MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0351887 9

ornl

ORNL/TM-12023

OAK RIDGE NATIONAL LABORATORY

MARTIN MARIETTA

High Temperature Materials Laboratory Fourth Annual Report (October 1990 Through September 1991)

V. J. Tennery
F. M. Foust

OAK RIDGE NATIONAL LABORATORY
CENTRAL RESEARCH LIBRARY
CIRCULATION SECTION
4500N ROOM 175
LIBRARY LOAN COPY
DO NOT TRANSFER TO ANOTHER PERSON
If you wish someone else to see this report, send in name with report and the library will arrange a loan.
NCN-7999-6 5-79

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831; prices available from (615) 576-8401, FTS 626-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, [illegible]

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any [illegible] makes any warranty, express or implied, or assumes any [illegible] liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ORNL/TM-12023

Metals and Ceramics Division

HIGH TEMPERATURE MATERIALS LABORATORY FOURTH ANNUAL REPORT (OCTOBER 1990 THROUGH SEPTEMBER 1991)

V. J. Tennery
F. M. Foust

Date Published: December 1991

Prepared for
U.S. Department of Energy
Assistant Secretary for Conservation and Renewable Energy
Office of Transportation Technologies
EE 51 04 00 0

Prepared by
High Temperature Materials Laboratory
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, TN 37831-6285
managed by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

MARTIN MARIETTA ENERGY SYSTEMS LIBRARIES

3 4456 0351887 9

CONTENTS

INDEX OF RESEARCH PROJECT SUMMARIES v

ABSTRACT 1

1. INTRODUCTION 1

2. COMMITTEE ACTIVITIES 2

3. USER AGREEMENTS 2

4. USERS 3

5. USER CENTER CAPABILITIES, INSTRUMENTS, RESEARCH PROJECT SUMMARIES 8

5.1 MATERIALS ANALYSIS USER CENTER (MAUC) 8
5.2 MECHANICAL PROPERTIES USER CENTER (MPUC) 15
5.3 PHYSICAL PROPERTIES (PPUC) AND X-RAY DIFFRACTION (XRDUC) USER CENTER 23
5.4 CERAMIC SPECIMEN PREPARATION USER CENTER (CSPUC) 27
5.5 RESIDUAL STRESS USER CENTER (RSUC) 28

6. HONORS, AWARDS, AND PRESENTATIONS 29

APPENDIX A. LISTING OF THE ADVISORY COMMITTEES A-1

APPENDIX B. SECOND USERS GROUP MEETING B-1

APPENDIX C. HTML CUMULATIVE USER EXPERIENCE C-1

APPENDIX D. PUBLICATIONS AND PRESENTATIONS D-1

INDEX OF RESEARCH PROJECT SUMMARIES

Research projects with significant activity during FY 1991 are listed by title, user institution, and the User Center involved. A brief summary is on the pages indicated.

PROJECTS OF INDUSTRIAL USER COMPANIES

Allied-Signal Aerospace Co.: "Determination of the High-Temperature Static Fatigue Limit in High Strength and High Toughness Silicon Nitride," MPUC 17

Allied-Signal Aerospace Co.: "Microstructural Evaluation of Time-Dependent Failure of NT-154 Silicon Nitride," MPUC 20

Aluminum Company of America: "Interfacial Properties Characterization in Nicalon SiC Fiber/Alumina-Based Composites," MPUC 22

American Superconductor: "Phase Stability of $Bi_2Sr_2Ca_1Cu_2O_x$ Superconductor at High Temperatures," MAUC, PPUC, XRDUC 25

Ceramic Process Systems: "Research on Duophase Sialons with a Controlled Grain-Boundary Phase," MAUC, MPUC, XRDUC 17

Coors Electronic: "Use of Cobalt and Nickel as a Barrier to Diffusion of Nickel through Gold," MAUC 9

Detroit Diesel Corporation: "Identification of Typical Microstructures and Phases of Sintered Silicon Nitride Cam Roller Followers," MAUC, XRDUC 9

DOW Chemical: "Crystallization of Intergranular Phases in AlN with Y_2O_3 Sintering Aids," XRDUC 25

McDonnell Douglas: "High Thermally Conductive Carbon-Carbon Composites," PPUC 24

Norton/TRW: "Thermal Diffusivity Dependence on Silicon Nitride Grain-Boundary Composition and Volume," PPUC 24

Norton/TRW: "High-Temperature Deformation Behavior of NT 154," MAUC, MPUC 18

Norton/TRW: "Characterization of Microstructural Changes Occurring During High-Temperature Deformation of NT 154," MAUC, MPUC 10

Sunstrand Power Systems: "Study of Fast Fracture, Dynamic, and Static Fatigue Behavior of Kyocera SN235 Silicon Nitride," MPUC 18

Teledyne Allvac: "Study of Major Alloying Elements and Morphology of Microstructural Components in Superalloys," MAUC 10

Thermacore, Inc.: "Evaluation of Refractory Metal Coatings Applied to Carbon-Carbon," MAUC 11

PROJECTS OF UNIVERSITY USERS

Clemson University: "Load Deflection Measurements of Silicon Microbeams," MPUC 22

Clemson University: "Study of the Character of Stresses Developed in an Iron-Based Alloy Subjected to Sulfidizing Mixed Gases at Elevated Temperatures, I,"MAUC, XRDUC 25

Clemson University: "Study of the Character of Stresses Developed in an Iron Based Alloy Subjected to Sulfidizing Mixed Gases at Elevated Temperatures, II," MAUC, XRDUC 25

Michigan State University: "Study of Wear Characteristics of Silicon Nitride," MPUC . . 21

New Mexico Institute of Mining and Technology: "Effect of Tin Dioxide Interlayer in Alumina/Glass Composites," MAUC, MPUC 22

New Mexico Institute of Mining and Technology: "Ceramic Particle/Metal Matrix Composites," MAUC 11

North Carolina State University: "High-Resolution Electron Microscopy on the Nucleation of Diamond Film," MAUC 11

Pennsylvania State University: "Microscopy of the Chemical Order Domains in Lead Perovskites," MAUC 11

Pennsylvania State University: "Size Effects of the Paraelectric:Ferroelectric Phase Transition in Ultra-Fine Particle Barium Titanate," MAUC 12

Pennsylvania State University: "Characterization of the Curie Point Transition in Barium Titanate Using Electron Microscopy," MAUC 12

Pennsylvania State University: "Microstructure and Interface Studies of Lead-Lanthanum-Titanate Thin Films," MAUC 12

Rice University: "Development of an Improved Method for Determining Hardness and Elastic Modulus in Thin Films and Small Volumes," MAUC 13

Stanford University: "Hardness and Elastic Modulus Measurements of InGaAs/InP and GexSi1-x/Si Superlattices," MPUC 21

Tuskegee University: "Characterization of Various Al_2O_3/SiC Composites at Room and Elevated Temperatures," MAUC, MPUC 17

University of Delaware: "The Investigation of Interphase Morphology and of an SiC Whisker-Reinforced Alumina Composite Using HREM," MAUC 13

University of Delaware: "Evaluation of Toughness of SiC Whisker-Reinforced Alumina Composites Fractured as a Function of Temperatures and Loading Rate," MPUC 16

University of Kentucky: "Analytical Characterization of Ultrafine Particles," MAUC, XRDUC 26

University of Kentucky: "Crystal Phase Formation in Hf-Zr Mixed Oxides," XRDUC .. 26

University of Michigan: "Characterization of Synthesized SiC Whiskers," MAUC 13

University of Tennessee: "Study of the Fracture Surfaces of Ni-20 at. % Mo Alloys Which Have Been Microalloyed With Various Elements," MAUC 14

University of Tennessee: "Analyses of the Fracture Surfaces of Samples of a High-Temperature Flange Bolt Material," MAUC 14

University of Tennessee: "Study of Interfacial Reaction Phases in Laser-Welded Ceramic Particulate/Al-alloy Metal Matrix Composite," MAUC 14

University of Tennessee: "Study of Thin Films of 316L Stainless Steel Exposed to Microbial Activity," MAUC 15

University of Tennessee: "Analysis of High-Temperature Phase Transformation in Inconel, 718," XRDUC 26

University of Wisconsin: "Supersolidus Investigation of the Bi-Sr-Ca-Cu Oxide System," XRDUC 27

HIGH TEMPERATURE MATERIALS LABORATORY
FOURTH ANNUAL REPORT*
(OCTOBER 1990 THROUGH SEPTEMBER 1991)

V. J. Tennery and F. M. Foust

ABSTRACT

The High Temperature Materials Laboratory has completed its fourth year of operation as a designated Department of Energy User Facility at the Oak Ridge National Laboratory. Growth of the user program is evidenced by the number of outside institutions who have executed user agreements since the facility began operation in 1987. A total of 118 nonproprietary agreements (62 university and 56 industry) and 28 proprietary agreements (2 university, 26 industry) are now in effect. Five other government facilities have also participated in the user program.

Sixty-five nonproprietary research proposals (38 from university, 26 from industry, and 1 other government facility) and four proprietary proposals were considered during this reporting period. Research projects active in FY 1991 are summarized.

---

1. INTRODUCTION

The High Temperature Materials Laboratory (HTML) is a modern research facility which houses an array of special research equipment used to meet research needs for advanced high-temperature materials, including structural ceramics and alloys. The research instruments in the original four 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.

*Research sponsored by the Department of Energy, Assistant Secretary for Conservation and Renewable Energy, Office of Transportation Technologies, as part of the High Temperature Materials Laboratory User Program, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

Two new user centers, (1) X-Ray Residual Stress and (2) Ceramic Specimen Preparation, were authorized in the previous report period. During this year, instruments were identified and purchased for these two centers. Installation of these instruments is nearly complete. There has been in-house activity in the Ceramic Specimen Preparation User Center, and the first users in the X-Ray Residual Stress area are expected in the first half of FY 1992.

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, 146 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. Membership 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 six members who represent the industrial and academic communities. The Committee meets at least annually and more frequently when its advice is urgently required on particular matters. A formal report containing conclusions from a meeting of the Committee is submitted to the Associate Director for Physical Sciences and Advanced Materials of the Oak Ridge National Labortory (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. This committee reviewed 65 nonproprietary research proposals in FY 1991 (November, March, June, and September).

HTML User Exchange Group. The second meeting of this group was held on March 21, 1991. 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 made more effective. The agenda and a short summary of the 1991 meeting are included in Appendix B.

3. USER AGREEMENTS

The two types of standard agreements utilized in the HTML User Program are: (1) "Nonproprietary 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 (2) "Proprietary Agreement" for all projects in which the user desires that the data and results be proprietary. Table 1 is a listing of institutions who have executed nonproprietary user agreements to date.

4. USERS

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

Table 1. Standard User Agreements executed July 15, 1987, through September 30, 1991

UNIVERSITIES - 62

Alfred University
Auburn University
Brown University
Clemson University
Cornell University
Dartmouth College
Georgia Institute of Technology
Illinois Institute of Technology
Johns Hopkins University
Kent State University
Lehigh University
Louisiana State University
Marquette University
Massachusetts Institute of Technology
Michigan State University
Michigan Technological University
NM Institute of Mining and Technology
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
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 Illinois
University of Kentucky
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 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

Table 1. Continued

INDUSTRIES - 54

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

Note: Due to the sensitive nature of many of the proprietary research activities, the names of the user institutions for proprietary agreements are not listed.

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

Table 2. HTML FY 1991 User Experience

Type of user	Number of institutions	Number of individuals	User days
Industry	27	48	3201
University	19	40	674
Local Oak Ridge users	2	90	5309

Figure 1 shows the total number of 8-h user days per quarter during FY 1991. The industrial user days varied from about 203 to 1613 during a particular quarter, university user days ranged from 121 to 213, and local user days ranged from 706 to 2275. Figure 2 shows the number of user days for all industrial and university users in the HTML for each quarter of FY 1991.

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.

To date, 211 research proposals have been submitted to the User Program. A breakdown by user category of these proposals is given in Table 3. Access clearances have been initiated on 291 individual researchers who were listed as principal investigators on these proposals.

Table 3. Status of research proposals submitted July 1987 through September 1991

User type	Research proposals submitted
Industry	77
University	118
Other Government	4
Total Nonproprietary	199
Proprietary	12
Grand Totals	211

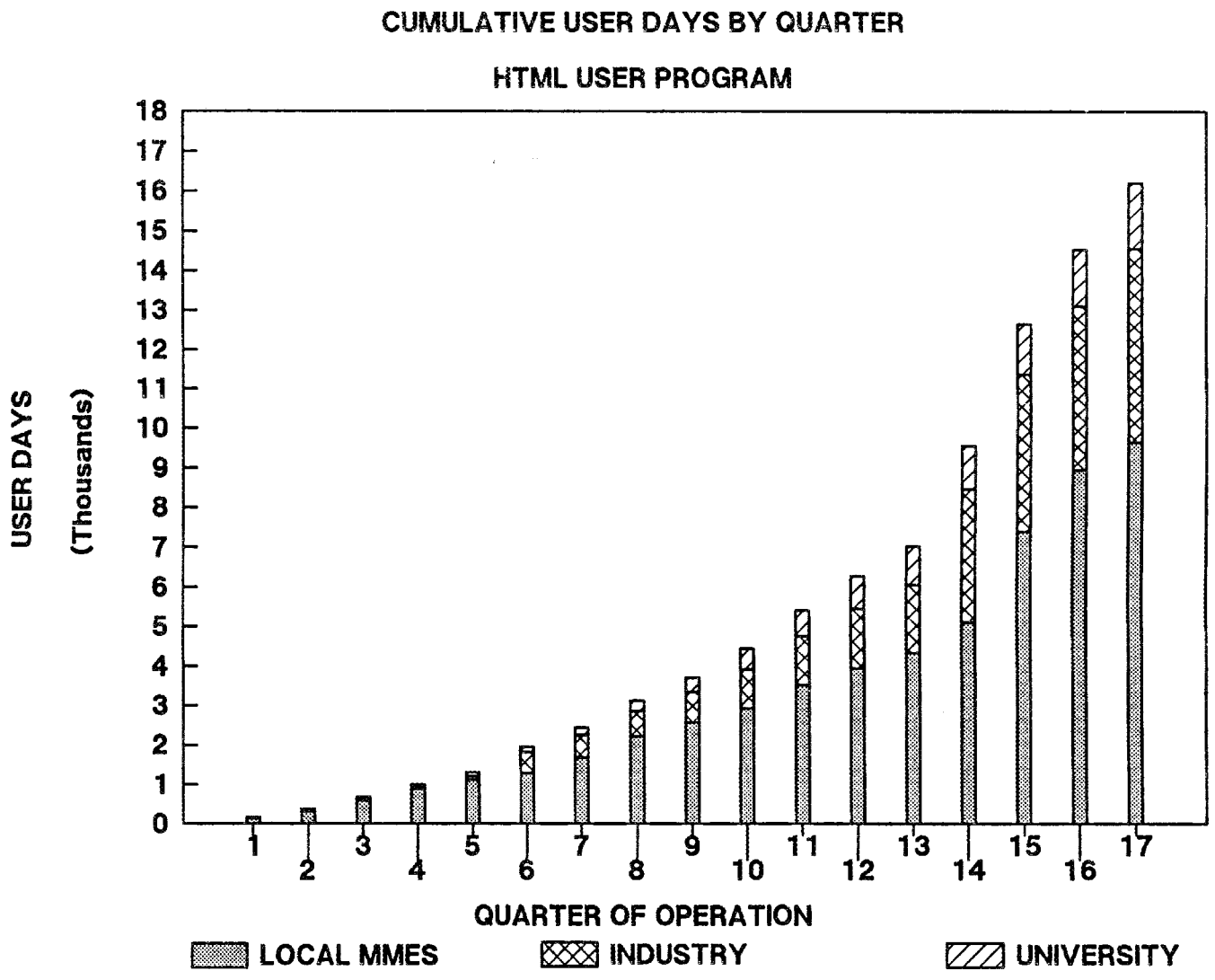

Fig. 1. Total HTML user days in FY 1991.

