covered under this agreement is being done at the Y-12 Facility and the CMC. Detroit Diesel Corporation and Lanxide Corporation have also signed CRADAs to perform research in the CMC.

7. HTML FELLOWSHIP PROGRAM

In late FY 1992, the HTML Fellowship Program was approved and a budget provided through the Office of Transportation Technologies, Conservation and Renewable Energy. The first two fellows have been identified and will start their research projects in the HTML during the first months of FY 1993.

The first Industrial Fellow is Suzanne Raebel (Cummins Engine Company, Columbus, Indiana). A Ph.D. graduate of the University of Wisconsin-Madison, she has been investigating wear and corrosion mechanisms in a number of diesel-engine fuel systems as well as plasma-spray processing as applied to bearing linings. Use of the HTML instruments will enable Raebel to conduct the surface chemistry and topography experiments needed to understand the mechanisms causing degradation of the diesel-engine components. In particular, she will analyze (1) surface chemistry by using scanning Auger electron spectroscopy and XPS and (2) surface topography by using AFM.

The first Graduate Fellow is Alex Cozzi, University of Florida—Gainesville. After earning a B.S. degree in Ceramic Engineering from Alfred University and an M.S. degree in the same field from the University of Florida, Cozzi has now completed the necessary course work for a Ph.D. degree and is starting his dissertation, which includes the synthesis, sintering, and determination of the mechanical properties of $BaO\text{-}Al_2O_3\text{-}2SiO_2$ ceramic materials. Powders of this potential structural ceramic material will be synthesized by using sol-gel techniques and sintered by both conventional heating and microwave processing. The resultant materials will be analyzed by several techniques: room-temperature and high-temperature XRD will determine phase composition, high-resolution transmission and analytical electron microscopy will determine crystallite size and volume fraction of phases, and high-resolution thermal analysis will identify and characterize phase reactions. This research is expected to require about two years. Professor David Clark is Cozzi's dissertation advisor at the University of Florida.

A detailed description of this Fellowship Program is given here.

7.1 JUSTIFICATION

There are a variety of needs in the education of U.S. technical personnel, which have a direct bearing on the future success of the U.S. DOE's programs, and the global competitiveness of U.S. companies. It is important to increase the number of highly qualified materials scientists and engineers required to conduct the advanced R&D necessary in the future. A majority of these researchers will have doctorate degrees.

In addition, in the past decade, the research instrumentation required to perform leading-edge materials research has become highly sophisticated and expensive. This situation has made it increasingly difficult for universities to obtain and maintain such instruments, which are so important to graduate materials education. It is also very difficult for universities to have highly skilled staff available to operate and maintain these facilities. In many cases, the attractiveness of a given university department for graduate study in advanced materials is impaired by lack of these expensive state-of-the-art research instruments for use by graduate students for their dissertation research. A major function of the HTML Fellowship Program is to strongly encourage and assist U.S. citizens to pursue graduate training in the area of ceramic education by means of stipends and easy access to modern materials research instruments in the User Centers of the HTML.

7.2 SPECIFIC PURPOSES AND OBJECTIVES OF THE PROGRAM

Graduate: Improve the number and quality of highly trained ceramic materials research personnel needed for successful accomplishment of DOE R&D programs in the United States.

Industrial: Improve the transfer of knowledge and communication between the industrial, university, and national laboratory research staffs.

Postdoctoral: Improve the quality and output of DOE research programs at the national laboratories, universities, and private companies.

Faculty: Increase the understanding of U.S. university faculty of DOE research program objectives and needs.

7.3 SPECIFIC GOALS OF THE FOUR TYPES OF FELLOWSHIPS

A. DOE HTML User Facility Graduate Fellowship: Increase the output of U.S. citizen doctoral graduates from U.S. universities in advanced ceramics of direct interest to the DOE's mission and goals via student support during their graduate training. These appointments will initially be for one, two, three, or four years.

B. HTML User Facility/Industrial Fellowship: Increase interactions between the research staffs of the User Centers of the HTML and U.S. industry by providing certain support for industrial researchers to perform research in the HTML or for HTML user research staff to spend time conducting research in an industrial laboratory. These appointments will vary from one to six months.

