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HIGH TEMPERATURE MATERIALS LABORATORY SIXTH ANNUAL REPORT (OCTOBER 1992 THROUGH SEPTEMBER 1993)

V. J. Tennery
F. M. Foust

MANAGED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
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Metals and Ceramics Division

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managed by
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Research projects with activity during FY 1993 are listed here by title, user institution, and the User Center involved. A brief summary can be found on the pages indicated.

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PROJECTS OF UNIVERSITY USERS

HIGH TEMPERATURE MATERIALS LABORATORY FIFTH ANNUAL REPORT* (OCTOBER 1992 THROUGH SEPTEMBER 1993)

V. J. Tennery and F. M. Foust

ABSTRACT

The High Temperature Materials Laboratory has completed its sixth 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 executing user agreements since the facility began operation in 1987. A total of 172 nonproprietary agreements (88 university and 84 industry) and 35 proprietary agreements (2 university, 33 industry) are now in effect. Six other government facilities have also participated in the User Program. Thirty-eight states are represented by these interactions.

Ninety-four nonproprietary research proposals (44 from universities, 47 from industry, and 3 from other government facilities) and three proprietary proposals were considered during this reporting period. Nonproprietary research projects active in FY 1993 are summarized.

1. INTRODUCTION

The High Temperature Materials (HTML) is a modern research facility that houses an array of special instruments used to meet research needs for advanced materials, including structural ceramics and alloys. The research instruments in the six HTML User Centers provide a comprehensive set of tools for performing state-of-the-art determination of the structure and properties of solids. A key part of the HTML concept includes a staff of highly trained technical personnel who work 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 involvement with research personnel such as (1) conducting research relating material 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 Energy Efficiency 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.

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, 207 agreements have been executed. The first users from the university and industrial community started their research projects in August 1987.

Three advisory committees assist in the successful operation of the HTML User Program. The function of these committees and a listing of their current members are found in Appendix A.

Two types of standard agreements are utilized in the HTML User Program: the "Non-proprietary Agreement" for research projects whose results are reported in the open literature within 6 months of the completion of the project in the HTML and the "Proprietary Agreement" for all projects in which the user desires that the data and results be proprietary. Appendix B is a listing of institutions executing nonproprietary user agreements to date. Due to the sensitive nature of many of the proprietary research activities, the names of the user institutions for proprietary agreements are not listed. In addition, researchers from five other government facilities have participated in the HTML User Program to date.

HTML MISSION STATEMENT

TO HELP INDUSTRY AND UNIVERSITY MATERIALS ENGINEERS AND SCIENTISTS CONDUCT THEIR RESEARCH ON IMPROVING CURRENT MATERIALS AND ON COMMERCIALIZING NEW MATERIALS AS EFFICIENTLY AS POSSIBLE.

2. USERS

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

The category of users and number of user days accumulated during this sixth year of HTML operation are shown in Table 1.

Table 1. HTML FY 1993 user experience

Type of user	Number of		User days
	Institutions	Individuals	
Industry	30	46	2695
University	20	47	1353
Local Oak Ridge users	1	101	9666
Totals:	51	194	13,714

Figure 1 shows the total number of 8-h user days per quarter during FY 1993. The industrial user days varied from 441 to 861 during a particular quarter, and university user days ranged from 175 to 409. An additional number of user days, ranging from 2027 to 3093, were accumulated by researchers employed at ORNL. Figure 2 shows the number of user days for all industrial and university users in the HTML for each quarter of FY 1993.

To date, 428 research proposals have been submitted to the User Program by researchers from universities, industrial companies, and government laboratories other than ORNL. (ORNL researh staff are not required to submit a research proposal; however, there is a fee charged to those researchers for use of the instruments.) A breakdown by user category of these proposals is given in Table 2. Figure 3 is a graph indicating proposals received by fiscal year of operation. Access clearances have been initiated on more than 500 individual researchers who were listed as principal investigators on these proposals. A listing of publications and presentations resulting from user research projects is given in Appendix D.