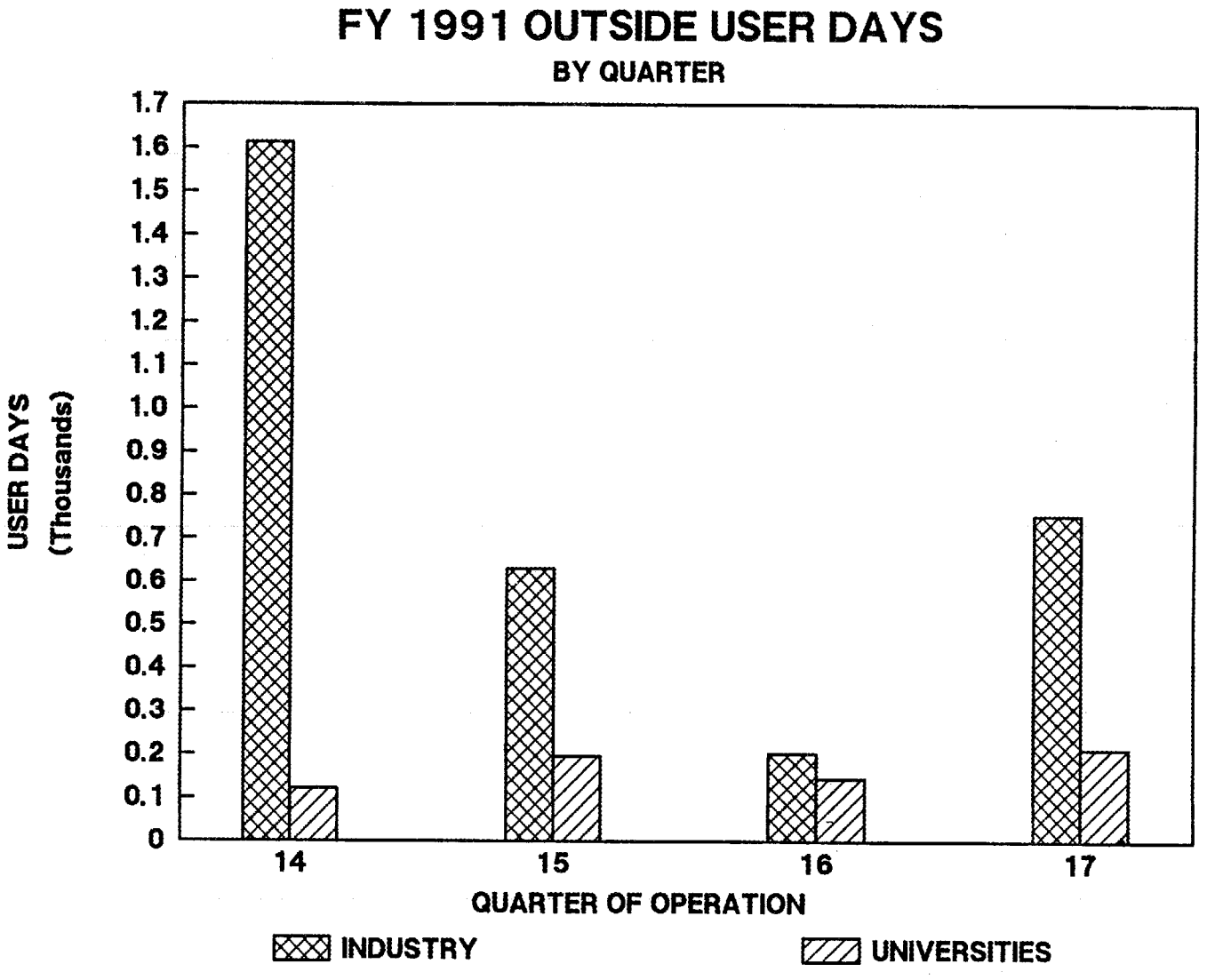

Fig. 2. Total industrial and university user days in the HTML in FY 1991.

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 which had significant activity during this report period. Many projects were active in more than one User Center as indicated in the listing of projects summarized (page v).

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 instrument for which they have primary operating responsibility:

Dr. L. F. (Larry) Allard, JEOL 4000EX (TEM) and JEOL 2000FX (AEM);
Dr. A. (Ashok) Choudhury, VG ESCA/SIMSLAB2 and Auger;
Ms. D. W. (Dorothy) Coffey, Hitachi S-800 (FE-SEM) and sample preparation;
Mr. T. J. (Tommy) Henson, JEOL 733 Electron Microprobe;
Ms. K. L. (Karren) More, JEOL 2000FX (AEM) and JEOL 4000EX (TEM); and
Mr. T. A. (Tad) Dodson, computer services support.

During the past year, user activities with researchers from universities and industrial companies have continued at about the same level as during 1990. Use of the surface chemistry facilities by ORNL researchers has increased significantly.

Several important new instruments are being added to the suite of user center instruments. A scanning probe microscope (SPM) incorporating both scanning tunneling and atomic force microscopy modes is being purchased. This instrument will have interchangeable heads that will allow the surfaces of large specimens to be imaged with near-atomic resolution. Another major new addition will be a Field Emission Gun-Transmission Electron Microscope (FEG-TEM). The contract for a Hitachi HF-2000 200-kV FEG-TEM was placed in September 1991, and delivery is planned for June 1992. 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. 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 techniques). 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. A Director's Fund initiative to develop electron holography and demonstrate it for solving materials characterization problems was approved, and work is under way.

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 U.S. manufacturer's Si_3N_4 ceramic material, which greatly improved the high-temperature creep resistance and other properties. TEM techniques were utilized to identify pure edge vacancy loops and antiphase domain boundaries as the primary crystal defects in α-Si_3N_4 grown by chemical vapor deposition (CVD). Aging secondary ion mass spectrometry was used to unequivocally detect intergranular lithium attack in Nb-Zr alloy (used in the SP-100 Space Power program) where none had been detected by conventional techniques. In association with the University of Tennessee and Pennsylvania State University, electron holography has been used for the first time to characterize details of ferroelectric domain walls in barium titanate. External user research projects with significant activity during this reporting period are summarized.

Coors Electronic **Status: Continuing**

Project Title: **Use of Cobalt and Nickel as a Barrier to Diffusion of Nickel through Gold,** *K. W. Baxter*

Using Auger surface and depth profile analysis, the effectiveness of various plated nickel and gold, nickel/cobalt and gold coatings on tungsten metallized ceramics in resisting thermal diffusion and oxidation of nickel on plated gold surfaces was determined. Accumulation of nickel oxide on the gold surface can seriously degrade the solderability and bondability of ceramic package metallization. This project has demonstrated that, for typical electroplated layers of nickel having 2 μm of electroplated gold, a surface concentration of about 30 at. % of nickel and oxygen is created by a 450°C, 15-min heat treatment in air. The nickel oxide extends 200 to 300 Å into the gold layer. In most cases, this is enough to seriously degrade solderability and bondability. Some improvement was found when an electroplated nickel-cobalt layer was substituted for the nickel. A CVD process for depositing nickel and nickel/cobalt was also explored. Recent utilization of an electroless gold deposit process has resulted in even better heat resistance. Future work will focus on the effectiveness of various post-plating heat treatments.

Detroit Diesel Corporation **Status: Continuing**

Project Title: **Identification of Typical Microstructures and Phases of Sintered Silicon Nitride Cam Roller Followers,** *Karen Weber*

The HTML X-ray diffraction (XRD), TEM, and scanning electron microscopy (SEM) equipment were used to study the intergranular phases and microstructure of various lots of new SN220 silicon nitride cam roller followers. These rollers were dry pressed, sintered, and fully machined with various production-viable techniques. All exterior surfaces were machined with at least one material removal process; some were machined with more than one process. Each of the exterior surfaces, representing different material removal processes as well as core samples, was examined.

The JEOL 2000FX TEM was used to establish the glassy phase constituents of new silicon nitride cam roller followers. Three rollers from different lots were examined to determine lot-to-lot variation. The Hitachi S800 SEM was used to baseline the typical pore size and distribution thereof within the silicon nitride cam roller followers and the variation between different lots. Both outer machined surfaces and core samples were examined. Similarly, rollers which have been subjected to various typical and extreme loading were examined. This research has revealed that the silicon nitride rollers consist of three crystalline phases: beta-silicon nitride, tungsten silicide, and 'X', which is unknown at this time, and a nonrecrystallized amorphous phase, Si-Al-Y-O-Ca. Variation crystalline and amorphous phases within a part, part to part, and lot to lot were determined to be minimal. Also, the total amount of tungsten silicide and 'X' has been determined to be a constant. Minor traces of W-Fe-Si were detected but not 'X'. Since these parts passed all of the functionality tests, the material and processing methods are considered acceptable. It should be noted that minor grain pullout and limited microcracking were seen within the highly overloaded components. Based on these results, these particular ceramic rollers have been approved for production volume procurement. Similar examinations will be performed on SN220 rollers which have experienced various modes of on-highway heavy-duty diesel truck (engine) operation.

Norton/TRW **Status: Continuing**

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

The modified HIP silicon nitride that exhibited improved fatigue life, as compared to material characterized in a previous user project, had no observable amorphous grain-boundary phase at two grain boundaries (determined using the JEOL 4000 EX high-resolution TEM). This is significantly different than the 2-nm amorphous film observed in the earlier NT154. The grain-boundary structure difference begins to explain the improved fatigue behavior of the modified HIP silicon nitride.

Teledyne Allvac **Status: Continuing**

Project Title: **Study of Major Alloying Elements and Morphology of Microstructural Components in Superalloys,** *Shelba R. Taylor*

During FY 1991, three areas of study were performed: (1) Effect of P and Y on the segregation of alloy 718, (2) effect of P and Y on the phase transformation in alloy 718, and (3) grain-boundary segregation and the ductility of Ni-base alloy.

More studies will be done in order to complete this project in FY 1992.

Thermacore, Inc. **Status: Completed**

Project Title: **Evaluation of Refractory Metal Coatings Applied to Carbon-Carbon Composites,** *John Hartenstine*

The purpose of the research was to determine the structural and metallurgical compatibility of carbon-carbon composites with refractory metals to form vacuum-tight enclosures for high-temperature heat pipe applications.

New Mexico Tech **Status: Continuing**

Project Title: **Ceramic Particle/Metal Matrix Composites,** *Krishan Chawla*

Preliminary SEM and analytical electron spectroscopy (AES) experiments were done on an aluminum matrix composite reinforced by SiC particles made by spray casting. The SEM examination of the samples showed a much more uniform distribution of SiC particles than that observed in the composite made by conventional casting. The AES results are being analyzed.

North Carolina State University **Status: Completed**

Project Title: **High-Resolution Electron Microscopy on the Nucleation of Diamond Film,** *Jeffrey Glass*

Interfacial areas between diamond films and Si substrate were examined using the high-resolution electron microscope (JEOL-4000 EX). We concluded that the diamond nucleated from an amorphous layer on top of the Si substrate.

Pennsylvania State University **Status: Continuing**

Project Title: **Microscopy of the Chemical Order Domains in Lead Perovskites,** *Clive Randall*

High-resolution electron microscopy (HREM) studies are in progress on single crystals of $Pb(Mg_{1/3}Nb_{2/3})O_3$:$PbTiO_3$, across the perovskite solid solution system. The role of ordering the B-site cations within these solid solutions is strongly correlated with the nature of the dielectric properties within these lead-based perovskites. The HREM study is beginning to be used to determine the atomic structure and distribution of these ordered regions. Computer simulations are in progress to determine ordered structures, as a function of imaging focus conditions, specimen thickness, and degree of order. The ordering in $Pb(Mg_{1/3}Nb_{2/3})O_3$ is in isolated domains approximately 40 Å in diameter and the volume fraction of ordered regions approximately 35 vol %. The structure is based on a $Fm3m2a_o \times 2a_o \times 2a_o$ unit cell as confirmed from the simulations. The occupancy and degree of order within ordered domains is the final part of this project still to be completed.

Pennsylvania State University **Status: Continuing**

Project Title: **Size Effects of the Paraelectric:Ferroelectric Phase Transition in Ultra-Fine Particle Barium Titanate**, *Clive Randall*

Owing to ferroelectricity being a cooperative phenomenon, there exists a size effect which thermodynamically influences the stability of a given phase. In bulk $BaTiO_3$, the stable ferroelectric phase is tetragonal at room temperature; however, recent work has shown that a particle size approximately 0.1 μm in diameter no longer has a stable room-temperature ferroelectric phase. Based on theoretical calculations we performed, a critical size much smaller (~ 300 Å) than this was predicted. Convergent beam electron diffraction analysis on particles ~ 700 Å appears to imply a ferroelectric non-cubic symmetry exists. However, interpretation of patterns was much more difficult than originally anticipated, owing to diffuse scattering and particle morphology.

Pennsylvania State University **Status: Continuing**

Project Title: **Characterization of the Curie Point Transition in Barium Titanate Using Electron Microscopy**, *Clive Randall*

The nature of the paraelectric-ferroelectric phase transitions in $BaTiO_3$ has long been debated. The phase transition shows evidence of both displacive and order-disorder characteristics. Diffuse scattering and microdomains existing within temperature ranges above the phase transition have previously been associated with an order-disorder behavior. The experiments performed at HTML did not show any evidence of microdomains above the Curie temperature. This was confirmed on crystals processed by different methods to eliminate this variable. In addition, the diffuse scattering presence was confirmed, but its temperature dependence was different than that previously reported by Comes et al. Hence, we believe that this phenomena is also not related to the order-disorder dependence.

Pennsylvania State University **Status: Continuing**

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

A high-resolution SEM study of multi-ion-beam-sputtered $Pb_{1-1.5x}La_xTiO_3$ thin films utilizing the JEOL 890 SEM revealed a direct dependence of the ferroelectric hysteretic properties upon the microstructure of the films. Film microstructures consist of fine grains approximately 20 to 50 nm in diameter. Grain size and pore formation are greatly influenced by the Pb content of the films. Films with high Pb contents exhibit larger grains and high porosity, while films with low Pb content are very dense, exhibit fine grains (20 nm or less), and also display a second large-scale structure consisting of approximately 1 μm groups of grains. This large scale-structure for the low Pb films is believed to be related to a (100) preferred orientation, which is observed by XRD. Detailed TEM analysis of the microstructure and substrate-film interface reactions of these fine-grained films is continuing.

Rice University **Status: Continuing**

Project Title: **Development of an Improved Method for Determining Hardness and Elastic Modulus in Thin Films and Small Volumes,** *George M. Pharr*

Micron and submicron indentations in soda-lime glass, fused silica, tungsten, aluminum, sapphire, quartz, and silicon produced using the nanoindenter were studied in the Hitachi S-800 SEM, taking advantage of its low-voltage operating capability to avoid charging problems in insulating ceramics. Indentations in the first six materials were carefully imaged to document indentation size for comparison with predictions of a new analysis procedure for determining hardness and modulus from indentation load-displacement data. The procedure works well, and the technique development is now complete. Indentations in the brittle materials were also examined to establish the extent of indentation cracking as it relates to our work on the development of a fracture toughness microprobe. SEM studies showed that we have successfully identified an indenter which encourages cracking in submicron indentations, thereby paving the way for further development of the technique. The nature of subsurface cracking was also studied using a special specimen of silicon which was cleaved to reveal the subsurface structure. Work on the development of the fracture-toughness microprobe will continue in the forthcoming year.

University of Delaware **Status: Completed**

Project Title: **The Investigation of Interphase Morphology and of an SiC-Whisker-Reinforced Alumina Composite Using HREM,** *A. Parvizi-Majidi*

HREM was undertaken on a hot-pressed 30 vol % SiC-whisker-reinforced Al_2O_3 composite (Product CC7000, ACMC Corp.) to investigate any effect of fracture temperature on the interface between whiskers and matrix. Samples were prepared from bend bars that were fractured at 20, 1000, 1200, and 1400°C and underwent the same service life (i.e., same test cross-head speed). The morphology and any visual change in the interface (or interphase) thickness were investigated and analyzed as a function of the fracture temperature.

University of Michigan **Status: Continuing**

Project Title: **Characterization of Synthesized SiC Whiskers,** *T. Y. Tien*

The research performed in the HTML involves the characterization of SiC whiskers synthesized at the Department of Materials Science and Engineering, University of Michigan, and the investigation of the interfacial characteristics of the ceramic (Si_3N_4) matrix composites prepared from one tailored SiC whisker synthesized at the University of Michigan and one commercial SiC whisker. Whiskers synthesized under a variety of conditions have been characterized, and a mechanism of vara-solid growth has been proposed.