C. HTML Postdoctoral Fellowship: Increase the technical quality and output of DOE research programs at universities, in companies, and in the HTML User Centers via temporary support of highly qualified individuals who are U.S. citizens to conduct research in ongoing DOE research projects. These appointments will be for either one or two years.

D. HTML University/HTML User Facility Faculty Fellowship: Increase the understanding of university faculty of the needs, goals, and objectives of DOE R&D programs by providing faculty support for research in the HTML user facilities. These appointments will vary from one month to one year.

7.4 CRITERIA FOR FELLOWSHIP SELECTION

A. DOE HTML User Facility Graduate Fellowship: Must be a U.S. citizen who has received a Bachelor of Science (or B.A.) degree, achieved an undergraduate grade-point average of at least 3.5 in a system where 4.0 = A, and been accepted into the graduate program of a U.S. university in a degree program directly related to one of the "DOE Materials Technical Areas."

B. HTML User Facility/Industrial Fellowship: Must be an employee of a U.S. company which has either existent or planned commercial activities of direct interest to DOE programs. Further, the candidate must have a minimum of a B.S. degree in a technical field directly related to one or more "DOE Material Technical Areas" and must have performed research for at least one year in an area of direct interest to DOE programs.

C. HTML Postdoctoral Fellowship: Shall be a U.S. citizen, have a Ph.D. or equivalent degree in an academic discipline directly related to one or more of the "DOE Material Technical Areas," and shall either have achieved a graduate grade-point average of at least 3.2 in a system where 4.0 = A and/or have demonstrated research ability in one of the "DOE Material Technical Areas."

D. HTML University/User Facility Faculty Fellowship: Must be a full-time academic staff member of a university department whose educational mission includes one or more of the "DOE Material Technical Areas." During the preceding year, the candidate must have personally taught both undergraduate and graduate courses in the technical specialty, and a statement to this effect is to be provided, including course titles and descriptions, by the applicant's department head.

7.5 CONDITIONS OF THE FELLOWSHIP PROGRAM

A. DOE HTML User Facility Graduate Fellowship: Can have a duration of one to four years.

B. HTML User Facility/Industrial Fellowship: Can have a duration from one to six months. Its purpose is to enhance technology transfer and communication in the HTML user facilities between research staff and that of U.S. companies by having industrial researchers spend extended time in the HTML, or having HTML user research staff conduct research on a project at a U.S. company site.

C. HTML Postdoctoral Fellowship: Has a duration of one or two years. Its purpose is to supplement research activities in HTML User Facilities on a temporary basis.

D. HTML University/User Facility Faculty Fellowship: Is directed to both junior and senior faculty who teach and advise students in ceramic engineering or materials science. Its purpose is to increase understanding and appreciation of the technical needs and goals of DOE programs by having the fellowship research be conducted in the HTML User Centers with national laboratory or industrial research staff who are working on these problems. This fellowship can have a duration of one month to one year.

7.6 MATERIALS TECHNICAL AREAS OF MAJOR INTEREST TO DOE PROGRAMS

The four types of HTML User Facility fellowships all have the performance of ceramic materials research as a major component. The HTML Graduate Fellowships also have the formal education of graduate students in advanced ceramics (including thesis or dissertation research) as a major objective. It is intended that these research and educational activities be focused and related to technical and scientific needs in DOE programs, and be oriented around the DOE HTML User Facilities. This orientation will increase the utilization of the HTML User Facilities for both national technical educational and industrial competitiveness purposes. With these objectives in mind, the following areas are identified as being of major interest and applicability to DOE ceramics programs. Where possible, it is anticipated that the research conducted on the fellowships be in one or more of the listed "DOE Material Technical Areas."

DOE Ceramic Material Technical Areas:

1. Mechanical Properties of Ceramics at High Temperatures
2. Physical and Electrical Properties of Ceramics at High Temperatures
3. Processing of Ceramics
4. Tensor Mechanics of Solids
5. Finite Element Computation Methods for Stress Analysis
6. Tribology of Solid Surfaces at Elevated Temperatures
7. High-Temperature Gas-Phase Reactions with Solids
8. Heat Transfer through Solids and Gases
9. Corrosion of Ceramics at High Temperatures
10. Structure of Ceramics