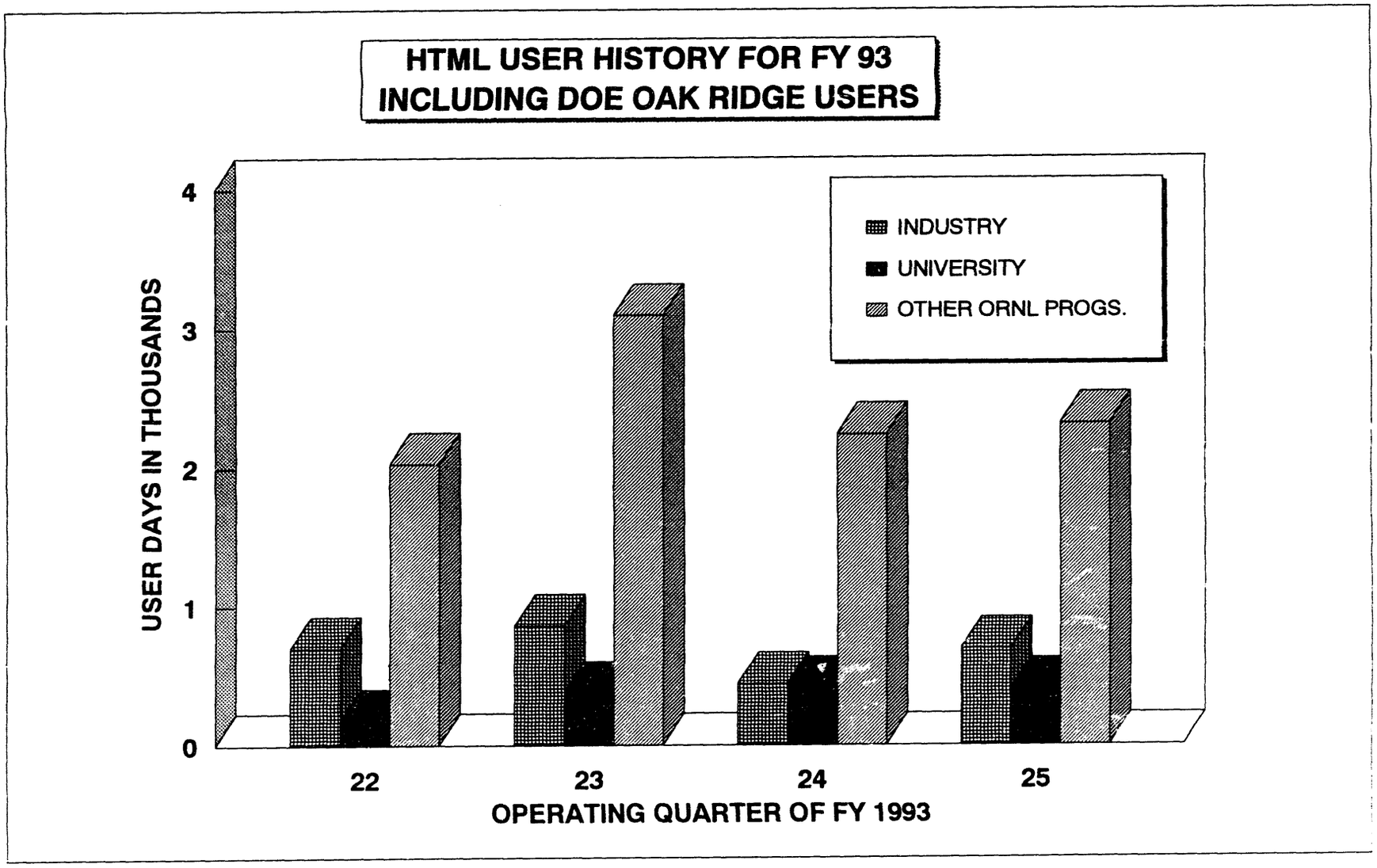

Fig. 1. Total user days in the HTML for FY 1993.