University of Tennessee/Knoxville **Status: Complete**

Project Title: **Study of the Fracture Surfaces of Ni-20 at. % Mo Alloys Which Have Been Microalloyed With Various Elements,** *Charlie R. Brooks*

An Auger spectroscopy analysis was conducted of the fracture surface facets of an Ni-20 at. % Mo alloy which was doped with Be (approximately 0.1 wt %) in an attempt to make the alloy ductile. This alloy showed some plastic deformation upon bending, and fracture occurred intergranularly as well as transgranularly. In the binary Ni-Mo alloy, fracture is entirely intergranular, and the material is very brittle.

No evidence of the segregation of Be to the high-angle grain boundaries was found, but the data did indicate that there is less Mo and P on these grain boundaries than in the bulk.

University of Tennessee/Knoxville **Status: Continuing**

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

In using secondary ion mass spectroscopy (SIMS) analysis to locate the position of carbon atoms, such as in fine carbides, the problem exists as to which carbon ion (e.g., C24) or complex to use to best represent the sputtered carbon during imaging of the surface to locate carbon-rich areas. SIMS analyses have been carried out on a sample of a 0.5% carbon steel in which the location of the carbon was known, since the microstructure consisted of primary ferrite and pearlite. Thus, the carbon image should reflect the high concentration of carbon in the pearlite, or at high magnification in the carbide itself. Also, this sample has allowed us to evaluate the effect of cratering, which occurs due to the sputtering on the carbon image.

University of Tennessee Space Institute **Status: Continuing**

Project Title: **Study of Interfacial Reaction Phases in Laser-Welded Ceramic Particulate/Al-alloy Metal Matrix Composite,** *N. Dahotre*

Composites of 20 and 10 vol % SiC/Al-alloy were pulse laser welded at selected pulse-duty cycles (50 to 91%). The extent of microstructural change increased with duty cycle. The microstructure of the laser-melted region was investigated using analytical electron microscopy (AEM), SEM, and electron microprobe analysis (EMA). The analysis revealed that the process produced complex precipitates such as Al_4C_3 and $Al_4Si_2C_5$ due to the rapid cooling rate. The associated rapid cooling also produced a fine distribution of nonequilibrium complex precipitates which remained unidentified as they are not referenced in the available literature. Further, attempts were made to characterize these unidentified phases using high-resolution transmission electron microscopy (HRTEM). Also, the HRTEM study of subnanometer grain-boundary films and particle-matrix interface layer in the weld region is presently under way.

University of Tennessee/Knoxville **Status: Continuing**

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 are characterized using TEM and X-ray diffractometry. The deposits are found to be fine grained, and the phases present in the films depend on the nature of the substrate. Films of various thicknesses deposited on microscope slides or oxidized stainless steel substrates contain a mixture of two phases: a body centered cubic (bcc) and a modified hexagonal ϵ-phase. The hexagonal phase appears 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. These films are hard and brittle, as indicated by microhardness measurements.

Films deposited on oxide-free austenitic stainless steel substrates, on the other hand, are mostly bcc and exhibit a dominant <200> texture. These films are softer and less brittle than those deposited on oxidized substrates. In-situ high-temperature X-ray diffractometry revealed that the ϵ-phase transforms to bcc when the films are annealed at 773 K. On annealing at 873 K, the bcc phase transforms to face center cubic, which remains stable on cooling to room temperature. These results agree with published data that suggest stability of the bcc phase up to 840 K. Some discrepancies from earlier published reports have been identified in the light of the present results.

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. 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 servo-hydraulic 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 (nanoindenter). The major research activities initiated in the areas of tension, flexure, and indentation testing during FY 1991 are included. In the paragraphs that follow, detailed descriptions of the tension, flexure, and indentation research facilities are provided, along with appropriate examples of data generated by these facilities.

The Group Leader of the MPUC is Dr. M. K. Ferber. Other staff and their equipment expertise are:

Dr. M. G. (Michael) Jenkins, tensile and flexure test facilities;
Mr. V. T. (Tyler) Jenkins, ceramic specimen preparation; and
Ms. L. (Laura) Reister, flexure and nanoindenter.

5.2.1 Flexure Strength Measurements

During FY 1991, extensive flexure strength research was conducted using both the FTF and a universal test machine (UTM) equipped with a high-temperature furnace and ceramic retort. 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 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-analogue converter. Data generated during an experiment are transferred directly to the computer using an external data acquisition system. 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 up to 1500° C.

Studies involving flexure testing have focused upon (1) the correlation of high-temperature strength of sialon ceramics with variations in the processing parameters, (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 stress intensity factor threshold for high-temperature crack growth in silicon nitride, (5) the effect of environment upon the fatigue resistance 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 — **Status: Completed**

Project Title: **Evaluation of Toughness of SiC-Whisker-Reinforced Alumina Composites Fractured as a Function of Temperatures and Loading Rate,** *A. Parvizi-Majidi*

A hot-pressed 30 vol % SiC-whisker-reinforced Al_2O_3 composite (Product CC7000, ACMC Corp.) was chosen to investigate effects of fracture temperature (20, 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•√m) remained relatively constant through 1200° C and decreased to about 4 MPa•√m at 1400° C. For all test temperatures, it appeared that as long as stable crack propagation was induced, the toughness appeared to be independent of the test cross-head speed (2, 5, 13, 32, and 79 μ/m). Fractography was used to understand the fracture mechanisms involved.

Allied-Signal Aerospace Co. **Status: Continuing**

Project Title: Determination of the High-Temperature Static Fatigue Limit in High-Strength and High-Toughness Silicon Nitride, *Philip J. Whalen*

The high-temperature static fatigue limit of GN-10 silicon nitride (fabricated in 1990 using an improved processing flowsheet) was determined at two temperatures, 1204 and 1371°C. The data obtained during this period will be compared to that obtained during 1990 on standard GN-10 to determine if the improved processing condition improved the high-temperature properties. The multi-stage, four-point flexure, high-temperature test stations were used for this research. For each temperature and load condition (five load levels), three flexure bars were statically loaded for 4 h and then fast fractured at high temperature. A drop in fast-fracture strength after the static exposure load indicated that slow crack growth (SCG) had occurred during the static load. The load-point displacement during the static load was also measured and will be used to determine if primary creep was effected by the improved processing of this silicon nitride.

Ceramics Process Systems **Status: Continuing**

Project Title: Research on Duophase Sialons with a Controlled Grain-Boundary Phase, *Ran-Rong Lee*

Duophase (a'/B') sialon is being developed for advanced engine applications by using the Quickset (TM) 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 flexural stress-rupture exposure at 1300°C and 340 MPa for 190 h. 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 a yttrium aluminum garnet (YAG) phase and elongated grains yielded better creep resistance. The correlation between mechanical properties and microstructure was analyzed.

Tuskegee University **Status: Continuing**

Project Title: Characterization of Various Al_2O_3/SiC Composites at Room and Elevated Temperatures, *S. Jeelani*

This project was to study the microstructure, defects, and fracture mechanisms of monolithic Al_2O_3, SiC whisker/Al_2O_3, and SiC platelet/Al_2O_3 composites. The flexural properties of both whisker- and platelet-reinforced SiC/Al_2O_3 composites were measured using four-point flexure strength measurements. Fracture toughness was determined for both straight and chevron-notched specimens. SEM examination reveals that there is lack of parallelism in platelet orientation, which necessitates the development of a technique to control the platelet orientation in processing. TEM examination of the specimens is under way to reveal the defects and interface characteristics of these alumina matrix composites.

Sundstrand Power Systems **Status: Completed**

Project Title: Study of Fast-Fracture, Dynamic, and Static Fatigue Behavior of Kyocera SN235 Silicon Nitride, *Timothy M. Pattison*

The fast-fracture and SCG behavior of Kyocera SN235 silicon nitride were studied at room temperature, 800, and 1000°C using four-point flexure and a quarter-span geometry. Average strengths were found to be 966, 984, and 878 MPa with Weibull moduli of 9.7, 17.6, and 13.1 at room temperature, 800, and 1000°C, respectively. A load rate of 40 N/s was used. Fractographic analysis of low-strength specimens revealed large grains to be the cause of failure. Thirty specimens were tested at each temperature.

Dynamic fatigue measurements were performed at 800 and 1000°C using a 0.4 N/s loading rate. A least-squares regression performed on the test data yielded values of 125.0 and 13.5 for the fatigue exponent at 800 and 1000°C, respectively. These values indicate that SCG is not a concern at 800°C; however, at 1000°C it is a serious problem. Again, 30 specimens were tested at each temperature.

Static fatigue strength measurements at 1000°C yielded a fatigue exponent of 15.9 when a least-squares regression was applied. Applied stresses of 345, 482, and 600 MPa were used. Six specimens were tested at each stress level.

The excellent agreement between dynamic and static fatigue test results clearly indicates that SCG is a serious problem at 1000°C and that SN235 is unsuitable for such applications as gas turbine rotors and nozzles.

Norton **Status: Continuing**

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

In this project, the fatigue and creep properties of a 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. Subsequent creep studies showed that creep cavitation and microcracking were the predominant damage mechanisms in the NT 154 when measured at 1370°C in air. A comparison of the flexural creep data with the tensile data revealed a significant discrepancy between the measured stress sensitivities of the creep strain rates. This discrepancy was attributed to stress redistribution in the flexure specimens, which invalidates the equations normally used to calculate the creep strain for this specimen geometry.

5.2.2 Tensile Testing

Proper tensile strength measurements for brittle materials require 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 approximately 0.5 μm at 1500°C. A Keithley 500 Data Acquisition System (DAS) and IBM-compatible microcomputer are used to monitor or control up to four test stations simultaneously.

A servohydraulic test machine (Instron 1332 with 8500 series electronics) is equipped with a 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 measurements. Gage lengths of 155 to 200 mm are possible for high-temperature measurements 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.

During FY 1991, extensive studies of the strength, creep, and fatigue behavior of silicon nitride button-head 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 research 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., Montman-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 **Status: Continuing**

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

These 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 the 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 the steady-state creep rate. 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 silicon nitride, while the fatigue life was a factor of 2 to 5 longer. These results have been instrumental to the introduction of the NTX 164 silicon nitride as a commercial material.

Allied-Signal Aerospace Co. **Status: Continuing**

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

Extensive tensile creep and fatigue measurements were conducted on a commercially available HIPed silicon nitride (Norton NT 154). Tests were conducted at temperatures of 980 to 1400° C. Results to date clearly show a transition in failure behavior from SCG to creep damage as the temperature is increased.

5.2.3 Mechanical Properties Microprobe (Nanoindenter)

The mechanical properties microprobe (nanoindenter) 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 sensitive 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 1991, 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.

Michigan State University **Status: Continuing**

Project Title: **Study of Wear Characteristics of Silicon Nitride,** *Harold Schock*

Hardness and elasticity measurements were made using the HTML nanoindenter. Two types of specimens were examined to determine if radiation damage could be found (the samples were irradiated by a 2.5-MeV 20-Ne beam). The samples were silicon nitride and a martensitic stainless steel. The stainless steel was chosen because its martensite phase is metastable reverting to the austenite phase under kinetic energy transformation. Results of the studies showed a damage threshold of 10^{14} ions/cm^2 for the stainless steel and no discernable damage for the same dose implanted in Si_3N_4.

Stanford University **Status: Completed**

Project Title: **Hardness and Elastic Modulus Measurements of InGaAs/InP and GexSi1-x/Si Superlattices,** *D. A. Stevenson*

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 by the development of models and using TEM to study the dislocation structure of the superlattices.

Clemson University **Status: Continuing**

Project Title: **Load Deflection Measurements of Silicon Microbeams,** *J. Almonte*

The load-deflection characteristics of micro-cantilever beams made of single-crystal silicon were measured. The beams were approximately 60 μm in width, 40 μm in thickness, and 1000 μm in length. The resulting data were compared with numerical calculations made at Clemson University.

New Mexico Tech **Status: Continuing**

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

Fiber/matrix interface modification with a view to obtain an enhanced fracture toughness in oxide/oxide composites is being done via application of coatings on fibers, tin dioxide on alumina + zirconia (PRD-166), and boron nitride on mullite (Nextel 480) fiber. SIMS was used to characterize these composites with reasonable success. Flexure strength measurements and nanoindentation experiments were performed on these composites to evaluate the effects of interface modification.

Thermal stress distribution and the effect of interface roughness and their effects on crack propagation in these composites were evaluated. It appears that in the case of the PRD-166 fiber, the interface roughness effect predominates vis a vis thermal stress effect. Consequently, we were unable to slide the fibers in nanoindentation experiments. In the case of BN-coated Nextel 480, there was evidence of sliding. Fiber pullout was observed during the flexure tests.

Aluminum Company of America **Status: Completed**

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

Several significant findings were obtained from this study:

1. For as-produced samples, carbon-coated Nicalon/AlBo has a lower interfacial shear strength (ISS) than duplex-coated or uncoated Nicalon/AlBo composites. However, the ISS of uncoated Nicalon/AlBo composites is stronger than the other systems. The results imply that the toughness of the interface may be controlled by applying fiber-coating materials in CMC systems.

2. Heat-treated samples of uncoated Nicalon/AlBo composites using the polymeric CPS binders system and heat-treated samples of deep/ex-coated Nicalon/AlBo composites using the aqueous binder system show lower ISS than as-produced samples. It also shows that the toughness of the interface for these two systems is not shown significantly by trade-off by temperature.

From this research, we conclude that nanoindentation can be used for the determination of interfacial shear strength and the coefficient of friction in the fiber-matrix interface of ceramic matrix composites.

5.3 PHYSICAL PROPERTIES AND X-RAY DIFFRACTION USER CENTERS

The facilities of these two User Centers provide both XRD and thermophysical property measurement techniques to characterize structure, phase content, stability, reactions, and thermal properties of advanced structural materials. The temperature range of the facilities is from room temperature to 1500°C and above. The knowledge obtained is used to improve synthesis, processing, and utilization of advanced materials and to develop models relating microstructure, phase content, and defect concentration to properties and performance. During the past year, overall user activity, both external and ORNL, increased steadily. A couple of examples are given below, along with a brief description of facility enhancements. Following these examples, a brief summary of research projects conducted within these centers is given for those projects with significant activity during this reporting period.

The capabilities of the high-temperature X-ray diffractometer were extended with the addition of computer control of the temperature controller, the installation of capability to use hydrogen gas for reduction and hydrogen embrittlement studies, and the modification of the furnace system to permit studies well above 1500°C in 1-atm nitrogen. The latter capability will be important in pressureless sintering studies of nitrogen ceramics and was first used by Dow Chemical Company in a study of the temperature and compositional dependence of Al_2O_3-Y_2O_3 intergranular phases in AlN. The completion of the automated xenon flash instrument for room-temperature thermal diffusivity measurement complements the high-temperature laser flash system, enabling rapid and more accurate measurements at room temperature. Work has commenced to develop a longitudinal bar thermal conductivity cryostat to provide thermal transport measurements from about 90 to 500 K, needed for modeling the contribution of microstructure and defects on thermal conductivity.

The Group Leader of both the PPUC and the XRDUC is Dr. C. R. Hubbard. Other staff and their equipment expertise are:

Mr. O. B. (Burl) Cavin, all X-ray equipment;
Dr. R. B. (Ralph) Dinwiddie, laser flash diffusivity; and
Mr. W. D. (Wally) Porter, STA, DSC, and dilatometer.

With United Technologies Research Center (UTRC), the thermal aging of plasma spray thermal barrier coatings was studied by laser flash thermal diffusivity measurements in combination with SEM and density determinations. Together, models to predict the thermal conductivity and density as a function of aging time and temperature were developed. Simultaneous thermal analysis (STA) and high-temperature X-ray diffraction (HTXRD) of polymer precursors for oxide ceramic synthesis were studied with a user from Cornell University. This study has led to a detailed understanding of the chemical processes and crystallization of ceramics involved in burnout and consolidation of the ceramic. The method holds promise for development of high-purity ceramics with unusually well-controlled chemical composition. STA

and HTXRD were also used to characterize the conversion of ZrO_2 and $(Zr,Hf)O_2$ gels prepared at the University of Kentucky Center for Applied Energy Research. The observation that either tetragonal or monoclinic zirconia could be formed by simply changing the rate of gel formation was clarified by determination that, in all cases, tetragonal zirconia forms upon heating above 400° C and only on cooling to below 200° C does the monoclinic form of zirconia evolve in the rapidly precipitated gels. The differences were determined not to be due to differences in crystallite or agglomerate size. Utilizing studies with sulfate anions, we determined that the transformation to monoclinic is likely due to differences in anionic defects on the surfaces of the particles.