APPENDIX A

LISTING OF THE ADVISORY COMMITTEES

HTML Advisory Committee Members

	Term Expires
Mr. Woodie Howe Coors Technical Ceramics Company 1100 Commerce Park Drive Oak Ridge, Tennessee 37830	1992
Professor John J. Hren Head, Materials Science and Engineering North Carolina State University Box 7907, Yarborough Drive Raleigh, North Carolina 27695-7907	1992
Dr. Maxine L. Savitz, Director Ceramic Components Division Garrett Processing Company 19800 South Van Ness Ave. Torrance, California 90509	1993
Dr. James W. Patten Director, Materials Engineering Cummins Engine Company 500 Jackson Street Columbus, Indiana 47201	1994
Dr. Carr Lane Quackenbush Bicron 12345 Kinsman Road Newbury, Ohio 44065	1994

HTML User Advisory Committee Members

Dr. V. J. Tennery, Director of the HTML, is permanent Chairman of this Committee.

	Term Expires
Mr. Lance Groseclose Allison Gas Turbine Operations General Motors Corporation Post Office Box 420 Indianapolis, Indiana 46206-0420	1992
Dr. Linda Horton Metals and Ceramics Division Oak Ridge National Laboratory P. O. Box 2008 Oak Ridge, Tennessee 37831	1992
Dr. Thomas J. Whalen Ford Motor Company 26362 Harriet Dearborn Heights, Michigan 48127	1993
Ms. Mary Harris Oak Ridge Field Office U.S. Department of Energy Oak Ridge, Tennessee 37831	1994
Dr. Norman L. Hecht The University of Dayton Research Institute 300 College Park Dayton, Ohio 45469-0001	1994

APPENDIX B

HIGH TEMPERATURE MATERIALS LABORATORY USER PROGRAM

THIRD USERS GROUP MEETING

FRIDAY, SEPTEMBER 25, 1992

OAK RIDGE, TENNESSEE

Mr. A. A. Chesnes, Assistant Deputy Secretary, Office of Transportation Technologies, U.S. Department of Energy, sponsor of the HTML User Program, attended the third HTML Users Group meeting, which was held in the HTML on Friday, September 25, 1992, along with Dr. S. Diamond, Manager for the HTML User Program at DOE Headquarters.

In addition to approximately 45 Energy Systems employees who attended the meeting, there were attendees from 20 outside user institutions represented by 26 individual researchers (12 researchers from 8 universities and 14 researchers from 12 industrial companies). Titles of the presentations given during the morning session are listed below:

> "*Characterization of Vapor Liquid Solid (VLS) Silicon Carbide Fibrils,*" William E. Hollar, Jr., Carborundum Company;
>
> "*High-Temperature Toughness of SiCw/Al_2O_3 Composites,*" A. A. Wereszczak, University of Delaware/HTML Staff;
>
> "*Status of Ceramic Manufacturability Center,*" V. J. Tennery, Director, HTML;
>
> "*Crystallization Behavior of Precursors to Forsterite and Cr-Doped Forsterite,*" M. Hogan E. Martin, Cornell University; and
>
> "*Summary of Research in the Residual Stress User Center,*" C. R. Hubbard, HTML Staff.

Other activities on the agenda included a ceremony, witnessed by the news media, to announce the signing of a Cooperative Research and Development Agreement with Lanxide Corporation.

In addition, a special recognition ceremony was held in honor of Mr. Chesnes, who had announced earlier in the week that he would retire on October 2, 1992. Acknowledgment of his invaluable support of the HTML User Program and other ceramic programs at the Oak Ridge National Laboratory, was made at this time. Several members of ORNL management were on hand to thank him for his contribution to ceramic research.

APPENDIX C

HTML CUMULATIVE USER EXPERIENCE

Figure C.1 illustrates the cumulative user days for industry, university, and local users in the HTML User Program for the entire 21 quarters of operation to date. Approximately 56% of the cumulative user days have been from local researchers, while about 34 have been from industry and 10% have been from universities. (For FY 1992, use by local researchers is 50%; by industry researchers 40%; and by university researchers 10%.)