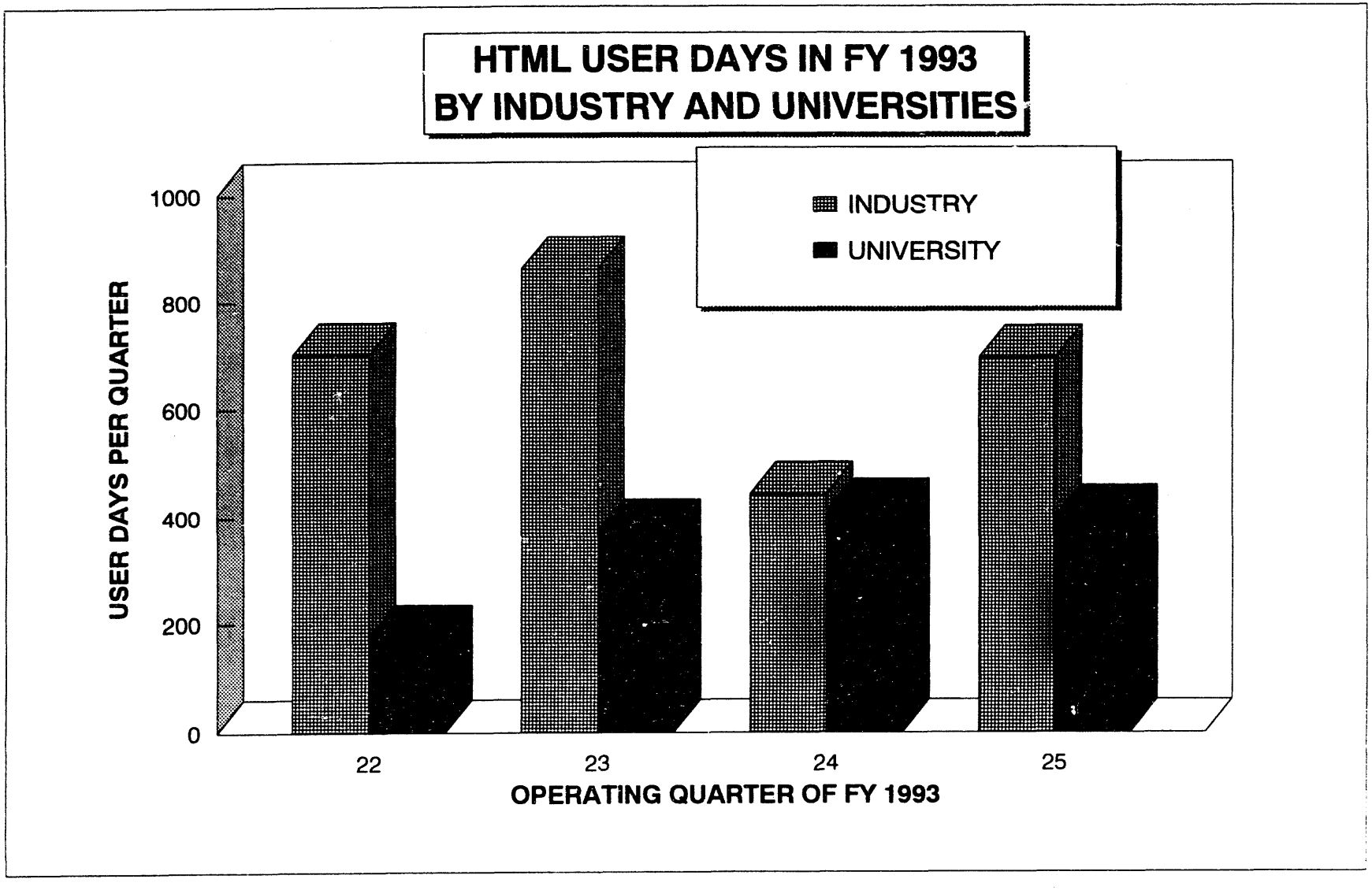

Fig. 2. Total industrial and university user days in the HTML for FY 1993.

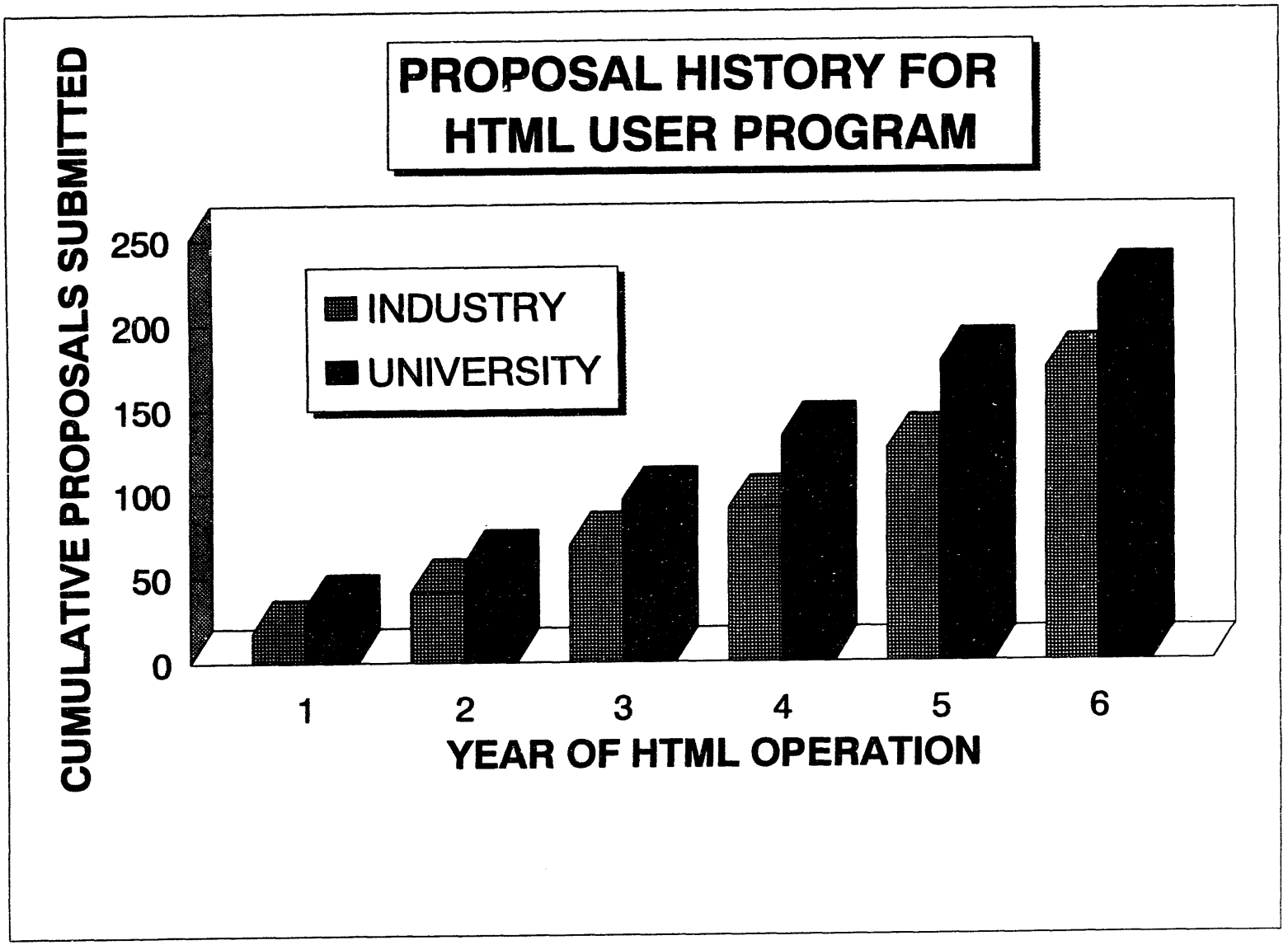

Fig. 3. Total number of research proposals submitted for consideration since the HTML User Program began in July 1987.

Table 2. Research proposals submitted July 1987 through September 1993

User type	Research proposals submitted		
	Jul 87-Sept 92	Oct 92-Sept 93	Total
Industry	126	47	173
University	177	44	221
Other Goverment	5	3	8
Total Nonproprietary	308	94	402
Proprietary	23	3	26
Grand Totals	331	97	428

3. MAJOR ACCOMPLISHMENTS IN FY 1993

A listing of many of the major accomplishments in the HTML User Program during this reporting period is given here.