The HTXRD study with Dow Chemical demonstrated furnace operation to above 2000° C and the ability, when combined with a position-sensitive detector (PSD), to study fast intergranular phase reactions and transformations in aluminum nitride ceramics at these extremely high temperatures.

5.3.1 Research Projects Performed in the Physical Properties User Center

McDonnell Douglas **Status: Completed**

Project Title: High Thermally Conductive Carbon-Carbon Composites, *James Sapp*

The thermal diffusivity was measured on different carbon-carbon composites, and the thermal conductivity was then calculated using literature values of the specific heat of graphite. The effects of composite density, fiber volume fraction, and fiber thermal conductivity were studied.

Norton/TRW **Status: Completed**

Project Title: Thermal Diffusivity Dependence on Silicon Nitride Grain-Boundary Composition and Volume, *R. Yeckley*

The thermal conductivity of four HIP silicon nitrides was characterized. The silicon nitrides were NT 154 and silicon nitride with three different ytterbia contents. Thermal conductivity dependence on rare earth type, amount, and silicon nitride grain size was determined.

The research conclusions included: (1) silicon nitride with yttria had higher conductivity than ytterbia, (2) thermal conductivity increased with volume fraction ytterbia, and (3) thermal conductivity exhibited a stronger dependence on volume fraction additive than silicon nitride grain size.

5.3.2 Research Performed in the X-Ray Diffraction User Center

American Superconductor **Status: Completed**

Project Title: Phase Stability of $Bi_2Sr_2Ca_1Cu_2O_x$ Superconductor at High Temperatures, *H. S. Hsu*

1. *In situ* phase formation from the melt was determined in the $Bi_2Sr_2Ca_1Cu_2O_x$ system for atmospheres of 0.01 and 1.0% O_2/He using the HTML HTXRD equipment.

2. STA was used to perform studies in 0.001, 0.10, 1.0, 7.6, 21, and 100% O_2/Ar atmospheres for the $Bi_2Sr_2Ca_1Cu_2O_x$ system to complement and extend the results from HTXRD.

These combined results have 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. Such understanding is essential to effectively develop methods for processing of Bi-based high-temperature superconductors.

DOW Chemical **Status: Continuing**

Project Title: Crystallization of Intergranular Phases in AlN with Y_2O_3 Sintering Aids, *A. K. Knudsen*

The development of crystalline yttrium aluminate phases during the sintering of aluminum nitride was studied with HTXRD. Using a PSD, dynamic diffraction data were obtained at temperatures as high as 2200° C in a nitrogen atmosphere. In yttria-doped AlN, $Y_4Al_2O_9$ forms at temperatures well below the liquidus. Recrystallization and melting occur at roughly 1800° C. In addition, crystallization of YN, $YAlO_3$, and $Y_4Al_2O_9$ from the melt was observed. At present, correlation of diffraction data with actual sample temperatures (via thermal expansion) is continuing.

Clemson University **Status: Completed**

Project Title: Study of the Character of Stresses Developed in an Iron-Based Alloy Subjected to Sulfidizing Mixed Gases at Elevated Temperatures, I, *James 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 crystallite 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 Status: Continuing

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

This research has been directed toward the characterization of ceramic scales naturally formed on 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 HTXRD Facility of the HTML at ORNL.

The experimental program is near completion with efforts currently being focused upon data analyses and interpretation.

University of Kentucky Status: Continuing

Project Title: **Analytical Characterization of Ultrafine Particles,** *John Stencel*

Data acquisition under He and hydrogen environments in the high-temperature X-ray diffractometer was performed. Analyses of these data have not been as timely because of the tasks of data transfer to our laboratory and the specialized manipulation that is needed. Additional data will be acquired on the HTXRD equipment, which should confirm our initial interpretations. We are currently obtaining X-ray photoelectron spectroscopy (XPS) and thermogravimetric analysis/mass spectrometer (TGA/MS) data at our laboratory on the same samples that were examined by XRD and SEM at the HTML to enable a more detailed explanation of structural genesis as a function of high-temperature exposure.

University of Kentucky Status: Completed

Project Title: **Crystal Phase Formation in Hf-Zr Mixed Oxides,** *Burtron H. Davis*

HTXRD studies indicated that the crystallization temperatures varied with the Hf content. The results were essential to a technical poster presented at the Pacific International Conference on X-ray Analytical Methods in August 1991.

University of Tennessee Status: Completed

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

The heating and cooling transformation temperatures for liquid and solid phases of Inconel 718 are required to model the effects of laser-pulse welding on crack sensitivity of the material. A differential scanning calorimeter (DSC) technique was used to determine temperature zones for different transformations in cast, wrought, and wrought grain-grown Inconel 718. The samples

were subjected to heating and cooling cycles with the rate of 40°C/min. Solid-to-solid-state transformation was studied by cycling the sample in the temperature range of 40 to 1200 to 400°C while solid-to-liquid and liquid-to-solid-state transformations were noted by cycling the sample in the temperature range of 40 to 1420 to 400°C. The solidus temperature is 1200°C, and the alloy melts at 1275°C. At 1170°C a high-temperature phase formation occurs by solid-state transformation in cast samples, which is absent in wrought samples. The two-time deviation in the slope of the baseline in the temperature range of 570 to 870°C was present in all types of samples, which indicated the occurrence of some type of low-temperature event. Just before melting at about 1250°C, the presence of a shoulder peak shows the formation of low-melting eutectic phase.

University of Wisconsin **Status: Completed**

Project Title: **Supersolidus Investigation of the Bi-Sr-Ca-Cu Oxide System,**
Richard Schartman

HTXRD was used to characterize the solid phases present above the incongruent melting point of several oxides whose compositions were close to that of the $Bi_2Sr_2CaCu_2O_x$ superconductor. The results did not agree with quench studies performed on the same oxides, leading us to question the applicability of the X-ray technique to this study. Two possible sources of error have been proposed. First, Pt was transferred from the heater to the sample during the experiment, and this altered the phase equilibria (suggested by Cam Hubbard and Brian Reardon of the HTML). Second, steep temperature gradients were present over the sample, which produced a nonequilibrium distribution of phases.

5.4 CERAMIC SPECIMEN PREPARATION USER CENTER (CSPUC) - M. K. FERBER

The CSPUC 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 (4) application of strain gages. Specific equipment includes a slicer/grinder with open-loop control, a computer numerically controlled (CNC) slicer/grinder, a CNC four-axis grinder, an optical comparator, and a computer-controlled profilometer. Facilities for the application of strain gages are also available. Gage outputs are monitored during testing using a dedicated computer and data acquisition system.

During FY 1991, CSPUC played a major role in several in-house programs. For example, research activities in the International Energy Agency (IEA) Annex II Subtask 5 agreement required extensive utilization of both the optical comparator and strain-gage facilities. The IEA Subtask 5 effort is aimed at comparing flexure and tensile strengths of a silicon nitride ceramic. These strength measurements were conducted at 10 U.S. laboratories. A total of 450 flexure and 150 button-head tensile specimens were fractured as part of the program. Because dimensional consistency is critical to the successful measurement of button-head tensile specimens, each specimen was inspected with the optical comparator and extensive data acquired. This inspection process resulted in identification of machining problems in the shank-to-button transitions of

several specimens by an industrial firm. By utilizing the inspection data, the machining source was able to correct the problem. Properly machined specimens were critical to the success of the IEA effort.

For the IEA flexure strength measurements, a gaged flexure specimen was used to evaluate each participant's room- and high-temperature flexure fixtures. The strain measurements were made with the strain-gage data acquisition system. The resulting data revealed some serious design problems with many high-temperature fixtures. The strain-gage data acquisition system was also used to measure the strain at various locations in the gage section of each button-head tensile specimen during actual testing. These data were then used to calculate the percent bending as a function of applied load.

Several special machining procedures were developed by CSPUC personnel. One procedure involved the fabrication of C- and O-ring specimens from both 2.5- and 5-in.-diam ceramic composite tubes. A minimum of 8 h has been required to machine one specimen from the 2.5-in.-diam tube while the preparation of specimens from the 5-in. tube was unsuccessful. Effective utilization of the CNC 4-axis grinder reduced the grinding time to a maximum of 15 min/specimen (after setup) for both tube sizes. This work was instrumental for the timely completion of the mechanical testing tasks in the ORNL Advanced Industrial Heat Exchangers Program.

Another example involving the CNC 4-axis grinder concerned the development of a procedure for machining 0.157- and 0.490-in.-diam disks of alumina from square blocks (0.20 x 0.20 x 0.062 in. for the 0.157-in.-diam disks and 0.55 x 0.55 x 0.062 in. for the 0.490-in.-diam disks). This procedure necessitated fabrication of a special ceramic arbor for holding the square stock in the grinder. The circular disks were required for physical properties measurements, which were part of a DOE-ORO milestone for the Modular High-Temperature Gas-Cooled Reactor-New Production Reactor (MHTGR-NPR) Program.

5.5 Residual Stress User Center (RSUC) - C. R. HUBBARD

The RSUC will initially consist of an XRD system for mapping macro residual stresses on the surface of ceramic and alloy materials. Delivery of the instrument is expected in early FY 1992. The system consists of a state-of-the-art stress and texture goniometer equipped with a Peltier-cooled solid-state detector and an 18-kW rotating anode generator. The system provides for complete flexibility in sample tilt and provides either divergent or parallel beam operation. Recruitment for a professional staff member is currently under way. User research is planned to begin in early 1992.

To develop competence in measurement of residual stresses within solid objects, a Director's Fund R&D project, "Development and Demonstration of Neutron and X-ray Residual Stress Mapping," was proposed and funded. This project is being conducted in collaboration with the Neutron Scattering Group of the Solid State Division and utilizes an existing triple axis spectrometer. Attachments for sample positioning, data collection, and automation have been developed and successfully tested on the spectrometer. The first demonstration study was a comprehensive mapping of the residual stresses within a 12- × 2- × 1/2-in. ferritic plate containing

a multipass weld. The results will be used to develop and verify models for stresses in multipass weldments. Studies of macro stresses in ceramic and micro stresses in composites are also to be conducted.

6. HONORS, AWARDS, AND PRESENTATIONS

Members of the HTML prepared and presented the three posters given First, Second, and Third place in the national competition in the non-student division at the 15th Annual Conference on Composites and Advanced Ceramics held at Cocoa Beach, Florida, January 13-16, 1991. The first-place poster, entitled "***Microstructural Characterization of Tensile and Flexural Creep Deformation and Fatigue in a Si_3N_4 Ceramic***," was prepared by Ted Nolan, Larry Allard, Matt Ferber, and Dorothy Coffey, all members of the HTML staff. The poster described the phenomenon of long-term damage accumulation that can result in mechanical failure at high temperatures in Norton NTX-154 high-performance Si_3N_4 ceramic. The second-place poster, entitled "**Creep of a SiC Whisker-Reinforced Si_3N_4 Composite in Compression and Bending**," was prepared by HTML staff member Karren More, along with Dave Koester of the Microelectronics Center of North Carolina and Robert Davis of North Carolina State University. The third-place poster, entitled "***Residual Strains in Al_2O_3/SiC_w Composite from 25-1000°C***," was prepared and presented by Cam Hubbard of the HTML. Ben Ballard and Paul Predecki, HTML Users from the University of Denver, were also authors on this poster.

APPENDIX A

LISTING OF THE ADVISORY COMMITTEES

HTML Advisory Committee Members

Term Expires	
Dr. Maxine L. Savitz, Director Ceramic Components Division Garrett Processing Company 19800 South Van Ness Ave. Torrance, California 90509	1993
Mr. Woodie Howe Coors Technical Ceramic 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. Joseph N. Panzarino Norton Company Advanced Ceramics Division Goddard Road Northboro, Massachusetts 01532	1991
Dr. James W. Patten Director, Materials Engineering Cummins Engine Company 500 Jackson Street Columbus, Indiana 47201	1991

HTML User Advisory Committee Members

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

	Term Expires
Dr. J. C. Danko, Director Center for Materials Processing The University of Tennessee 101 Perkins Hall Knoxville, Tennessee 37996-2000	1991
Mr. Lance Groseclose Allison Gas Turbine Operations General Motors Corporation Post Office Box 420 Indianapolis, Indiana 46206-0420	1992
Dr. Arvid Pasto GTE Laboratories, Inc. 40 Sylvan Road Waltham, Massachusetts 02254	1991
Ms. M. J. Rohr DOE Field Office, Oak Ridge P.O. Box 2008 Oak Ridge, Tennessee 37831	1991
Dr. Linda Horton Metals and Ceramics Division Oak Ridge National Laboratory P. O. Box 2008 Oak Ridge, Tennessee 37831	1992

APPENDIX B

HIGH TEMPERATURE MATERIALS LABORATORY USER PROGRAM

SECOND USERS GROUP MEETING

THURSDAY, MARCH 21, 1991

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 second HTML Users Group meeting, which was held in the HTML on Thursday, March 21, 1991, along with Mr. T. Vojnovich, Manager for the HTML User Program at DOE Headquarters.

Attendees at this meeting included past and present users representing 20 outside institutions [10 universities (19 individuals) and 10 companies (14 individuals)] as well as Energy Systems users (24 individuals) and special guests.

The agenda included the following presentations based on research performed in the HTML:

High-Resolution Transmission Electron Microscopy of Dispersion-Toughened Silicon Nitride
Jesse Hefter, GTE Laboratories Incorporated, 40 Sylvan Road, Waltham, MA 02254

Fatigue and Creep Behavior of Hot Isostatically Pressed Silicon Nitride at High Temperature
R. L. Yeckley, Norton Company, Goddard Road, Northboro, MA 01532-1545

Thermal Conductivity of Plasma-Sprayed Zirconia as a Function of Thermal Aging H. Eaton, United Technologies Research Center, MS 129-26, Silver Lane, East Hartford, CT 06108

Residual Stresses in Al_2O_3/SiC Whisker-Reinforced Composites as a Function of Temperature
P. K. Predecki, University of Denver, Engineering Department, Denver, CO 80208

The announcement was made at this meeting that the two new User Centers for the HTML User Program, Residual Stress and Ceramic Specimen Preparation, are expected to be fully operational early in calendar year 1992.

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 17 quarters of operation to date. Approximately 59% of the user days have been from local researchers, while about 30% have been from industry and 11% from universities.

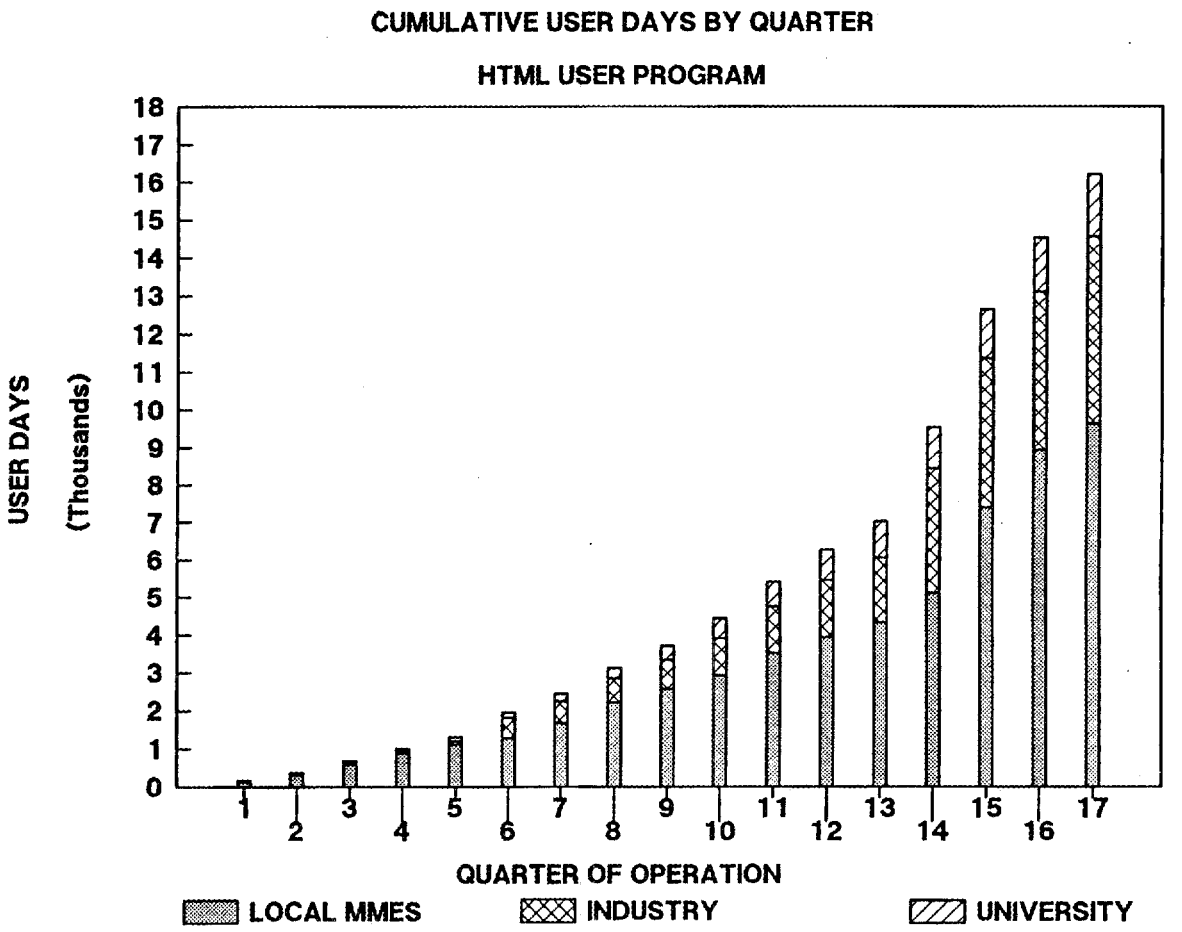

Fig. C.1. HTML cumulative user days from start of User Program.