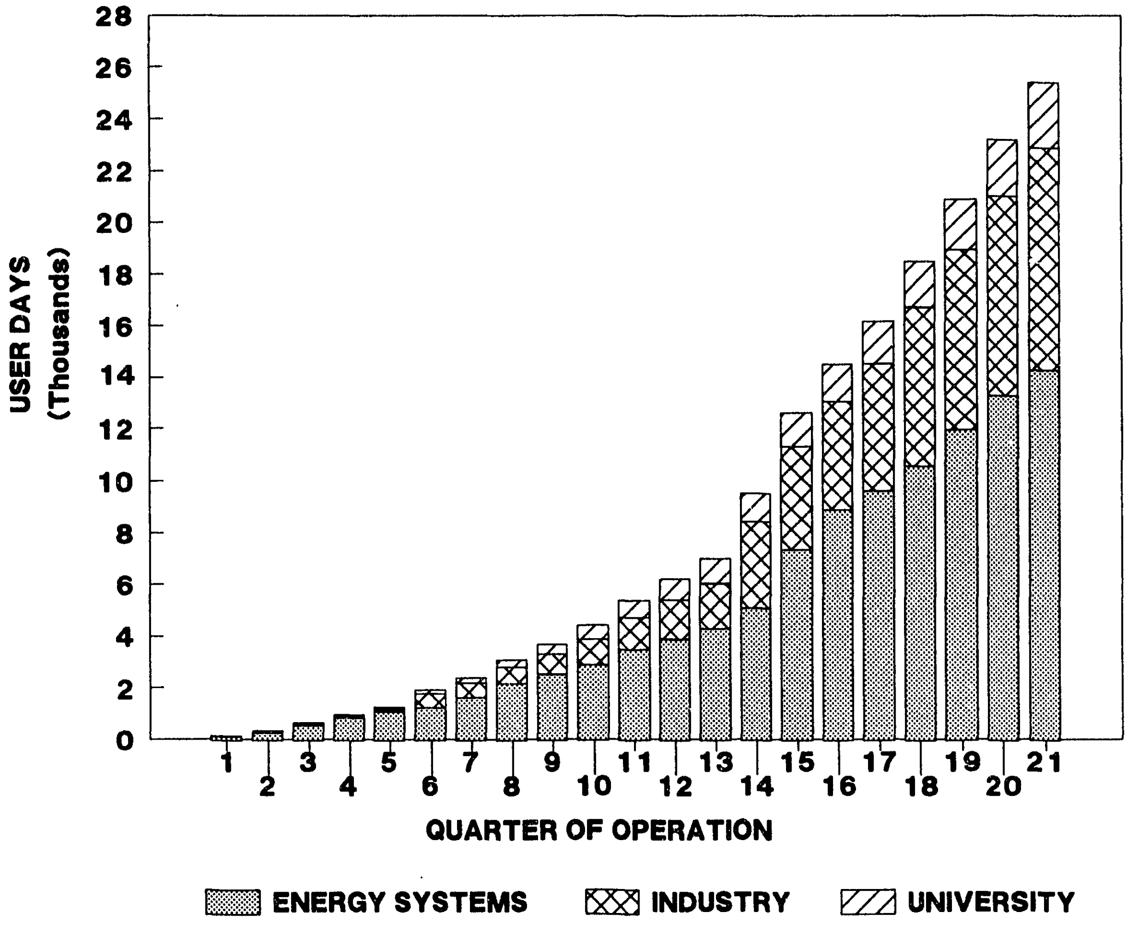

Fig. C.1. Cumulative user days in the HTML User Program.

APPENDIX D

PUBLICATIONS AND PRESENTATIONS

User Center Staff are indicated by an underline. The User's home institution is listed at the end of the citation. These citations are listed in order of the name of the User Institution.

1. INDUSTRY USERS PUBLICATIONS AND PRESENTATIONS

W. E. Hollar, Jr., "Characterization of Vapor Liquid Solid (VLS) Silicon Carbide Fibrils," presented at the HTML User Group Meeting, September 25, 1992. CARBORUNDUM COMPANY

R.-R. Lee, B. E. Novich, G. V. Franks, D. Ouellette, M. K. Ferber, C. R. Hubbard, and K. L. More, "Mechanical Properties and Microstructure of Pressureless Sintered Duophase Sialon," presented at the 4th International Symposium on Ceramic Materials and Components for Engines, and published in the proceedings edited by R. Carlson, T. Johannson, and L. Kahlman, Elsevier Applied Science, 1992. CERAMICS PROCESS SYSTEMS CORPORATION