- The first scanning thermal conductivity microscope in existence was installed in the Physical Properties User Center (PPUC). This microscope can simultaneously obtain and display a topographic and thermal conductivity image of the surface of a test specimen with a resolution of < 1 μm. Tests have demonstrated the microscope's ability to thermally resolve 2-μm pads of silicon oxide (thermal conductivity, K = 1.4 W/m K) spaced 1 μm apart on a silicon substrate (K = 148 W/m K).

- Capability to determine the thermal diffusivity of coatings has been developed by the PPUC staff. This new capability will greatly enhance the HTML's ability to study the effects of processing conditions, thermal history, and microstructure on the thermal conductivity of high performance thermal barrier coatings. The software was tested using a Rene 142 specimen with a plasma spray zirconia coating and a free-standing plasma spray zirconia coating supplied by General Electric (GE)-Aircraft Engines.

- An initial study was conducted to determine the cylindricity and circular trueness of a cylinder liner from an 8V-92 production engine of the Detroit Diesel Corporation. Initial results indicated approximately 15 mm of form error near the top of the liner and < 5 mm error at a depth of 5 cm from the top of the liner.

- At the HTML user group meeting, personnel from Coors Electronic Packaging in Chattanooga, Tennessee, made a presentation describing how work performed in the Materials Analysis User Center (MAUC) has been instrumental in improving solder seals and connections on electronic packages manufactured by Coors.

- Research being conducted by personnel from Florida Tile Industries involves utilizing electron microscopes and other instruments in the MAUC to determine causes of defects in the tile body and glaze (localized discolorations and surface irregularities) in tiles produced in the Lawrenceburg, Kentucky, production facility. Minimization of these defects will result in improved productivity and competitiveness in the international tile market.

- Work has been completed on a preliminary study involving the use of the nanoindenter as a diagnostic tool in the investigation of component failure.

- A workshop on measurement of residual stress by X-ray and neutron diffraction methods provided over 30 industrial, academic, and laboratory users and prospective users an opportunity to gain an in-depth understanding of micromechanics, measurement methods, and data analysis. The principal instructor was Dr. I. C. Noyan from IBM's T. J. Watson Research Center. Other contributors to the workshop were from the University of Denver, the University of Missouri, Los Alamos National Laboratory, and Oak Ridge National Laboratory (ORNL).

- Caterpillar, Inc., in collaboration with the Residual Stress User Center (RSUC) staff, has determined the diffraction elastic constants of partially stabilized zirconia (PSZ) deposited by plasma spray techniques to determine if the stabilizers and/or the thermal spray method lead to differences in elastic properties. Neutron diffraction techniques are being tested as a possible faster, more accurate measurement method.

- The RSUC facilities have been used in support of a Naval project to characterize the residual stresses that resulted from a series of grinding severities of aluminum oxide. The residual stresses typically decreased as the grit size decreased. Stresses in typical flexure bars used for flexure strength testing were observed to be lower than the corresponding stresses in large plates ground in an identical manner.

- Thermal conductivity of thermal barrier coatings (TBCs) as a function of processing and thermal aging was studied by GE-Aircraft Engines. The TBCs are being developed to increase the efficiency and life of turbine engines.

- Special procedures required to calibrate the depth-area relationship for the nanoindenter diamond probe were applied and verified. The verification process involved the use of the atomic force microscope (AFM) to directly measure the indent geometry. The resulting calibration curve relating depth to area is currently being applied to the calculation of hardness and elastic modulus of superconducting materials.

- The 95th Annual Meeting of the American Ceramic Society (ACeRS) was held in Cincinnati, Ohio, on April 18-22, 1993. The registration for this meeting was in excess

Dr. L. F. (Larry) Allard	Hitachi HF-2000 field emission gun-transmision electron microscope (FEG-TEM)
Mr. D. N. (Dave) Braski	PHI 660 scanning Auger microscope (SAM) and Topometrix scanning probe microscope (SPM)
Ms. D. W. (Dorothy) Coffey	Hitachi S-800 field emission-scanning electron microscope (FE-SEM) and S-4500 FE-SEM
Dr. K. L. (Karren) More	JEOL 4000EX high-resolution transmission electron microscope (HRTEM) and 2000FX analytical electron microscope (AEM)
Ms. R. (Rebecca) Shupe	Group Administrative Support

An important new instrument is being added to the suite of User Center instruments. An order was placed with Hitachi for the S-4500 SEM, which will begin beneficial operation in January 1994. This instrument combines very high spatial resolution at low accelerating voltage with the ability to observe a large portion of specimens with diameters of up to .15 cm. Thus, it will be ideal for the study of fine surface detail on large ceramic specimens such as high-temperature tensile fatigue-tested Si_3N_4 specimens. We were able to use the existing vacuum generator (VG) electron spectroscopy for chemical analysis (ESCA)/secondary ion mass spectrometry (SIMS) in trade for the new SEM. The Chemistry Department of Ohio State University purchased the VG with Hitachi acting as broker. The VG instrument has many capabilities but was difficult to operate and had proven not suitable for a user environment.