APPENDIX D

PUBLICATIONS AND PRESENTATIONS

User Center Staff are indicated by an underline and the speaker by an asterisk. 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

H. Hsu, F. A. List, O. B. Cavin, and C. R. Hubbard, "High-Temperature X-ray Diffraction Methods for the Study of the Y-Ba-Cu-O and Bi-Sr-Ca-Cu-O Superconductor Systems," presented at the Pacific-International Congress on X-ray Analytical Methods, Honolulu, Hawaii, August 1991. AMERICAN SUPERCONDUCTOR

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, Goteborg, Sweden, June 1991; to be published in the Proceedings. CERAMIC PROCESS SYSTEMS CORPORATION

M. V. Parish and J. D. Hodge, "Simultaneous Thermal/Mass Spectrometric Analysis of $YBa_2Cu_3O_{7-x}$ Powder Compacts," presented at the Third International Conference on Ceramic Powder Processing Science, San Diego, Calif., February 1990. CERAMIC PROCESS SYSTEMS CORPORATION

R. R. Lee, B. E. Novich, G. Franks, D. Ouellette, M. K. Ferber, C. R. Hubbard, and K. L. More,* "Duophase Sialon For Ceramic Engine Component Applications," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. CERAMIC 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," pp. 111-23 in *Proceedings of the Annual Automotive Technology Development Contractors' Meeting*, Society of Automotive Engineers, Inc., Dearborn, Mich., October 1990. CERAMICS PROCESS SYSTEMS CORPORATION

J. C. Birkbeck, L. S. Kasten, D. S. Foose, W. E. Moddeman, and L. F. Allard,* "Auger, XPS, EDS, and TEM Examinations of Chromate Conversion Coated Cadmium Metal," presented at the 13th Symposium on Applied Surface Analysis, Minneapolis, Minn., June 1991; to be published in *Applications in Surface Science*, North Holland. EG&G MOUND APPLIED TECHNOLOGIES

W. S. Hong, J. M. Sater, and M. A. Rigdon, and M. G. Jenkins, "The Effect of Varying Test Parameters on the Measured High-Temperature Strength of SCS-6 Fiber," presented at the 15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1991. INSTITUTE FOR DEFENSE ANALYSES

M. G. Jenkins,* M. K. Ferber, W. S. Hong, J. M. Sater, and Michael A. Rigdon, "Effect of Exposure Time and Strain Rate on the Elevated-Temperature Tensile Strength of SiC Monofilaments," presented at the International Conference on Fracture Mechanics of Ceramics, Nagoya, Japan, July 1991; to be published by Plenum Press, N.Y. INSTITUTE FOR DEFENSE ANALYSES

R. B. Dinwiddie, J. W. Sapp,* and D. A Bowers, "Temperature Dependence of the Thermal Conductivity of Commercial Carbon/Carbon Composites," presented by James W. Sapp at the Symposium on Composites, Processing, Microstructure, and Properties, Orlando, Fla., November 1990, and presented by R. B. Dinwiddie at the 20th Biannual Carbon Conference, Santa Barbara, Calif., June 1991. MCDONNELL DOUGLAS MISSILE SYSTEMS.

J. W. Sapp, Jr., D. A. Bowers, and R. B. Dinwiddie, "Development of High Thermal Conductivity Carbon-Carbon Composites," presented at the 20th Biannual Carbon Conference, Santa Barbara, Calf., June 1991. MCDONNELL DOUGLAS MISSILE SYSTEMS COMPANY

J. W. Sapp, J. W. Davis, and J. R. Haines, "Carbon-Carbon Composites for CIT," Carbon 90, International Carbon Conference, Paris, France, June 1990. MCDONNELL DOUGLAS MISSILE SYSTEMS COMPANY

H. C. Mantz, D. A. Bowers, F. R. Williams, J. W. Sapp, and M. A. Witten, "First Wall Inboard Limiter Design Development for the Compact Ignition Tokamak," Ninth Topical Meeting on the Technology of Fusion Energy, Oak Brook, Ill., October 1990; published in *Fusion Technol.* **19**, (May 1991). MCDONNELL DOUGLAS MISSILE SYSTEMS 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 37th Sagamore Conference on Structural Ceramics, October 1990; to be published in the Sagamore Conference Proceedings. NORTON COMPANY

T. A. Nolan, L. F. Allard, M. K. Ferber, D. W. Coffey, and R. L. Yeckley, "Microstructural Characterization of Creep Deformation and Fatigue in a Silicon Nitride Ceramic," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. NORTON/TRW

C. R. Hubbard,* R. B. Dinwiddie, T. N. Tiegs, M. K. Ferber, and R. L. Yeckley, "Si_3N_4 Intergranular Phases and Thermal Diffusivity," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. NORTON/TRW

D. M. Ernst, J. R. Hartenstine, K. D. Horner-Richardson, J. E. Toth, "Refractory Metal Heat Pipes, Old Materials, New Problems," presented at the Advanced Aerospace Materials/Process Conference and Exposition, Long Beach, Calif., May 1990. THERMACORE, INC.

J. R. VanValzah, H. E. Eaton,* and R. B. Dinwiddie, "Thermal Aging Behavior in Plasma Sprayed Yttria Fully Stabilized Zirconia: Correlation of Thermal Conductivity Increase and Dimensional Shrinkage," presented at the International Conference of Metallurgical Coatings and Thin Films, San Diego, Calif., April 1991, and presented by R. B. Dinwiddie at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. UNITED TECHNOLOGIES RESEARCH CENTER

V. Srinivasan, R. A. Padgett, A. Choudhury, "Sulfidation Behavior of Fe-25Cr-20Ni-Si in $H_2H_2O/H_2S/Ar$ at 700 C," *Corros.* 47(9), 703-12 (1991).

2. INDUSTRY USERS' PENDING PUBLICATIONS AND PRESENTATIONS

T. A. Nolan, L. F. Allard, D. W. Coffey, C. R. Hubbard, R. A. Padgett, M. H. Rawlins, and R. W. Nixdorf, "Microstructure and Crystallography of Titanium Nitride Whiskers Grown by a VLS Process," *J. Am. Ceram. Soc.* (in press). AMERICAN MATRIX

H. F. Wu and M. K. Ferber, "Interfacial Mechanical Properties Characterization of Nicalon SiC Fiber/Alumina-based Composites," will be submitted to the *Sixth Japan-U.S. Conference on Composite Materials*, Orlando, Fla., June 1992. ALUMINUM COMPANY OF AMERICA

F. A. List, H. Hsu, O. B. Cavin, W. D. Porter, T. J. Henson, B. J. Reardon, C. R. Hubbard, and D. M. Kroeger, "Phase Development in the $Bi_2Sr_2Ca_1Cu_2O_y$ System: Effects of Oxygen Pressure," to be presented at the Materials Research Society Fall Meeting, Boston, Mass., December 3, 1991 (poster session), and to be submitted to *Physica C.* AMERICAN SUPERCONDUCTOR

D. A. Bowers and J. W. Sapp, "Thermal Characterization Tests of High Diffusivity Carbon Materials for High Heat Flux Applications," to be presented at the Fifth International Conference on Fusion Reactor Materials, Clearwater, Fla., November 1991. MCDONNELL DOUGLAS MISSILE SYSTEMS COMPANY

3. UNIVERSITY USERS

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," presented at the Pacific-International Congress on X-Ray Analytical Methods, Honolulu, Hawaii, August 1991. ALFRED UNIVERSITY

P. K. Choudhury, H. J. Rack, O. B. Cavin,* and C. R. Hubbard, "Effect of V on Elevated-Temperature Phase Relations in Titanium Aluminides Containing 43 at. % Al+," presented at International Conference on High-Temperature Aluminides and Intermetallics, San Diego, Calif., August 1991; abstract to be published in *Mater. Sci. Eng.* CLEMSON UNIVERSITY

H. Martin, C. K. Ober, and D. L. Kohlstedt, "Organoelement Polymer Precursors for Glass-Ceramic Composites," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991; in *Annual Meeting Abstracts*, p. 201. CORNELL UNIVERSITY

M. H. E. Martin and C. K. Ober, "Methacrylate Precursors to Oxide Glasses and Ceramics," pp. 534-35 in *Polymer Preprints*, Division of Polymer Chemistry, Inc., American Chemical Society, Vol. 32, No. 3, August 1991. CORNELL UNIVERSITY

J. A. Hanigofsky, K. L. More, W. J. Lackey, W. Y. Lee, and G. B. Freeman, "Composition and Microstructure of Chemically Vapor-Deposited Boron Nitride, Aluminum Nitride, and Boron Nitride + Aluminum Nitride Composites," *J. Am. Ceram. Soc.* **74**(2), 301-5 (1991). GEORGIA INSTITUTE OF TECHNOLOGY

T. E. Schlesinger, "Mechanical Properties of Granular Metal Thin Films and Kinetics of Silicide Formation in Chromium-Amorphous Silicon Multilayered Films," Ph.D. thesis, Johns Hopkins University, 1991. JOHNS HOPKINS UNIVERSITY

T. E. Schlesinger, R. C. Cammarata, A. Garvin, C. L. Chien, M. K. Ferber, and C. Hayzelden, "Enhanced Mechanical and Magnetic Properties of Granular Metal Thin Films," *J. Appl. Phys.* 70(6), 3275-80 (1991). JOHNS HOPKINS UNIVERSITY

H. J. Schock, Y. Chung, T. Rachel, D. S. Grummon, R. Schalek, R. M. Ronningen, W. C. McHarris, Y. Hsieh, and P. Gaydos, "Production and Implantation of Ions for Wear Studies and Monitoring of Machining Processes," MSUERL Report 91-3, June 1991. MICHIGAN STATE UNIVERSITY

E. Meyer, "Microstructural Characterization of the Oxidation Processes of Pyrite Single Crystals," presented at Women in Science, Knoxville, Tenn., April 1991. MOUNT HOLYOKE COLLEGE

M. H. Siadati, K. K. Chawla, and M. K. Ferber, "The Role of SnO_2 Interphase in an Alumina/Glass Composite: A Fractographic Study," *J. Mater. Sci.* **26**, 2743 (1991). NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

K. K. Chawla, "Interface Engineering in Alumina/Glass Composites," invited seminar, Department of Materials Science & Engineering, University of Tennessee, Knoxville, Tenn., March 1991. NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

K. K. Chawla, A. Choudhury,* R. Venkatesh, and J. R. Hellmann, "Microstructural Characterization of Alumina Fiber/Glass Composites With and Without an SnO_2 Interphase," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

A. Choudhury,* K. K. Chawla, J. Fernando, and J. R. Hellmann, "SIMS Characterization of Nextel 480 Fiber/Glass Composites," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

D. A. Koester, K. L. More, and R. F. Davis, "Steady-State Creep of Hot-Pressed SiC Whisker-Reinforced Silicon Nitride," presented at the U. S.-Japan Seminar on Processing, Microstructure, Development and Properties of Advanced Ceramics and Their Composites, Tokyo, Japan, August 1990; presented at the 1st Canadian International Composites Conference and Exhibition, Montreal, Canada, September 1991; presented at the 1st International Symposium on the Science of Engineering Ceramics, Osaka, Japan, October 1991; and submitted for publication to Elsevier Science Publishers. NORTH CAROLINA STATE UNIVERSITY

D. A. Koester, K. L. More, and R. F. Davis, "Deformation and Microstructural Changes in SiC Whisker-Reinforced Si_3N_4 Composites," *J. Mater. Res.* **6**(12), 2735 (1991). NORTH CAROLINA STATE UNIVERSITY.

K. L. More, D. A. Koester, and R. F. Davis,* "Creep of a SiC Whisker-Reinforced Si_3N_4 Composite in Compression and Bending," poster presentation at the 15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1991 (second-place award). NORTH CAROLINA STATE UNIVERSITY

K. L. More,* D. A. Koester, and R. F. Davis, "Creep Behavior of a SiC Whisker-Reinforced Si_3N_4 Composite in Air and Nitrogen," presented at the 2nd International Ceramic Science and Technology Congress, Orlando, Fla., November 1990. NORTH CAROLINA STATE UNIVERSITY

K. L. More, D. A. Koester, and R. F. Davis, "Microstructural Characterization of a Creep-Deformed SiC Whisker-Reinforced Si_3N_4," presented at the Frontiers in Electron Microscopy Meeting, Oak Brook, Ill., May 1990, and published in *Ultramicroscopy* **37**, 263-78 (1991). NORTH CAROLINA STATE UNIVERSITY

T. Dolney, "Data Processing of 3-D SIMS Ion Images on a Macintosh IIcx," presented at Women in Science, Knoxville, Tenn., April 1991. SAN HOUSTON STATE UNIVERSITY

A. A. Wereszczak* and A. P. Majidi, "Effect of Fracture Temperature and Relative Crack Propagation Rate on the Fracture Behavior of Whisker-Reinforced Ceramic Matrix Composites," presented at the 14th Annual Meeting of Advanced Ceramics and Composites, January 1990, and published in *Ceram. Eng. Sci. Proc.* **11** (7-8), 721-33 (July-August 1990). UNIVERSITY OF DELAWARE

B. Ballard, P. Predecki, and C. R. Hubbard,* "Residual Strains in Al_2O_3/SiC_w Composite from 25-1000°C," poster presentation at the 15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1991 (third-place award). UNIVERSITY OF DENVER

R. Srinivasan, B. H. Davis, O. B. Cavin, and C. R. Hubbard, "Phase Transformation and Crystallization Phenomena in $(Hf\text{-}Zr)O_2$ System; A High-Temperature X-ray Diffraction Study," poster at the PICXAM Conference in Honolulu, August 1991. UNIVERSITY OF KENTUCKY

R. Srinivasan, B. H. Davis, C. R. Hubbard,* O. B. Cavin, and W. D. Porter, "Phase Transformation in Sol-Gel Prepared Zirconia Using 'in-situ' High-Temperature X-ray Diffraction," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991. UNIVERSITY OF KENTUCKY

L. Wang, H. Wada, and L. F. Allard,* "Synthesis and Characterization of SiC Whiskers," presented at the 93rd Annual Meeting and Exposition of the American Ceramic Society, Cincinnati, Ohio, April 1991, and in *J. Mater. Res.* (in press). UNIVERSITY OF MICHIGAN

L. Wang, "Synthesis and Characterization of SiC Whiskers and Their Application in Si_3N_4 Matrix Composites," Ph.D. Thesis, The University of Michigan, Ann Arbor, Mich. 1991. UNIVERSITY OF MICHIGAN

L. F. Allard,* S. L. Mahan, T. A. Nolan, A. K. Datye, and R. T. Paine, "Atomic Structure of Model Ceramic-Ceramic Interfaces," presented at ASU Workshop on Structure and Properties of Interfaces, Wickenburg, Ariz., January 1991. UNIVERSITY OF NEW MEXICO

T. T. Borek, X. Qiu, L. M. Rayfuse, A. K. Datye, R. T. Paine, and L. F. Allard, "Boron Nitride Coatings on Oxide Substrates: Role of Surface Modifications," published in *J. Am. Ceram. Soc.* 74(10), 2587-91 (October 1991). UNIVERSITY OF NEW MEXICO

A. Choudhury and C. R. Brooks, "A Secondary Ion Mass Spectrometry (SIMS) Analysis of the Microstructure of an Embrittled 12% CR Steel Bolt," presented at the 24th Annual Convention of the International Metallographic Society, Monterey, Calif., July 1991. THE UNIVERSITY OF TENNESSEE

A. J. Pedraza, "Characterization of Sputter-Deposited 316L-Stainless Steel Films," in collaboration with M. J. Godbole, L. F. Allard, and G. Geesey, for the EPRI Review Panel, March 1991; submitted to *J. Mater. Sci.* THE UNIVERSITY OF TENNESSEE

N. B. Dahotre, S. Gopinathan, M. H. McCay, T. D. McCay, and C. M. Sharp, "An Evaluation of Strength of Pulsed Laser-Welded SiC Particulate Reinforced Al-alloy Metal Matrix Composite," in *Light Weight Alloys for Aerospace Applications: Advanced Composites*, ed. E. Lee, The Metallurgical Society of AIME, Warrendale, Pa., 1991. THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE

N. B. Dahotre, M. H. McCay, T. D. McCay, C. M. Sharp, S. Gopinathan, and L. F. Allard, "Laser Welding of a SiC/Al-Alloy Metal Matrix Composite," presented at the 9th International Congress on Applications of Lasers and Electro-Optics, Boston, Mass., November 1990.