B. E. Novich, R.-R. Lee, G. V. Franks, D. Ouellette, and M. K. Ferber, "Fabrication of Low-Cost and High-Performance Ceramic Gas Turbine Engine Components," presented at the Annual Automotive Technology Development Contractors' Coordination Meeting, Dearborn, Michigan, October 22-25, 1990; published in the proceedings of the meeting, pp. 111-123. CERAMICS PROCESS SYSTEMS CORPORATION

C. R. Hubbard, O. B. Cavin, and J. Ghinazzi, "HRXRD Study of the Phase Evolution During Firing of Green Alumina," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16 1992. COORS TECHNICAL CERAMICS

C. R. Hubbard, O. B. Cavin, R. A. Newman, and A. K. Knudsen, "High-Speed, High-Temperature XRD Data Collection Using a Position-Sensitive Detector," presented at the Denver X-ray Conference, Colorado Springs, Colorado, August 1992; to be published in the proceedings of the conference. DOW CHEMICAL COMPANY

G. E. Potter, A. K. Knudsen, J. C. Tou, and A. Choudhury, "Measurements of the Oxygen and Impurity Distribution in Polycrystalline Aluminum Nitride with Secondary Ion Mass Spectrometry," to be published in *J. Am. Ceram. Soc.* DOW CHEMICAL COMPANY

M. K. Ferber, M. G. Jenkins, T. A. Nolan, and R. Yeckley, "Creep-Fatigue Response of Structural Ceramics: I, Comparison of Flexure, Tension, and Compression Testing," presented at the Sagamore Conference on Structural Ceramics, October 1990. NORTON COMPANY

4B. Harkins, "High-Pressure Heat Exchange Systems, Phase II, Second Annual Report, May 1991 to May 1992," published by Solar Turbines, September 1992. SOLAR TURBINES INCORPORATED

B. Harkins and M. E. Ward, "Preliminary Design for an Advanced Ceramic Air Heat Exchanger Integrated in a Gas Turbine Power Generation Cycle," published by Solar Turbines, November 1991. SOLAR TURBINES INCORPORATED

V. Parthasarathy, B. Harkins, W. Beyermman, J. Keiser, W. Elliott Jr., and M. K. Ferber, "Evaluation of SiC/SiC Composites for Heat Exchanger Applications," January 1992, published in *Ceram. Eng. Sci. Proc.*, p. 503 (July-August 1992). SOLAR TURBINES INCORPORATED

2. UNIVERSITY USERS PUBLICATIONS AND PRESENTATIONS

B. J. Reardon and C. R. Hubbard, "Using Simulated XRD Patterns in New Materials Analysis," presented at the American Ceramic Society 94th Annual Meeting and Exposition 94th Annual Meeting, Minneapolis, Minnesota, April 12-16, 1992. ALFRED UNIVERSITY

B. J. Reardon, C. R. Hubbard, and O. B. Cavin, "The $CaCu_2O_3$-$SrCu_2O_3$ Pseudo-Binary System at 1223K and 1263K," to be published in *J. Mater. Res.* ALFRED UNIVERSITY

B. J. Reardon and C. R. Hubbard, "A Review of the XRD Data of the Phases Present in the CaO-SrO-PbO System," published in *Powder Diffr.* 7(2), 96-99 (June 1992). ALFRED UNIVERSITY

B. J. Reardon and C. R. Hubbard, "A Review of the XRD Data of the Phases Present in the CaO-SrO-CuO System," published in *Powder Diffr.* 7(2), 96-99 (June 1992). ALFRED UNIVERSITY

B. J. Reardon and C. R. Hubbard, "A Comprehensive Review of the XRD Data of the Primary and Secondary Phases Present in the BSCCO Superconductor System (Part I: Ca-Sr-Cu Oxides)," Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., ORNL/TM-11948, January 1992. ALFRED UNIVERSITY

B. J. Reardon and C. R. Hubbard, "A Comprehensive Review of the XRD Data of the Primary and Secondary Phases Present in the BSCCO Superconductor System (Part II: Ca-Sr-Cu Oxides)," Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab, ORNL/TM-11949, February 1992. ALFRED UNIVERSITY