The Hitachi HF-2000 200-kV FEG-TEM has completed 1-1/2 years of beneficial operation. This instrument added three major new capabilities. On specimens having ideal geometry, it provides the highest lateral resolution presently attainable for X-ray elemental analysis. Elemental composition of regions as small as 1 nm can be determined, thus greatly enhancing our abilities to analyze ceramic grain boundary compositions. This capability has been used to determine conclusively the presence of yttrium in grain boundaries of the latest production Si_3N_4 ceramic (NT164) manufactured by Norton. Several other Si_3N_4-based ceramics have been analyzed in a similar manner, including the intermediate-temperature Norton NT-451 material and a Russian silicon nitride cutting tool material.

AlliedSignal GS-44 and A-series Si_3N_4 are presently under study as a part of another HTML User Project. The second capability introduced on the HF-2000, and soon to follow on the JEOL 4000EX HRTEM, is digital imaging. A retractable 1024 by 1024 charge-coupled device (CCD) slow-scan camera system has been retrofit to run in conjunction with the standard TV camera system on the instrument. This CCD camera effectively supplants film as the recording medium on the microscope and gives an enormous advantage to the throughput of data and the quality of the result available during an operating session. In fact, we have not had film in the microscope since April 1993, and all images are now acquired and stored directly on optical disks. A third unique capability offered by the HF-2000 is electron holography, made possible by the coherent beam from the FEG. Electron holograms preserve image phase information (lost in conventional TEM). The phase image

is very sensitive to changes in specimen thickness, composition, or degree of strain, and so can be used to determine to a high degree of accuracy the morphology of small particles, or the nature of interfaces. For example, we have been able to definitively conclude that 1-nm-diam palladium crystals, produced in a study of model combustion catalyst materials, were single crystals which contained voids in the crystal center. Also, utilizing the additional phase information, lens aberration corrections can be made that result in greatly improved resolution.

The second year of a 3-year Director's Fund initiative to develop electron holography has been completed, and we are now routinely taking high-resolution electron holograms on the HF-2000. Dr. E. Voelkl, an international leader in electron holography, has started his second year as a postdoctoral fellow on the holography project. He has developed numerous subprograms that run efficiently within the Macintosh software used to run the digital camera system. These programs allow us to perform all the image reconstructions possible with holography.

The first Faculty Fellow in this Center from the HTML Fellowship Program, Professor A. K. Datye of the University of New Mexico in Albuquerque, performed research during the summer. His research centered on characterization of the microstructure of a number of model catalyst and commercial catalyst materials. Palladium nanocrystals on the order of 10 nm in diameter on microspheres of SiO_2 were shown by electron holography on the HF-2000 to contain voids in the center of the nanocrystal. The results of this work have been submitted for publication. (A more detailed description of this project is given in Sec. 6.)

Several new user research projects were initiated this year. High resolution electron microscopy, combined with SEM; high-spatial-resolution energy-dispersive spectroscopy (EDS) on the HF-2000; and low-glancing-angle X-ray diffraction (XRD) techniques have been utilized to identify possible contaminants on the surfaces of alumina-insulating rings used in chemical-vapor deposition (CVD) furnaces (Coors Technical Ceramics). The characterization of various interface coatings in CFCCs was accomplished using the high- resolution capabilities of the JEOL 4000EX. For example, it was shown that boron-doped carbon interface layers, which are beneficial for enhanced oxidation resistance of the composite, have a highly aligned, turbostratic carbon structure as compared to the partially graphitized carbon coating commonly used in CFCCs.

The AFM has contributed to a number of important projects over the past year. Instrumentally, the microscope has been upgraded with the addition of a "non-contact" module that should be useful for imaging softer materials such as polymers or materials which are covered with bothersome contamination layers.

The work in characterizing the ceramic coatings on advanced piston rings (Cummins Piston Ring Division) has aided in the initiation of a Cooperative Research and Development Agreement (CRADA) between Martin Marietta Energy Systems, Inc. (MMES) and Cummins. Analyses of the surface roughness of anodized aluminum components (Rockwell International) have shown why O-ring life is being shortened by contacting these surfaces. In work with universities, a tin oxide/silicon oxide interface was examined (Alfred University) and thin molybdenum films were analyzed (University of Denver). For other government

laboratories, the pore sizes of polycarbonate filters were determined (Department of the Navy) and the surface topography of the inside of glass capillary tubes studied (Y-12 Plant).

The auger electron spectroscopy (AES) instrumentation was upgraded this past year with new computer capability and operational software. An important industrial collaboration was completed with Dr. Suzanne Raebel (Cummins Engine Company), who spent 3 months as an Industrial Fellow in the HTML Fellowship Program. She used AES to analyze the surfaces of numerous diesel engine components and bearing materials (see more complete write up in Sec. 6).

Researchers from Coors Electronic Packaging made numerous research visits to HTML to relate the surface composition of electronic packages to the conditions used in processing. Work with universities includes the identification of very small SiC islands deposited on silicon wafers by CVD techniques (Stevens Institute of Technology), the identification of elements present after the laser treatment of aluminum oxide wafers (University of Tennessee), the in situ fracture and analysis of Nicalon fibers (Rensselaer Polytechnic Institute), and the analyses of ceramic films produced by the sol-gel process (University of Arizona). In-house projects include the identification of contaminants on HFIR fule element protection tubes and an extensive investigation of the whisker/matrix interfaces in SiC whisker-reinforced/Al_2O_3 matrix composites.