M. H. McCay, C. M. Sharp, M. G. Womack, T. D. McCay, and N. B. Dahotre, "The Effect of Laser Pulse-Tailored Welding on Inconel 718," pp. 325-42 in *Laser Materials Processing*, Proceedings of ICALEO '90, LIA Vol. 71, Orlando, Fla., 1991. THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE

M. H. McCay, T. D. McCay, C. M. Sharp, and N. B. Dahotre, "Laser Welding Techniques for Alloy 718," pp. 719-34 in *Superalloys 718, 625, and Various Derivatives*, ed. E. A. Loria, The Minerals, Metals and Materials Society, Warrendale, Pa., 1991. THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE

N. B. Dahotre, M. H. McCay, T. D. McCay, and S. Gopinathan, "Pulse Laser Processing of a SiC/Al-Alloy Metal Matrix Composite," *J. Mater. Res.* 6(3), 514-29, March 1991. THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE

E. R. Puknys, J. J. Wert, and R. L. Freeny, "Effect of Composition and Processing Parameters on the Morphology, Mechanical Properties, and Subsequent Erosion Behavior of Thin Intermetallic Coatings," M. S. Thesis (E. R. Puknys, 1991); presented at the 5th International Conference on Surface Modification Technologies at the University of Birmingham, United Kingdom, September 1991, and published in the Proceedings. VANDERBILT UNIVERSITY

4. PENDING UNIVERSITY USERS' PUBLICATIONS & PRESENTATIONS

B. J. Reardon and C. R. Hubbard, "A Review of the XRD Data of the Phases Present in the CaO-SrO-CuO System," to be published in *Powder Diffr.*, ASM International. 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," to be published in *Powder Diffr.*, ASM International. ALFRED UNIVERSITY

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

M. H. E. Martin and C. K. Ober, "Methacrylate Precursors to Oxide Glasses and Ceramics," to be published in Am. Chem. Soc. Proc. (1991). CORNELL UNIVERSITY

R. U. Vaidya, J. Fernando, K. K. Chawla, and M. K. Ferber, "Effect of Fiber Coating on the Mechanical Properties of a Nextel 480 Fiber-Reinforced Glass Matrix Composite," accepted for publication in *Mater. Sci. & Eng.* NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

M. K. Ferber, R. L. Jackson, M. G. Jenkins, R. A. Lowden, and K. K. Chawla, "Evaluation of the Interfacial Mechanical Properties in Fiber-Reinforced Ceramic Composites," to be presented at the 16th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1992. NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY

B. R. Stoner, G.-H. M. Ma, S. D. Wolfen, and J. T. Glass, "Characterization of Bias-Enhanced Nucleation of Diamond on Silicon by In-Vacuo Surface Analysis and Transmission Electron Microscopy," submitted to Phys. Rev. B, October 1991. NORTH CAROLINA STATE UNIVERSITY

D. A. Koester, K. A. More, and R. F. Davis, "The High-Temperature Creep of a SiC Whisker-Reinforced Silicon Nitride in an Air Ambient," submitted to *J. Mater. Res.* NORTH CAROLINA STATE UNIVERSITY

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

J. M. Stencel, A. Sethuraman, R. E. Gonzalez, X.-X. Bi, A. Rubel, F. J. Derbyshire, P. E. Eklund, and C. R. Hubbard, "Ultrafine Particle Iron Carbide Catalysts Synthesized by Laser Pyrolysis: Physical and Chemical Transformations at High Temperature and Under Sulfating Conditions," to be presented at the Materials Research Society Fall Meeting, Boston, Mass., December 3, 1991 (Paper U060). UNIVERSITY OF KENTUCKY

L. Wang, H. Wada, and L. F. Allard, "Synthesis and Characterization of SiC Whiskers," *J. Mater. Res.* (in press). UNIVERSITY OF MICHIGAN

A. Choudhury, R. A. Padgett, M. Sanganeria, and C. R. Brooks, "An Auger Spectroscopy Examination of Fracture Surfaces in Be-Microalloyed Ni_4Mo," submitted to *J. Mater. Res.* THE UNIVERSITY OF TENNESSEE

A. J. Godbole, A. J. Pedraza, L. F. Allard, and G. Geesey, "Characterization of Sputter-Deposited 316L Stainless Steel Films," submitted to *J. Mater. Res.* THE UNIVERSITY OF TENNESSEE

5. HTML AND LOCAL USERS

L. F. Allard, "High-Resolution Electron in Microscopy in Materials Science," presented at WATTEC 91, Knoxville, Tenn., February 1991.

L. F. Allard, "High-Resolution Electron Microscopy of BN on MGO," ORAU traveling lecture presented at the Department of Chemical and Nuclear Engineering, University of New Mexico, Albuquerque, N.M., October 1990, and Department of Materials Science and Engineering, University of Michigan, Ann Arbor, Mich., November 1990.

L. F. Allard, "Image Processing in High-Resolution Electron Microscopy," presented at the 1991 Oak Ridge ASM International Educational Symposium, "Image-Based Analysis Techniques," Oak Ridge, Tenn., April 1991.

L. F. Allard, "The Ultrastructure of SiC Whisker-Reinforced Composites," presented at the Technical Seminar, Center for Laser Applications, University of Tennessee Space Institute, Tullahoma, Tenn., July 1991.

C. E. Bamberger, L. F. Allard, D. W. Coffey, and M. R. Bennett, "Whiskers of TiN by Topochemical Reaction," *J. Cryst. Growth* **112**, 47-54 (1991).

L. F. Allard,* D. C. Joy, and T. A. Nolan, "Initiative To Establish National Leadership in Electron Holography For High-Resolution Imaging and other Coherent Imaging Techniques," Director's Fund Proposal, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., September 1991.

L. F. Allard and T. A. Nolan, "Microcharacterization of Advanced Ceramics at the High Temperature Materials Laboratory (HTML), Oak Ridge National Laboratory," presented at the Seminar, Agency for Defense Development, Taejon, Korea, April 1991, and at the TEM 1991 Seminar, EG&G Mound Laboratories, Miamisburg, Ohio, May 1991.

L. F. Allard, T. A. Nolan, D. W. Coffey, and V. K. Sikka, "The Microstructure of a Tin Whisker-Reinforced Nickel Aluminide Metal-Matrix Composite," presented at the American Ceramic Society 92nd Annual Meeting and Exposition, Dallas, Tex., April 1990.

L. F. Allard, T. A. Nolan, V. J. Tennery, and E. E. Meyer, "The Use Of Pyrite Single-Crystal Substrates For Studies Of Pyrite Oxidation Mechanisms," presented at the 7th Annual Coal Preparation, Utilization, and Environmental Control Contractors Conference, where July 1991.

L. F. Allard and T. A. Nolan, "The Microstructure of Ceramic Whiskers," presented at the 49th Annual Meeting of the Electron Microscopy Society of America, San Jose, Calif., August 1991, in pp. 918-19 *Proceedings of the 49th Annual Meeting of the Electron Microscopy Society of America*, ed., G. W. Bailey, 1991; published by San Francisco Press.

V. K. Sikka, E. K. Ohriner,* and L. F. Allard, "Processing and Mechanical Properties of Ni_3Al-Based Intermetallic Composites," pp.137-45 in *P/M in Aerospace and Defense Technologies Symposium, Tampa, Florida, March 4-5, 1991*, Metal Powder Industries Federation, Princeton, N.J., 1991.

T. B. Lindemer, F. A. Washburn, C. S. MacDougall, and O. B. Cavin, "Synthesis of Y-Ba-Cu-O Superconductors in Subatmospheric Oxygen," *Physica C* **174**, 135-43 (1991).

A. Choudhury, "Secondary Ion Mass Spectrometry and Its Applications To Materials Science," presented at WATTEC 91, Knoxville, Tenn., February 1991.

A. Choudhury, "Secondary Ion Mass Spectrometry and Applications to Microstructural Analysis," presented at the 24th Annual Convention International Metallographic Society, Monterey, Calif., July-August 1991, and presented at the Graduate Student Seminar, Michigan Tech. University, Houghton, Mich., September 1991.

L. J. Romana,* P. S. Sklad, C. W. White, J. C. McCallum, A. Choudhury, L. L. Horton, and C. J. McHargue, "Formation and Annealing Behavior of an Amorphous Layer Induced by Tin Implantation Into Sapphire," presented at the 7th International Conference on Ion Surface Modification of Metals by Ion Beams, Washington, D.C., July 1991.

R. B. Dinwiddie, "An Introduction to Thermal Conductivity for Material Scientists," presented at a Materials Science and Engineering Seminar, University of Tennessee, Knoxville, Tenn., April 1991.

R. B. Dinwiddie,* T. D. Burchell, and C. F. Baker, "Investigation into the Effect of Heat Treatment on the Thermal Conductivity of 3-D Carbon/Carbon Fiber Composites," presented at the 20th Biennial Conference on Carbon, University of California at Santa Barbara, June 1991.

M. K. Ferber, "A New Ceramic Specimen Preparation User Center at the ORNL High Temperature Materials Laboratory," presented at The Workshop on Cost-Effective Ceramic Machining, Pollard Auditorium, Oak Ridge, Tenn., May 1991.

T. N. Tiegs,* M. K. Ferber, and J. O. Kiggans, "Microwave Annealing of Dense Si_3N_4 Ceramics," presented at the Properties of Irradiated Ceramics Conference, Los Alamos, N.M., June 1990; presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991; and presented at the European Ceramic Society Second Conference, Augsburg, Federal Republic of Germany, September 1991.

T. N. Tiegs,* J. O. Kiggans, M. K. Ferber, K. L. More, C. R. Hubbard, and D. W. Coffey, "Microstructure Development During Microwave Annealing of Dense Silicon Nitride," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991, and pp. 411-20 in *Ceramic Transactions: Microwave Theory and Applications in Materials Processing,* Vol. 21, ed. D. E. Clark, F. D. Gac, and W. H. Sutton, The American Ceramic Society, Westerville, Ohio.

T. N. Tiegs, J. O. Kiggans, M. K. Ferber, and H. D. Kimrey, "Microwave Processing of Si_3N_4 Ceramics" (D. P. Stinton, speaker only), presented at the 4th International Symposium on Ceramic Materials and Composites for Engines, Goteborg, Sweden, June 1991.

R. A. Lowden,* R. H. Krabill, and M. K. Ferber, "The Use of Fiber Coatings to Control the Mechanical Properties of Fiber-Reinforced Ceramic Composites," presented at the 15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1991.

C. R. Hubbard, "The High Temperature Materials Laboratory User Program and X-ray Diffraction and Thermophysical Properties of Selected Commercial and Experimental Silicon Nitrides," presented at the Materials Characterization Group, GTE Laboratories, Waltham, Mass., November 1990.

Camden R. Hubbard, "HTML User Program," *Am. Crystallographic Assoc. Newsletter* 1, February 1991.

C. R. Hubbard, R. B. Dinwiddie, O. B. Cavin, and W. D. Porter,* "Research Highlights from the X-ray Diffraction and Thermophysical Properties User Centers, High Temperature Materials Laboratory," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991.

C. R. Hubbard,* R. B. Dinwiddie, W. D. Porter, and M. K. Ferber, "The Effect of Intergranular Phases on the Thermophysical Properties of Silicon Nitride," presented at the Department of Materials Engineering, Virginia Polytechnic Institute and State University, Blacksburg, Va., November 1990, and presented at the Materials Research Society Fall Meeting, Boston, Mass., November 26-December 3, 1991.

C. R. Hubbard,* R. B. Dinwiddie, T. N. Tiegs, M. K. Ferber, and R. Yeckley, "Si_3N_4 Intergranular Phases and Thermal Diffusivity," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991.

C. R. Hubbard, D. Kroeger, R. K. Williams, and J. Brynestad, "$Y_1Ba_2Cu_40_9$ Superconductor - Preparation and Ca Doping," presented at The American Ceramic Society 92nd Annual Meeting and Exposition, Dallas, Tex., April 1990.

C. H. Hubbard, G. Zorn, and A. G. Siemens, "High-Temperature Diffraction," presented at the Pacific-International Conference on X-ray Analytical Methods, University of Hawaii, Hilo, Hawaii, September 1991.

J. O. Kiggans,* C. R. Hubbard, R. R. Steele, H. D. Kimrey, C. E. Holcombe, and T. N. Tiegs, "Characterization of Silicon Nitride Synthesized by Microwave Heating," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991.

M. G. Jenkins* and M. K. Ferber, "Determination of High-Temperature Creep Parameters in Structural Ceramics From Constant-Displacement, Load-Relaxation Tests," presented at the 1991 SEM Spring Conference on Experimental Mechanics, Milwaukee, Wis., June 1991.

M. G. Jenkins* and M. K. Ferber, "Stress-Relaxation Tests Used to Determine the Elevated-Temperature Creep Parameters of Structural Ceramics," presented at the International Conference on Fracture Mechanics of Ceramics, Nagoya, Japan, July 1991.

Asish Ghosh, Michael G. Jenkins,* Mattison K. Ferber, Jouko Peussa, and Jonathan A. Salem, "Elevated-Temperature Fracture Resistances (K_{IC}, R-Curves, γ_{WOF}) of Monolithic and Composite Ceramics Using Chevron-Notched, Bend Tests," presented at the International Conference on Fracture Mechanics of Ceramics, Nagoya, Japan, July 1991; to be published by Plenum Press, N.Y.

M. G. Jenkins,* M. K. Ferber, A. Ghosh, J. Peussa, and J. A. Salem, "Chevron-Notched, Flexure Tests for Measuring the Elevated-Temperature Fracture Resistances of Structural Ceramics," presented at the ASTM E24 Committee Meeting, Chevron-Notch Fracture Test Experience Metals and Non-Metals, Indianapolis, Ind., May 1991.

K. L. More, "Defect Characterization in a CVD α-Si_3N_4," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991; presented at the 49th Annual Meeting of the Electron Microscopy Society of America, San Jose, Calif., August 1991; and published in the Proceedings of the 49th Annual Meeting of the Electron Microscopy Society of America, ed. G. W. Bailey, San Francisco Press, 936-937 (1991).

R. E. Clausing,* L. Heatherly, L. L. Horton, K. L. More, D. D. Specht, and Z. L. Wang, "Structural Characterization of CVD Diamond," presented at the American Chemical Society National Meeting, New York, August 1991.

R. D. James, R. A. Lowden, and K. L. More, "The Effects of Oxidation and Combustion Environment on the Properties of Nicalon/SiC Composites," presented at the 2nd International Ceramic Science and Technology Congress, Orlando, Fla., November 1990; published in "Advanced Composite Materials," ed. M. D. Sacks, The American Ceramic Society, Westerville, Ohio *Ceram. Trans.* **19**, 925 (1991).