M. Grujicic, S. Tangril, O. B. Cavin, and C. R. Hubbard, "Effect of Iron Additions on Structure of Laves Phases in Nb-Cr-Fe Alloys," to be published in *Mater. Sci. Eng. A*. CLEMSON UNIVERSITY

J. S. Wolf, O. B. Cavin, and J. H. DeVan, "The Oxidation of Type 310S Stainless Steel in Mixed Gases at Elevated Temperatures," Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., ORNL/TM-11887, April 1992. CLEMSON UNIVERSITY

D. K. Peeler, T. D. Taylor, and D. R. Dinger, "The Effect of Initial Particle Size Distributions on the Melting Kinetics of Soda-Lime-Silica Glasses," to be published in *Trans. Am. Ceram. Soc.* CLEMSON UNIVERSITY

O. B. Cavin and J. S. Wolf, "X-Ray Examination of Type 310S Stainless Steel During Its Oxidation in Air at 900°C," presented at the 38th Annual Denver X-Ray Conference, Denver, Colorado, July 31 - August 4, 1992; published in *Adv. X-Ray Anal.* 36. CLEMSON UNIVERSITY

M. H. E. Martin, "Crystallization Behavior of Precursors to Forsterite and Cr-Doped Forsterite," presented at the HTML User Group Meeting, September 25, 1992. CORNELL UNIVERSITY

T. E. Schlesinger, R. C. Cammarata, A. Gavrin, J. Q. Xiao, C. L. Chien, M. K. Ferber, and C. Hayzelden, "Enhanced Mechanical and Magnetic Properties of Granular Metal Thin Films," to be published in *J. Appl. Phys.* JOHN HOPKINS/HARVARD UNIVERSITY

A. Vasudev, K. L. More, K. S. Ailey-Trent, and R. F. Davis, "Kinetics and Mechanisms of High-Temperature Creep in Polycrystalline Aluminum Nitride," submitted to *Journal of Materials Research* (1992). NORTH CAROLINA STATE UNIVERSITY

G. R. Fox, S. B. Krupanidhi, K. L. More, and L. F. Allard, "Composition/Structure/Property Relations of Multi-Ion-Beam Reactive Sputtered Lead Lanthanum Titanate Thin Films: Part I, Composition and Structure Analysis," submitted to *J. Mater. Res.* PENNSYLVANIA STATE UNIVERSITY

G. R. Fox, S. B. Krupanidhi, K. L. More, and L. F. Allard, "Microstructure Property Relations of Lead Lanthanum Titanate Thin Films," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16, 1992. PENNSYLVANIA STATE UNIVERSITY

C. A. Randall, T. R. Shrout, A. S. Bhalla, L. E. Cross, and L. F. Allard, "A High-Resolution Electron Microscopy Study of Ordering in Complex Lead Perovskites," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16, 1992; to be published in the proceedings of the conference. PENNSYLVANIA STATE UNIVERSITY

E. Lara-Curzio, S. S. Sternstein, C. R. Hubbard, O. B. Cavin, and W. D. Porter, "High-Temperature Structural Stability of Chemical Vapor-Deposited Silicon Carbide Fibers," to be published in *J. Am. Ceram. Soc.* RENSSELAER POLYTECHNIC INSTITUTE

T. Dolney and A. Choudhury, "Data Processing of 3-D SIMS Ion Images on a Macintosh IICX," presented at the American Association of Physics Teachers - Winter Meeting, Orlando, Florida, January 4, 1992; published in the proceedings of the meeting (January 1992). SAM HOUSTON STATE UNIVERSITY

K. L. More, V. J. Tennery, and N. Hecht, "Microstructural Evolution During the Tensile Static and Cyclic Fatigue of Silicon Nitride," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16, 1992; to be published in the proceedings of the meeting. UNIVERSITY OF DAYTON RESEARCH INSTITUTE

A. A. Wereszczak, "High-Temperature Toughness of SiCw/Al_2O_3 Composites," presented at the HTML User Group Meeting, September 25, 1992. UNIVERSITY OF DELAWARE

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C.-K. Lin and D. Socie, "Static and Cyclic Fatigue of Alumina at Room and High Temperatures," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16, 1992; to be published in the proceedings of the meeting. UNIVERSITY OF ILLINOIS