Coors Electronic Packaging Center: MAUC

Project Title: **"AES Analysis of Electronic Packaging Device Surfaces,"** *W. K. Baxter*

These studies, using auger electron spectroscopy, focus on the use of barrier layers to retard or eliminate the diffusion of nickel through gold components. The analysis of component surfaces with AES has also been useful in identifying the source of problems that developed during processing. This ability to identify a particular problem led to quick solutions that eliminated costly production delays or shutdowns.

Cummins Engine Company Center: MAUC

Project Title: **"AES and XPS Analyses of Diesel Engine Components,"** *S. Raebel*

AES and X-ray photoelectron spectroscopy (XPS) were used to determine the surface chemistry of a number of diesel engine components including fuel system parts, camshafts, cylinder liners, and valve stems. The diffusion of indium through connecting rod bearings with varying degrees of wear was also investigated.

University of Tennessee Center: MAUC

Project Title: **"Laser-Enhanced Adhesion of Metallic Films to Ceramic Substrates,"** *A. J. Pedraza*

AES and XPS were used to help identify mechanisms that cause the enhanced adhesion of plated films on laser-irradiated alumina substrates. One of the possible mechanisms is the destruction of Al-O bonds in the surface layers to form metallic

or "free" Al atoms, whose presence apparently aids in the adhesion of subsequently-plated metallic films.

AlliedSignal, Inc. Center: MAUC

Project Title: "Microstructural Analysis of Si_3N_4 Ceramics Using Field Emission Transmission Electron Microscopy," *J. P. Pollinger*

GS-44 silicon nitride (baseline and microwave annealed) was analyzed using the FEG-TEM in July 1993. The goal was to determine the difference between the grain boundary phases of the two materials (the microwave-annealed material shows an order-of-magnitude increase in creep resistance). This work is being coordinated with the CRADA effort that processed the materials (Microwave Annealing with personnel of the Ceramic Science and Technology Section of the Metals and Ceramics Division).

The second task of the effort will be to evaluate the microstructures of creep-tested sintered AS800 and hot-isostatically pressed (HIPed) HT-05 Si_3N_4 materials. These materials should be tested in October 1993 on another recently initiated HTML User Program in the Mechanical Properties User Center (MPUC). Specimens are expected to be provided in November 1993 for analysis.

Carborundum Company Center: MAUC

Project Title: "Characterization of Vapor-Liquid-Solid Silicon Carbide Fibrils," *W. E. Hollar, Jr.*

SEM and TEM techniques have been used to evaluate the ultrastructure of (vapor-liquid solid (VLS) SiC fibrils produced under different synthesis and postprocessing conditions. Large-diameter fibrils were evaluated by TEM using a procedure in which fibrils were consolidated in chemical vapor infiltration (CVI) SiC followed by ion milling to produce electron-transparent thin sections. This technique has been used to study surface morphology and crystallography and to characterize the nature of internal defect structures.

University of Arizona Center: MAUC

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

The research performed at the HTML during the past fiscal year was focused on light element analysis of sol-gel derived borosilicate glass (BSG) films. Surface analysis and depth profiling of the films was done using the SAM. It is necessary to use SAM because SEM X-ray detectors (e.g., EDS) cannot detect boron or carbon. In this research, we are primarily concerned with the amount of boron retained in the films after different processing methods, but it is also important to examine carbon retention.

Boron is a volatile additive to silica glass, which makes the processing of BSG difficult. By using a wet chemical technique to make BSG, the high temperatures involved with melt processing can be avoided. However, boron is lost through the densification step, possibly

due to furnace firing. It was the hypothesis of this research that by laser firing the undense BSG films, more boron would be retained in the fired films due to the extremely rapid firing conditions. Through the use of SAM at HTML, we are examining this hypothesis and learning more about laser-fired, sol-gel-derived thin films.

Allison Engines Center: MAUC

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

During this period, the electron diffraction patterns obtained from the thermally cyclic exposed and the unexposed magnesium aluminosilicate (MAS) samples were identified to determine the nature of phases that develop during cycling.

Georgia Institute of Technology Center: MAUC

Project Title: **"The Composition of the Interfacial Layer of SiC Fiber-Reinforced Oxide-Ceramic Matrix Composites,"** *Y. Berta*

This research project consists of the investigation of interfacial phenomenology of SiC-fiber-reinforced brittle matrix composites. At our visit in April 1993, we obtained the light element analysis with the ultrathin window EDS system on the JEOL 2000FX. In response to the results, Mr. Shin is doing thermodynamic calculations to explain the presence of a carbon-rich interphase when the composites are fabricated under low-oxygen atmosphere, and the presence of silica- and carbon-rich interphase layers when the fabrication is under high-oxygen conditions.

Microstructural development related to thermal cycles in MAS regenerator (heat exchanger) material for gas turbine engines has been studied by AEM techniques.

Plans for further work include varying the composition of the matrix phase and observing the interphases formed under the new conditions.

Georgia Institute of Technology Center: MAUC

Project Title: **"Inorganic Dispersing Phase in Barium Ferrite Thin Films,"** *Y. Berta*

Nano-sized grains with a narrow size distribution are crucial for the application of barium ferrite thin films to storage media. Our prime goal in this research is to determine the role of the inorganic dispersing phase in the path of microstructural development. TEM investigation of heat-treated barium ferrite thin films was performed on four compositions of dispersing phase, and crystal sizes were measured to determine whether these phases had any effect on microstructure. Further study will concentrate on the dispersing phases and their role in the development of microstructure.