R. A. Lowden and K. L. More, "The Effect of Fiber Coatings on Interfacial Shear Strength and the Mechanical Behavior of Ceramic Composites," pp. 205-14 in *Materials Research Society Symposium Proceedings*, Vol. 170, symposium held at Boston, Mass., November 1990, Materials Research Society, Pittsburgh, 1991.

R. A. Lowden, K. L. More, T. M. Besmann, and R. D. James, "Microstructural Characterization of Multiphase Coatings Produced by Chemical Vapor Deposition," in "Chemical Vapor Deposition of Refractory Metals and Ceramics," ed. T. M. Besmann and B. M. Gallois, Materials Research Society, Pittsburgh *Mater. Res. Soc. Symp. Proc.*, **168**, 159 (1990).

R. A. Lowden,* R. D. James, and K. L. More, "Effects of Oxidizing and Combustion Environments on the Interface and the Mechanical Properties of Nicalon SiC Composites," presented at the 5th Annual Conference on Fossil Energy Materials, Oak Ridge, Tenn., May 1991.

D. P. Stinton, D. M. Hembree, Jr., K. L. More, B. W. Sheldon, and T. M. Besmann, "Characterization of Ceramic Matrix Composites Fabricated by Chemical Vapor Deposition," in "Chemical Vapor Deposition of Refractory Metals and Ceramics," ed. T. M. Besmann and B. M. Gallois, Materials Research Society, Pittsburgh *Mater. Res. Soc. Symp. Proc.* **168**, 273 (1990).

T. A. Nolan, L. F. Allard, M. K. Ferber, and D. W. Coffey, "Microstructural Characterization of Creep Deformation and Fatigue in a Si_3N_4 Ceramic," poster presentation at the 15th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Fla., January 1991. (first place award).

T. A. Nolan and L. F. Allard, "The High Temperature Materials Laboratory at Oak Ridge National Laboratory (ORNL) - A National Resource," presentation at the Agency for Defense Development, Taejon, Korea, April 1991.

J. H. Schneibel,* J. A. Horton, and W. D. Porter, "Bend Ductility and Physical Properties of Extruded Chromium-Modified Al_3Ti," presented at the International Conference on High-Temperature Aluminides and Intermetallics, San Diego, Calif., September 1991.

Monica A. Schmidt, "Recent Confocal Microscopy Work at ORNL," presented at the 1991 Oak Ridge ASM International Educational Symposium, "Image-Based Analysis Techniques," Oak Ridge, Tenn., April 18, 1991.

M. A. Schmidt, C. E. Holcombe, Jr., and C. E. Bamberger, "Analysis of Surface-Modified B_4C Powders," presented at the American Ceramic Society 93rd Annual Meeting and Exposition, Cincinnati, Ohio, April 1991.

M. A. Schmidt, M. A. Janney, and J. R. Mayotte, "SEM Image Analysis of Grain Growth in Microwave-Annealed Alumina," presented at the International Metallographic Society Meeting, Charlotte, N.C., July 1989; published in *Microstructural Science*, Vol. 18, ed. T. A. Place, J. D. Braun, W. E. White, and G. F. Vander Voort, 1989.

M. A. Schmidt, "Applications for Confocal Reflected-Light Microscopy in Materials Science," presented at the ASTM Symposium on Metallography: 75 Years Later, Atlantic City, N.J., May 1991.

M. A. Schmidt, "Recent Experience with a Confocal Tandem-Scanning Microscope," presented at the Appalachian Regional Electron Microscopy Society Meeting, Kingsport, Tenn., October 1990.

V. J. Tennery, "The Maximum Likelihood Method for Determining Weibull Distribution Function Estimators of Ceramic Materials" and "Recent Observations of Unexplained Variations in Measured α and β Parameters of 30 Specimen Data Sets of GN-10 Silicon Nitride - IEA Annex II, Subtask 5 -Room Temperature," presented at ASTM Committee C-28, Advanced Ceramics for the Probabilistic Procedures Task Group, San Antonio, Tex., November 1990.

V. J. Tennery and F. M. Foust, "High Temperature Materials Laboratory Third Annual Report (October 1989 through September 1990)," ORNL/TM-11735, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab., December 1990.

6. PENDING HTML & LOCAL USERS' PUBLICATIONS AND PRESENTATIONS

L. F. Allard, T. A. Nolan, and T. N. Tiegs, "Planar Defect Structures in SiC Whiskers," submitted to *J. Mater. Res.*

D. F. Wilson and O. B. Cavin, "Thermal Expansion Behavior of $NiSi/NiSi_2$," submitted to *Scr. Metall.* (1991).

A. Choudhury, J. R. DiStefano, and J. W. Hendricks, "Secondary Ion Mass Spectrometry (SIMS) Analysis of Nb-1% Zr Alloys," to be presented at the 9th Symposium on Space Nuclear Power Systems, Albuquerque, N.M., January 1992.

M. K. Ferber and M. G. Jenkins, "Empirical Evaluation of Tensile Creep and Creep Rupture in a HIPed Silicon Nitride," to be presented at the Fifth International Conference on Creep, Orlando, Fla., May 1992. (Published by ASM International.)

Y. Zhang, J. M. Stewart, C. R. Hubbard, and B. Morosin, "The Importance of Consistent 1/d Scans in Determining Size and Strain by Powder Diffraction Profile Analysis," submitted to *Adv. X-Ray Anal.*

M. G. Jenkins, M. K. Ferber, R. L. Martin, V. T. Jenkins, and V. J. Tennery, "Study and Analysis of the Stress State in a Ceramic, Button-Head, Tensile Specimen," ORNL/TM-11767, Martin Marietta Energy Systems, Inc., Oak Ridge Natl. Lab. (in press).

D. P. Stinton, D. M. Hembree, Jr., K. L. More, B. W. Sheldon, T. M. Besmann, M. H. Headinger, and R. F. Davis, "Matrix Characterization of Fiber-Reinforced SiC Matrix Composites Fabricated by Chemical Vapor Infiltration," submitted to *J. Am. Ceram. Soc.*

J. L. Ding, K. C. Liu, K. L. More, C. R. Brinkman, and D. R. Johnson, "Experimental Characterization of Creep and Creep Rupture of Advanced Silicon Nitride Ceramics," submitted to *J. Am. Ceram. Soc.*

M. A. Schmidt and R. D. Compton, "Confocal Microscopy," submitted to *Metals Handbook: Friction, Lubrication and Wear Technology*, Vol. 4, 10th Ed. (1991).

INTERNAL DISTRIBUTION

1-2. Central Research Library
3. Document Reference Section
4-5. Laboratory Records Department
6. Laboratory Records, ORNL-RC
7. ORNL Patent Section
8-10. M&C Records Office
11. J. Bentley
12. A. Bleier
13. K. W. Boling
14. R. A. Bradley
15. V. R. Bullington
16. O. B. Cavin
17. D. F. Craig
18. R. B. Dinwiddie
19-423. F. M. Foust
424. F. M. Haggag
425. L. L. Horton
426. C. Hsueh
427. C. R. Hubbard
428. M. A. Janney
429. M. G. Jenkins
430. M. A. Karnitz
431. E. A. Kenik
432. O. F. Kimball
433. G. M. Ludtka
434. R. C. Martin
435. J. R. Mayotte
436. D. J. McGuire
437. T. A. Nolan
438. S. D. Nunn
439. J. T. Parks
440. A. E. Pasto
441. D. F. Pedraza
442. P. S. Sklad
443. G. M. Slaughter
444. J. O. Stiegler
445-449. V. J. Tennery
450. L. J. Turner
451. J. R. Weir, Jr.
452. A. A. Wereszczak
453. A. D. Brailsford (Consultant)
454. Y. A. Chang (Consultant)
455. H. W. Foglesong (Consultant)
456. J. J. Hren (Consultant)
457. M. L. Savitz (Consultant)
458. J. B. Wachtman (Consultant)

EXTERNAL DISTRIBUTION

459. AIR PRODUCTS, R&D Lab No. 1, 7201 Hamilton Boulevard, Allentown, PA 18195

F. Kimock

460-464. ALFRED UNIVERSITY, New York State College of Ceramics, P.O. Box 771, Alfred, NY 14802

D. F. Dockery
D. P. Matheis
J. J. Simmons
R. L. Snyder
J. Taylor

465-466 ALLIED-SIGNAL AEROSPACE COMPANY, 717 N. Bendix Drive, South Bend, IN 46620

R. Silvers
R. Rateich

467-469. ALLIED-SIGNAL AEROSPACE COMPANY, Garrett Ceramic Components Division, 19800 South Van Ness Avenue, Torrance, CA 90509

B. J. Busovne, Jr.
M. V. Mitchell
H. C. Yeh

470-477. ALLIED-SIGNAL AEROSPACE COMPANY, Garrett Engine Division, MS 301-2N, 111 S. 34th St., P.O. Box 5217, Phoenix, AZ 85010

C. Balis
M. Cardenos
H. Fang
J. T. Hartman
L. J. Lindberg
M. N. Menon
B. O'Halloran
J. Schienle

478-480. ALLIED-SIGNAL CORPORATE TECHNOLOGY, Box 1021 R, Morristown, NJ 07960

C. Ballard
J. Nick
P. Whalen

481. ALLIED-SIGNAL ENGINEERED MATERIALS RESEARCH CENTER, 50 East Algonquin Road, Box 5016, Des Plaines, IL 60017

K. R. Karasek

482-484. ALUMINUM COMPANY OF AMERICA, Alcoa Technical Center, Product Design and Mechanics Division, Alcoa Center, PA 15069

M. Fishkis
R. A. Marra
H. F. Wu

485-486. AMERICAN SUPERCONDUCTOR, 149 Grove Street, Watertown, MA 02172

P. Gallien
H. S. Hsu

487. APPLIED SCIENCES, INC., 800 Livermore Street, Yellow Springs, OH 45387

J.-M. Ting

488. CARBOMEDICS, INC., 1300 B East Anderson Lane, Austin, TX 78753

D. W. James

489-492. THE CARBORUNDUM CO., P.O. Box 832, Bldg. 100, Niagara Falls, NY 14302

K. Y. Chia
K. R. Sellkregg
S. G. Seshadri
R. Storm

493-494. CATERPILLAR, INC., Technical Center, Bldg. E, P.O. Box 1875, Peoria, IL 61656-1875

H. D. Lawerence
L. Voellinger

495-501. CERAMICS PROCESS SYSTEMS, 155 Fortune Blvd., Milford, MA 01757

J. D. Hodge
W. C. James Wei
L. J. Klemptner
R-R. Lee
B. Novich
M. Parrish
D. L. Quellette

502. CHRYSLER CORPORATION, CIMS 418-17-09, 12000 Chrysler Drive, High Land Park, MI 48288-1118

D. Wetzel

503-520. CLEMSON UNIVERSITY, College of Engineering, Department of Mechanical Engineering, 318 Riggs Hall, Clemson, SC 19634-0921

P. K. Chaudhury
C. C. Fain
M. Grujicic
J. Jara-Almonte
B. Lee
M. C. Long
V-V. Modi
C. P. Narayan
D. Peeler
J. Polizzi
K. Preston
H. J. Rack
D. K. Rogers
S. Tangrila
T. D. Taylor
P. K. Wachtel
J. S. Wolf
J-M. Wung

521-523. COORS CERAMICS CO., 17750 W 32nd Avenue, Golden, CO 80401

J. Coors
M. Readey
J. Sibold

524-527. COORS TECHNICAL CERAMIC CO., 1100 Commerce Park Drive, Oak Ridge, TN 37830

W. K. Baxter
J. Ghinazzi
W. Howe
E. D. Winters

528-529. CORNELL UNIVERSITY, Bard Hall, Ithaca, NY 14853-1501

H. Maring
C. K. Ober

530-531. CORNING GLASS WORKS, Technical Ceramics, MP WX 01 05, Corning, NY 14831

M. J. Henderson
W. R. Prindle

532. CONSULTING SERVICES, 12 Holland Road, Pittsburgh, PA 15235

C. Schacht

533-537. CUMMINS ENGINE CO., INC., Box 3005, MC 50183, Columbus, IN 47202

J. Bokelman
J. E. Denton
J. W. Patten
B. E. Wilde
T. Yonushonis

538. CVC Products, 6305 York Road, Suite 40, Baltimore, MD 21212

F. Grunewald

539. DARTMOUTH COLLEGE, Thayer School of Engineering, Hanover, NH 03755

I. Baker

540-545. DETROIT DIESEL CORPORATION, World Headquarters, 13400 Outer Drive West, Detroit, MI 48239-4001

D. Giles
C. L. Hoberty
J. Hyde
T. Kaushal
W. Lakso
K. Weber

546. DETROIT GASKET, Route 7, Box 7, Newport, TN 37821

M. L. Surbrook

547. DG TRIM PRODUCTS, 3204 Regal Drive, Alcoa, TN 37701

M. W. Hobbs

548-559. THE DOW CHEMICAL CO., Analytical Science Laboratory, Midland, MI 48674

Y-H. Chiao	B. Newman
T. Guiton	G. Potter
C. Haney	A. Pyzik
A. M. Hart	W. Rafaniello
C. J. Hwang	P. D. Stone
A. Knudson	J. Tou

560-562. DOW CORNING CORPORATION, 3901 S. Saginaw Road, MS 43, Midland, MI 48640

W. E. Hauth
M. J. Loboda
S. S. Snow

563-566. EG&G MOUND APPLIED TECHNOLOGIES, INC., P.O. Box 3000, Miamisburg, OH 45343

J. Birkbeck
P. Gray
D. Kramer
W. E. Moddeman

567. EKZO CHEMICAL, INC., Livingston Avenue, Dobbs Ferry, NY 10522

K. Kiss

568-569. ENERGY CONVERSION DEVICES, INC., 1675 West Maple Road, Troy, MI 48084

B. Chao
R. T. Young

570-573. ENGELHARD CORPORATION, Menlo Park, P.O. Box 2900, Edison, NJ 08818-2900

T. A. Gegan	T. W. Langner
M. Hamil	J. St. Amand

574. ETHEL CORPORATION, Ethyl Technical Center, P.O. Box 14799, Baton Rouge, LA 70898

M. Gluckstein

575-576. FIBER MATERIALS, INC., Biddeford Industrial Park, Biddeford, ME 04005

D. Dunn
M. K. Marshall

577-578. FMC CORPORATION, Advanced Systems Center, 1300 South Second Street, P.O. Box 59043, Minneapolis, MI 55459-0043

M. J. Kneer
J. Koo

579. FORD ELECTRONICS TECHNOLOGY CENTER, 17000 Rotunda Drive, Dearborn, MI 48121

K. Salisbury

580-581. FOSTER-MILLER, INC., 350 Second Avenue, Waltham, MA 02254

R. D. Tetrault
P. Stark

582. GBC CORPORATION, Monestary Drive, LaTrobe, PA 15650

E. R. Tostevin

583-584. GENERAL ELECTRIC AIRCRAFT ENGINES, 1 Neumann Way, Cincinnati, OH 45215

R. Harrision
L. Lisiecki

585-586. GENERAL MOTORS, Allison Gas Turbine Operations, P.O. Box 420, Indianapolis, IN 46206-54748

L. Groseclose
H. E. Helms

587-595. THE GEORGIA INSTITUTE OF TECHNOLOGY, 110 Baker Building, Atlanta, GA 30332

G. Freeman
J. A. Hanigofsky
T. L. Jennings
W. J. Lackey
H. R. Last
J. Lyons
D. L. Mohr
T. H. Sanders
T. L. Starr

596-599. THE GEORGIA INSTITUTE OF TECHNOLOGY, Center for the Advancement of Computational Mechanics, Department of Civil Engineering, MC 0355, Atlanta, GA 30332

S. N. Atlur
P. Desai
J. S. Epstein
G. May

600-601. GREAT LAKES RESEARCH, P.O. Box 1831, Elizabethton, TN 37643

L. A. Joo
E. G. Morris

602-607. GTE LABS, INC., 40 Sylvan Road, Waltham, MA 02254

F. E. Ivo
J. Hefter
F. Strouse
C-M. Sung
W. C. VanSchalkwyk
G. Wei

608. GTE LABS, Valenite, Materials Manufacturing, 1100 West 13th Mile Road, Madison Heights, MI 48071

J. T. Smith

609. HiTc SUPERCONCO, INC., Lambertville, NJ 08530

R. B. Cass

610-612. INSTITUTE FOR DEFENSE ANALYSES, 1801 N. Beauregard Street, Alexandria, VA 22311