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J. M. Stencel, A. Sethuraman, R. E. Gonzales, X.-X. Bi, A. Rubel, F. J. Derbyshire, P. C. Eklund, and C. R. Hubbard, "Ultrafine Particle Iron Carbide Catalysts Synthesized by Laser Pyrolysis: Physical and Chemical Transformations at High Temperatures and Under Sulfiding Conditions," presented at the 1991 Materials Research Society Fall Meeting, Boston, Massachusetts, November 1991; published in the proceedings of the meeting. UNIVERSITY OF KENTUCKY

R. Srinivasan, B. H. Davis, O. B. Cavin, and C. R. Hubbard, "Crystallization and Phase Transformation Process in Zirconia: An In Situ High-Temperature X-Ray Diffraction Study," submitted to *J. Am. Ceram. Soc.* UNIVERSITY OF KENTUCKY

J. B. Bates, N. J. Dudney, G. R. Gruzalski, R. A. Zuhr, A. Choudhury, C. F. Luck, and J. D. Robertson, "Electrical Properties of Amorphous Lithium Electrolyte Thin Films," to be presented at the 8th International Conference on Solid State Ionics, Lake Louis, Canada, October 20-26, 1992. UNIVERSITY OF KENTUCKY

X. Qiu, A. K. Datye, R. T. Paine, and L. F. Allard, "The Oxidation Stability of Boron Nitride Thin Films on MgO and TiO_2 substrates," pp. 275-79 in *Proceedings of the Materials Research Society Symposium*, held at Boston, Massachusetts, December 2-6, 1991, Vol. 250, Materials Research Society, Pittsburgh, 1992. UNIVERSITY OF NEW MEXICO

T. T. Borek, X. Qiu, L. M. Rayfuse, A. K. Datye, and R. T. Paine, "Boron Nitride Coatings on Oxide Substrates: Role of Surface Modifications," published in *J. Am. Ceram. Soc.*, 2587-2591 (1991). UNIVERSITY OF NEW MEXICO

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N. B. Dahotre, M. H. McCay, T. D. McCay, C. R. Hubbard, and W. D. Porter, "Effect of Grain Structure on Phase Transformations in the Inconnel 718: A Differential Thermal Analysis Approach," submitted to *Phys. Status Solidi.* UNIVERSITY OF TENNESSEE

M. J. Godbole, A. J. Pedraza, L. F. Allard, and G. Geesey, "Characterization of Sputter-Deposited 316L Stainless Steel Films," published in *J. Mater. Sci.* 27 (1992). UNIVERSITY OF TENNESSEE

H. L. Webb and O. B. Cavin, "Discovery of Kolbechite in Georgia - Two Possible Lattices Suggested," published in Southeastern Geology 32(1), 21-28 (July 1991). UNIVERSITY OF TENNESSEE

A. Choudhury, C. L. White, and C. R. Brooks, "The Intergranular Segregation of Boron in Ni_3Al: Part 2, Segregation Kinetics," *Acta Metall. Mater.* 40(1), 57-68 (1992). UNIVERSITY OF TENNESSEE

Y. Zhang, R. D. Bunn, D. C. Joy, X. Zhang, L. F. Allard, T. A. Nolan, and T. Hashimoto, "Software for Electron Holography," presented at the Frontiers of Electron Microscopy in Materials Science, Oakland, California, April 21-24, 1992; to be published in *Ultramicroscopy*. UNIVERSITY OF TENNESSEE

A. Choudhury, C. D. Lundin, C. R. Brooks, and K. K. Khan, "Scanning Auger Spectroscopy of Reheat Crack Surfaces of Cr-Mo and HSLA Steel Weld HAZs," presented at the 25th Annual Convention - International Metallographic Society, Denver, Colorado, August 2-5, 1992. UNIVERSITY OF TENNESSEE

A. Choudhury, D. L. Joslin, C. W. White, C. J. McHargue, and L. J. Romana, "XPS Characterization of Ion Implanted Oxide Films on Sapphire," presented at the American Ceramic Society 94th Annual Meeting and Exposition, Minneapolis, Minnesota, April 12-16, 1992. UNIVERSITY OF TENNESSEE

D. C. Joy, X. Zhang, Y. Zhang, T. Hashimoto, L. F. Allard, and T. A. Nolan, "Practical Aspects of Electron Holography," presented at the Frontiers of Electron Microscopy in Materials Science, Oakland, California, April 21-24, 1992; to be published in *Ultramicroscopy*. UNIVERSITY OF TENNESSEE