Coors Technical Ceramics Center: MAUC

Project Title: "An Investigation of the Differences in Alumina Rings Used in Semiconductor Plasma Etch Equipment," *J. Ghinazzi*

The surfaces of several alumina-insulating rings used in plasma etch equipment for semiconductor (Si wafer) processing have been examined using a variety of characterization techniques, including high-resolution SEM/EDS and cross-section TEM. Two specific types of alumina rings have been analyzed--a "bad" ring that was in the system for a relatively short period of time and may have caused surface deposits on the Si wafer, and a "good" ring that was in place within the system for an extended period of time and was not associated with any surface deposits.

Florida Tile Industries Center: MAUC

Project Title: "Improve Ceramic Tile Manufacturing Yield by the Identification of Major Defects," *M. S. Love*

Two types of defects, "dents" that are characterized by a depression in the tile glaze and "pinholes" that are characterized by a series of small holes grouped together, have been characterized in an attempt to not only identify the defect, but to identify the defect source. Excess scrap tile has been generated as a result of these particular defects, and improvements in tile manufacturing yields are expected as a result of the identification of the source of the defects. Thus far, both types of defects have been associated with excess aluminum and silicon within the tile body.

Allison Engines Center: MAUC

Project Title: "Evaluation of the Effect of Filler Metal Composition on Joint Microstructure for Titanium Alloys Brazed with Ti-Cu-Ni Materials," *J. Chang*

Commerically available brazing alloys derived from the Ti-Cu-Ni ternary system provide high-integrity braze joints for advanced titanium alloys. It has been found that subtle changes in filler metal composition can produce a wide variation in some mechanical properties. Three different brazing compositions have been characterized using AEM to determine any microstructural differences.

GTE Laboratories, Inc. Center: MAUC

Project Title: "Characterization of Interfaces in InAs/InGaAs Optoelectronic Multilayers," *C. M. Sung*

Electron holography and high-resolution electron microscopy were used to determine details of the atomic structure of interfaces in InAs/InGaAs multilayers. The advantages of obtaining pure-phase, perfectly energy filtered images of the multilayers were shown.

University of New Mexico Center: MAUC

Project Title: **"Microstructural Characterization of Catalyst Materials,"** *A. K. Datye*

Catalyst systems (Pd/SiO_2, Pt/Al_2O_3, and Rh/TiO_2) were studied by HTTEM, high-resolution SEM, AEM, and electron holography. Palladium nanocrystals on SiO_2 microspheres were shown to be hollow, and the central voids were observed to have crystallographic facets. The size and dispersion of heavy element particles on oxide substrates were characterized and related to processing treatments.

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

The MPUC is dedicated to the study of the mechanical performance of high-temperature materials. Dr. M. K. Ferber is the Group Leader; other staff and their areas of expertise are as follows:

Dr. A. A. (Andy) Wereszczak - characterization of the tensile creep/fatigue behavior of structural ceramics, fracture-toughness testing, and evaluation of interfacial properties in fiber-reinforced ceramic composites;

Dr. E. (Edgar) Lara-Curzio - characterization of macro- and micro-mechanical behavior of ceramic composites, fiber testing, and modeling of the constitutive equations for mechanical performance in ceramic composites;

Ms. L. (Laura) Riester - indentation and flexure testing;

Mr. T. P. (Tim) Kirkland - measurement of mechanical properties of structural materials; and

Ms. D. Green - Group Administrative Support.

A major thrust of the MPUC is to examine the influence of temperature, time, and applied stress level upon properties such as strength, toughness, fatigue, and creep resistance of advanced materials. 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 ten high-temperature tensile testing load frames (one with environmental capability), a fiber test machine, a composites test machine, and servo-hydraulic universal test machine (UTM) equipped with tension/compression grips; (3) a general-purpose testing lab comprising two UTMs; (4) a mechanical properties microprobe (MPM) [Nanoindenter]; and (5) an Interfacial Test Facility (ITF). Currently, one of the UTMs is equipped with a ceramic retort so that the high-temperature mechanical properties can be evaluated in inert environments or in vacuum. Detailed descriptions of flexure, tension, and indentation studies conducted in FY 1993 using the aforementioned facilities are provided.

4.2.1 Flexure Testing

During FY 1993, extensive flexure (and c-ring) testing was conducted using the FTF, a UTM equipped with a high-temperature furnace (designated as UTM-A), and a UTM equipped with a high-temperature furnace and ceramic retort for environmental testing (designated as UTM-E). The FTF is dedicated to high-temperature fatigue studies of structural ceramic materials and consists of six test frames, each having the capability of loading three flexure samples. For a given load frame, the specimen loading can be specified as a function of time. This feature permits the user to implement a number of standard fatigue tests including (1) static fatigue (time to failure measured as a function of static stress), (2) dynamic fatigue (fracture stress measured as a function of loading rate), and (3) cyclic fatigue (cycles to failure measured as a function of cyclic stress). Fast-fracture testing is also possible.