W. S. Hong
M. A. Rigdon
J. M. Sater

613. INSTRON, Corporate Technical Director, 100 Royall Street, Canton, MA 02021

J. Amaral

614. INSTRON CORPORATION, 3781 N.E. Expressway, Suite 132, Atlanta, GA 30340

M. Mayer

615-618. IONIC ATLANTA, INC., 1347 Spring Street, Atlanta, GA 30309

K. O. Legg
S. G. Pope
H. Solnick-Legg
J. R. Stevenson

619-620. IOWA STATE UNIVERSITY, 110 Engineering Annex, Amex, Iowa 50011

M. Akinc
A. Thom

621. ISCAR CERAMICS, INC., 36733 Amrhein, Livonia, MI 48150

J. Barta

622-623. THE JOHNS HOPKINS UNIVERSITY, Department of Materials Science and Engineering, 102 Maryland Hall, Baltimore, MD 21218

R. C. Cammarata
T. E. Schlesinger

624. KEMET ELECTRONICS, P.O. Box 5928, Greenville, SC 29606

L. Mann

625. KANTHAL CORPORATION, 119 Wooster Street, Bethel, CN 06801

R. Watson

626. LANXIDE CORPORATION, Tralee Industrial Park, P. O. Box 6077, Newark, DE 19714-6077

J. Wang

627. LAWRENCE LIVERMORE LABORATORY, P.O. Box 808, MS L 566, Livermore, CA 94550

A. Tesar

628. LITTON INDUSTRIES, P.O. Box 87, 5500 Canoga Avenue, Woodland Hills, CA 91367

R. A. Hansen

629. MARTIN MARIETTA AERO NAVAL SYSTEMS, 103 Cheasepeake Park Plaza, Baltimore, MD 21220

D. Trawinski

630. MARTIN MARIETTA LABORATORIES, 1450 South Rolling Road, Baltimore, MD 21227

A. Barrett

631-632. MASSACHUSETTS INSTITUTE OF TECHNOLOGY, Department of Materials Science and Engineering, 77 Massachusetts Avuenue, Cambridge, MA 02139

S. B. Brown
J. Connally

633-634. MCDONNELL DOUGLAS CORPORATION, P.O. Box 516, St. Louis, MO 63166

D. A. Bowers
J. W. Sapp, Jr.

635-637. MICHIGAN STATE UNIVERSITY, Engineering Research Facility, 2857 Jolly Road, Okemos, MI 48864

T. Rachel
R. Schalek
H. Schock

638-643. MICHIGAN TECHNOLOGY UNIVERSITY, Houghton, MI 49931

R. Buist
C. Cauchy
L. Heldt
J. Lukowski
D. Nelson
C. White

644-646. MINIATURE PRECISION BEARINGS, Division of MPB Corporation, Precision Park, P.O. Box 547, Keene, NH 03431-0547

K. M. Gordon
R. A. Hanson
R. L. Houghton

647-648. MOBIL RESEARCH AND DEVELOPMENT CORPORATION, Central Research Laboratory, Pennington Rocky Hill Road, Pennington, NJ 08534

C. R. Aguinaldo
D. Olson

649-650. MONARCH TILE MANUFACTURING, INC., P.O Box 999, Florence, AL 35630

D. Brosnan
J. Ranney

651. MONTANA TECHNOLOGY COMPANY, INC., Applied Testing Laboratory, 321 West Galena Street, Butte, MT 59701

F. Bradley

652-653. MOUNT HOLYOKE COLLEGE, Physics Department, South Hadley, MA 01075

E. Meyer
H. Nicholson

654. MSE, INC., P.O. Box 3767, Butte, MT 59702

V. Griffiths

655-656. NASA LANGLEY RESEARCH CENTER, Mail Stop 194, Hampton, VA 23665

R. K. Clark
K. E. Wiedeman

657-659. NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY, Department of Materials and Metallurgical Engineering, Socorro, NM 87801

K. K. Chawla
J. Hao
P. A. Lessing

660-661. NATIONAL INSTITUTE OF STANDARDS TECHNOLOGY, Ceramics Division, Gaithersburg, MD 20899

L. K. Ives
N. Peterson

662-667. NORTH CAROLINA STATE UNIVERSITY, Department of Materials Engineering, Box 7907, Raleigh, NC 27695-7907

S. Ailey-Trent
J. Cirra
R. F. Davis
J. Glass
D. A. Koester
M. Ma

668-669. NORTH CAROLINA STATE A&T UNIVERSITY, Department of Mechanical Engineering, Greensboro, NC 27411

J. Sankar
R. Vaidyanathan

670-682. NORTON COMPANY, High Performance Ceramics, Goddard Road, Northboro, MA 01532-1545

N. D. Corbin
R. Cravens
D. J. Devlin
S. Fitzgerald
M. Foley
S. D. Hartline
J. N. Panzarino
D. O. Patten
G. Rossi
K. N. Siebein
G. J. Sundberg
R. L. Yeckley
C. Willkins

683. NUCLEAR & AEROSPACE MATERIALS CORPORATION, 16716 Martincoit Road, Poway, CA 92064

G. B. Engle

684. OFFICE OF SCIENCE AND TECHNOLOGY POLICY, Old Executive Office Building, 175th Street and Pennsylvania Avenue, Washington, DC 20506

W. D. Phillips

685. OKLAHOMA STATE UNIVERSITY, Physics Department, Stillwater, OK 07478

C. Allison

686. OPTICAL COATING LABORATORY, 2789 Northpoint Parkway, Santa Rosa, CA 95407

J. Swab

687. PCC AIRFOILS, 5105 Rex McLeod Road, Stanford, NC 27330

T. Crofts

688-701. THE PENNSYLVANIA STATE UNIVERSITY, 410 Walker Building, University Park, PA 16802

J. H. Adair
A. S. Bhalla
L. E. Cross
G. Fox
S. B. Krupanidhi
S. K. Kurtz
K. Newman
C. A. Randall
G. Rossetti
A. E. Segall
K. Spear
R. Tressler
T. R. Trout
P. Zhang

702-705. QUADRAX CORPORATION, 300 High Point Avenue, Portsmouth, RI 02871

A. Bailey
R. Florentine
R. Kiernan
M. Mello

706. REFRACTORY TESTING ASSOCIATION, 91 Wallis Road, Chestnut Hill, MA 02167

O. Buyukozturk

707. RELIABILITY AND PERFORMANCE ASSOCIATES, P.O. Box 53102, Knoxville, TN 37950-3102

W. Lau

708-709. RENSSELAER POLYTECH INSTITUTE, Troy, NY 12180

E. Lara-Curzio
S. S. Sternstein

710-713. RICE UNIVERSITY, Department of Mechanical Engineering and Materials Science, P.O. Box 1892, Houston, TX 77251

P. Gillespie
S. Harding
G. M. Pharr
W. Wilson

714. ROCKWELL INTERNATIONAL CORPORATION, Science Center, Thousand Oaks, CA 91360

C. Banton

715-717. RUTGERS UNIVERSITY, Center for Ceramics Research, P.O. Box 909, Piscataway, NJ 08855-0909

R. Anderson
D. Niesz
A. Zutshi

718. SAINT VINCENT COLLEGE, LaTrobe, PA 15650

Brother Norman

719. SAN JOSE STATE UNIVERSITY, Department of Physics, San Jose, CA 95192-0106

F. Freund

720. SELEE CORPORATION, P.O. Box 747, Hendersonville, NC 38793

P. A. Bosomworth

721-723. SOLAR TURBINES INCORPORATED, P.O. Box 85376, San Diego, CA 92186

B. Harkins
V. Parthasarathy
M. Van Roode

724-725. SOUTHERN ILLINOIS UNIVERSITY, Rural Route 10, Box 99B, Carbondale, IL 62901

W. Trimble
D. E. Wittmer

726. SOUTHERN UNIVERSITY, Mechanical Engineering Department, Baton Rouge, LA 70813

A. R. Mirshams

727. SPALDING SPORTS WORLDWIDE, 425 MEADOW STREET, P.O. Box 901, Chicopee, MA 01021-0901

T. C. Walton

728. STANDARD OIL ENGINEERED MATERIALS, 501 New York Avenue, Falcon, NY 14733

A. R. Lebold

729-730. STANFORD UNIVERSITY, Peterson Building, Stanford, CA 94305

L. J. Farthing
D. A. Stevenson

731-733. SULLIVAN MINING CORPORATION, P.O. Box 4615, San Diego, CA 92104

T. M. Pattison
D. W. Peterson
T. M. Sullivan

734-742. TELEDYNE ALLVAC, P.O. Box 5030, Monroe, NC 28110-0531

W. D. Cao
A. Gallagher
B. P. Hudson
L. Jackman
R. L Kennedy
V. Lowery
J. Moyer
S. Taylor
S. Vallandingham

743. TENNESSEE CENTER FOR RESEARCH AND DEVELOPMENT, Institute of Resonance Ionization Spectroscopy, 10521 Research Drive, Suite 300, Knoxville, TN 37932

J. E. Parks

744. TENNESSEE TECHNOLOGICAL UNIVERSITY, Center for Manufacturing Research, Brown Hall, Room 222, Cookeville, TN 38505

T. C. Ramaraj

745-752. TEXTRON SPECIALTY MATERIALS, 2 Industrial Avenue, Lowell, MA 01851

T. Carroll
J. D. Casey
S. DiPietro
D. Giannelli
E. Honig
R. Krutenat
R. Pepper
G. Thurston

753-756. THERMACORE, INC., 780 Eden Road, Lancaster, PA 17601

G. Y. Eastman
D. M. Ernst
J. R. Hartenstine
R. Keller

757. THERMO CERAMICS, INC., Box 923, Augusta, GA 30903

M. Katagiri

758-759. THIRD MILLENNIUM TECHNOLOGIES, INC., 408 Cedar Bluff Road, Suite 383, Knoxville, TN 37923

S. C. Weaver
S. G. Padron

760-765. TUSKEGEE UNIVERSITY, Tuskegee, AL 36088

H. A. Aglan
A. Haque
M. Huq
S. Jeelani
F. Wilks
D. Zadoo

766. UNION CARBIDE CORPORATION, 1500 Polco Street, Indianapolis, IN 46224

A. Sue

767-768. UNIVERSAL ENERGY SYSTEMS INC., 4401 Dayton-Xenia Road, Dayton, OH 45432-1894

R. S. Bhattacharya
V. Srinivasan

769-774. THE UNIVERSITY OF ALABAMA-TUSCALOOSA, School of Mines and Energy Development, P.O. Box 870204, Tuscaloosa, AL 35487-0204

J. B. Gantenburg
R. Holland
W. R. Lacefield
J. R. Levine
L. Lucas
J. L. Ong

775-777. UNIVERSITY OF ARIZONA, Department of Materials Science and Engineering, Tucson, AZ 85721

B. Fabes
G. Frantiskonis
T. Kundu

778. UNIVERSITY OF CALIFORNIA-DAVIS, Department of Mechanical Engineering, Davis, CA 95616

J. C. Gibeling

780-780. UNIVERSITY OF CALIFORNIA-LOS ANGELES, Department of Earth and Space Sciences, 405 Hilgard Avenue, Los Angeles, CA 90024

P. Bird
A. Gratz

781. UNIVERSITY OF CALIFORNIA-SANTA BARBARA, Santa Barbara, CA 93106

F. F. Lange

782. THE UNIVERSITY OF DAYTON RESEARCH INSTITUTE, 300 College Park, Dayton, OH 45469-0001

N. L. Hecht

783-784. UNIVERSITY OF DELWARE, Department of Mechanical Engineering, Newark, DE 19716

I. Hall
A. Parvizi-Majdi

785-786. UNIVERSITY OF DENVER, Engineering Department, University Park, Denver, CO 80208

D. Ballard
P. K. Predecki

787-794. UNIVERSITY OF FLORIDA, Department of Materials Science and Engineering, Gainesville, FL 32611

D. E. Clark	M. J. Kaufman
A. Duncan	A. Marrone
F. Feller	J. Mecholsky
L. Hehn	S-M. Sim

795. UNIVERSITY OF ILLINOIS, Department of General Engineering, 117 Transportation Building, 104 S. Mathews Avenue, Urbana, IL 61801-2996

H. LM D. Reis

796-803. UNIVERSITY OF ILLINOIS, Department of Materials Science and Engineering, 105 South Goodwin Avenue, Room 212A, Urbana, IL 61801

A. Burreson	M. A. Kelly
J. P. Chu	J. M. Rigsbee
D. M. Hansen	M. M. Shaw
J. Homeny	J. R. Vanvalzah

804-810. UNIVERSITY OF KENTUCKY, Center for Applied Energy Research, 3572 Iron Works Pike, Lexington, KY 40511-8433

B. H. Davis	A. R. Sethuraman
P. Eklund	R. Srinivasan
Y. Q. Fei	J. Stencel
R. Gonzalez	

811-817. THE UNIVERSITY OF MICHIGAN, Materials Science and Engineering, 2201 H. H. Dow Building, Ann Arbor, MI 48109-2136

W. C. Bigelow
J. W. Jones
S. D. Nunn
T. Y. Tien
H. Wada
L. Wang
G. S. Was

818-819. UNIVERSITY OF MINNESOTA, Department of Chemical Engineering and Materials Science, 421 Washington Avenue SE, Minneapolis, MN 55455

M. L. McCartney
Y. J. Zhang

820. THE UNIVERSITY OF NEW MEXICO, Department of Chemical and Nuclear Engineering, Albuquerque, NM 87131

A. K. Datye

821-840. THE UNIVERSITY OF TENNESSEE, Department of Geological Sciences, Knoxville, TN 37996-2200

C. R. Brooks
S. Cao
N. B. Dahorte
J. Danko
J. F. Fellers
G. G. Geesey
M. J. Godbole
S. Gopinathan
D. Joslin
K. K. Khan
Y-C. Lin
C. D. Lundin
D. McCay
M. McCay
A. J. Pedraza
J. Shireman
J. E. Spruiell
C. Swindeman
S. D. Webb
H. White

841. THE UNIVERSITY OF TENNESSEE, Center for Materials Processing, 101 Perkins Hall, Knoxville, TN 37996-2000

C. G. McHargue

842-845. THE UNIVERSITY OF TENNESSEE SPACE INSTITUTE, Center for Laser Applications, Tullahoma, TN 37388-8897

B. Boss
N. B. Dahotre
M. H. McCay
T. D. McCay

846-847. UNIVERSITY OF UTAH, Department of Materials Science and Engineering, Salt Lake City, UT 84112

D. Drobeck
A. V. Virkar

848. U.S. BUREAU OF MINES, Tuscaloosa Research Center, P.O. Box L, Tuscaloosa, AL 35486

L. Sadler

849-859. VANDERBILT UNIVERSITY, Box 6309 Station B, Nashville, TN 37235

G. Bertero
G. Carro
L. Hall
W. H. Hofmeister
R. Hopkins
R. Kegley
J. Olive
R. A. Poggie
E. Puknys
J. J. Wert
J. Wettig

860-869. VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY, Materials Engineering Department, 301 Burruss Hall, Blacksburg, VA 24061

M. Annen
J. J. Brown
M. Davis
S. Desu
R. Gordon
R. Hendricks
C-H. Peng
F. Rivet
R. E. Swanson
S. M. Van Aken

870. WQED - THE INFINITE VOYAGE, 3171 Los Seliz Blvd., Suite 201, Los Angeles, CA 90039

R. Corsini

871-876. DOE, OFFICE OF ASSISTANT SECRETARY FOR CONSERVATION AND RENEWABLE ENERGY, Office of Transportation Technology, Forrestal Building, 1000 Independence Avenue, Washington, DC 20585

A. A. Chesnes (CE-30)
J. J. Eberhardt (CE-34)
T. G. Gross (CE-34)
R. B. Schulz (CE-34)
T. Vojnovich (CE-34)
A. M. Zerega (CE-34)

877. DOE, P.O. Box 10940, Pittsburgh, PA 15236-0940

E. Ramer

878. DOE, OAK RIDGE FIELD OFFICE, P.O. Box 2008, Oak Ridge, TN 37831-6269

Assistant Manager for Energy Research and Development

879-888. DOE, OFFICE OF SCIENTIFIC AND TECHNICAL INFORMATION, P.O. Box 62, Oak Ridge, TN 37831

For distribution by microfiche as shown in DOE/OSTI-4500, Distribution Categories UC-322 (Ceramics/Advanced Materials) and UC-365 (High-Temperature Materials).