L. F. Allard, T. A. Nolan, D. C. Joy, and T. Hashimoto, "Digital Imaging for High-Resolution Electron Holography," presented at the Electron Microscopy Society of America, Boston, Massachusetts, August 16-20, 1992; published by San Francisco Press. UNIVERSITY OF TENNESSEE

D. C. Joy, X. Zhang, Y. Zhang, T. Hashimoto, L. F. Allard, and T. A. Nolan, "Applications of Electron Holography to Materials Science," presented at Micro 1991, London, England, July 7-11, 1992. UNIVERSITY OF TENNESSEE

L. F. Allard, T. A. Nolan, D. C. Joy, T. Hashimoto, X. Zhang, and Y. Zhang, "Digital Imaging for High-Resolution Electron Holography," presented at the Frontiers of Electron Microscopy in Materials Science, Oakland, California, April 21-24, 1992; to be published in *Ultamicroscopy*. UNIVERSITY OF TENNESSEE

3. HTML AND LOCAL USERS PUBLICATIONS

L. F. Allard and D. W. Coffey, "Structure and Morphology of Carborundum VLS Silicon Carbide Whiskers," presented at the Carborundum Company Seminar, Niagara Falls, New York, April 2, 1992.

L. F. Allard, "Electron Holography: Techniques and Perspectives for Materials Science," presented at the North Carolina State University-Charlotte, Charlotte, North Carolina, February 29, 1992.

L. F. Allard, "Electron Holography: Techniques and Perspectives for Materials Science," presented at Mount Holyoke College, Holyoke, Massachusetts, December 4, 1991.

L. F. Allard, F. J. Walker, and R. A. McKee, "MBE Growth of Epitaxial Perovskites on Silicon: The Atomic Structure of $BATIO_3$/BAO Interfaces by High-Resolution Electron Microscopy," presented at the 1991 Materials Research Society Fall Meeting, Boston, Massachusetts, December 2-6, 1991; published in the proceedings of the meeting (December 1991).

L. F. Allard, T. A. Nolan, and T. N. Tiegs, "The Nature of Planar Defects in Silicon Carbide Whiskers," submitted to *J. Mater. Res.*

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4. PENDING PRESENTATIONS

L. F. Allard, W. E. Hollar, D. W. Coffey, and R. A. Lowden, "The Ultrastructure of Bulk SiC Fibrils Grown by VLS Methods," to be presented at the American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. CARBORUNDUM COMPANY

S. C. Beecher and R. B. Dinwiddie, "Modeling the Thermal Conductivity of Fiber-Reinforced Ceramic Composites," to be presented at the 17th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 10-15, 1993.

S. C. Beecher and R. B. Dinwiddie, "The Thermal Conductivity of Fiber-Reinforced Ceramic Composites," to be presented at the American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

R. B. Dinwiddie, "The Scanning Thermal Conductivity Microprobe: Theoretical Treatment," to be presented at the 17th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 10-15, 1993.

R. B. Dinwiddie, S. C. Beecher, D. A. Bowers, and J. W. Sapp, "Thermal Conductivity of 1-D Carbon Composite Constituents after Processing," to be presented at the American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. MCDONNELL DOUGLAS

M. K. Ferber, "Creep and Fatigue Behavior of a HIPed Silicon Nitride," to be presented at 45th Pacific Coast Regional Meeting, San Francisco, California, November 1-4, 1992.

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C. R. Hubbard, S. A. David, X.-L. Wang, and S. Spooner, "Nondestructive Residual Stress Mapping in Ceramic-To-Metal Joints," to be presented at the 17th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 10-15, 1993.

C. R. Hubbard, X.-L Wang, S. Spooner, S. David, and T. A. Dodson, "Residual Stress Mapping Throughout a Brazed Fe-ZrO_2 Sample by Neutron and X-ray Diffraction," to be presented at American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

D. K. Peeler, T. D. Taylor, and O. B. Cavin, "Melting Kinetics of Soda-Lime-Silica Glasses by In Situ High-Temperature X-Ray Diffraction," to be presented at the American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. CLEMSON UNIVERSITY

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