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

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

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

University of Illinois Center: MPUC

Project Title: "High-Temperature Mechanical Properties of Ceramic and Ceramic Composites," *J. F. Stubbins*

The object of the study is to examine the crack propagation behavior in ceramic materials. Three types of ceramic materials are being studied in the HTML. The first and second materials are both monolithic aluminas with either fine- (material A) or coarse-grain (material B) structure. The third material is SiC_f/SiC fiber-reinforced composite (material C). The selected materials exhibit some of the common crack-wake-induced toughening mechanisms at room temperature. Thus, the study is oriented toward studying these mechanisms and how they operate at high temperature. Four types of activities are being performed at the HTML: (1) fracture toughness measurements at room and high temperatures (flexure, 3-PT); (2) static fatigue measurements at room and high temperatures (flexure, 3-PT); (3) dynamic fatigue measurements at room and high temperatures (flexure, 3-PT); and (4) SEM.

SiC_f/SiC: Fracture-toughness measurements clearly showed a marked decrease in crack resistance at high temperatures. SEM showed that oxidation results in strong bonding between fiber and matrix and reduces the amount of fiber pullout and, hence, the crack propagation resistance. Static fatigue measurements also show that oxidation is the major phenomenon that reduces crack propagation resistance.

Alumina: Fracture-toughness measurements of both aluminas showed a rising crack propagation resistance with crack length at 25, 1000, and 1200°C, but a decrease in the resistance with increasing temperature was observed. Dynamic fatigue measurements revealed clear differences in the behavior of materials A and B. At 25°C, material A exhibits less dynamic fatigue sensitivity than material B. At 1000°C, material A has no dynamic fatigue effect, and material B exhibits lesser dynamic fatigue than at 25°C. These differences in behavior are assumed to arise from the difference in grain sizes and, hence, different crack-wake interactions and creep effects.

AlliedSignal, Inc. Center: MPUC

Project Title: "Cyclic Fatigue Properties of Two Sintered Silicon Nitride Materials," *R. G. Rateick*

This study, which began in FY 1992, focused on the evaluation of the fatigue properties of a silicon nitride (AS-44) ceramic at 25 and 950°C. The effect of R ratio (ratio of minimum to maximum stress) was also investigated. Dynamic fatigue testing (flexure strength measured as a function of stressing rate) was used to access the extent of static stress-rupture effects in this material. The experimental data revealed the existence of a stress-rupture process at room temperature that was attributed to environmentally assisted slow crack growth (SCG). This material was also susceptible to cyclic fatigue, as evidenced by a significant increase in fatigue life with increasing R ratio. The similarity of the crack growth exponents for the static and cyclic fatigue data indicated that SCG was the underlying mechanism. These results will ultimately be used in the development of a design strategy

Virginia Polytechnic Institute and State University Center: MPUC

Project Title: "Toughness of CMZP and CMZP Composites," *D. Hirschfeld*

In this study, the multiple-indent technique was used to measure the fracture toughness of a hot-pressed calcium-magnesium-zirconium phosphate (CMZP), a low-expansion ceramic for heat engine applications. Both unreinforced and SiC whisker-reinforced CMZP were tested by first introducing three indents in the polished face of each specimen. The initial crack lengths were then measured using a microscope equipped with a filar eyepiece. All specimens were then fractured in four-point bending. The lengths of the surviving cracks were measured and used along with the fracture load to calculate fracture toughness.

4.2.2 Tensile Testing

Proper tensile strength measurements of 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 mesurement, such that only 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.

Ten electro-mechanical 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 that 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, capacitance extensometers that have resolutions of ~0.1 μm at room temperature and ~0.5 μm at 1500°C. A Keithley 500 DAS and IBM-ompatible computer are used to monitor or control up to four test stations simultaneously.

A servo-hydraulic test machine (Instron 1332 with 8500 series electronics) is equipped with the tension-compression grip system. A unique feature of the servo-hydraulic 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) interface. 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 studying primarily cylindrical, button-head specimens. Two additional electro-mechanical tensile test machines (Instron Model 1380) in the TTF provide for the evaluation of the tensile mechanical properties of fibers and flat composite specimens. Fiber testing is achieved through the use of a

pneumatically actuated, kinematic fiber grip system. The water-cooled, fiber grips are equipped with flat, titanium grip faces between which the fiber is squeezed without slippage or grip-related damage. The gripping force is adjustable through changes in the applied pneumatic pressure. Fiber gage length can be varied from 25 to 200 mm for room-temperature testing. Gage lengths of 155 to 200 mm are possible for high-temperature testing using a resistance-heated furnace capable of temperatures up to 1400°C for sustained testing in ambient air.

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

During FY 1993, 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 work was that when failure was controlled by creep damage generation and accumulation, the fatigue life was uniquely determined by the steady-state creep rate (i.e., Monkman-Grant behavior). A second objective was to verify the expected improvements in creep and fatigue resistance of a HIPed silicon nitride that resulted from processing modifications to the intergranular phase. Specific examples of these research activities are provided below.

Saint Gobain-Norton Industrial Ceramics Center: MPUC

Project Title: "Creep Behavior of NC5102," *R. Yeckley*

Static tensile creep studies were conducted in ambient air at 1260 and 1370°C on a HIPed silicon nitride (NCX-5102) subjected to one of two different proprietary annealing schedules. Four of eight tensile specimens were subjected to a post-HIP/pre-machining anneal, while all eight were subjected to a post-machining anneal procedure. The post-HIP anneal did not significantly improve the creep resistance. High-temperature oxidation was responsible for the creation of an advancing amorphous grain boundary front (whose morphology and extent were investigated using XRD) in all the specimens. The rate of its advance appeared to be independent of the annealing treatment. The combination of tensile stress and oxidation ultimately catalyzed specimen failure by producing a stress-oxidation damage zone whose extent coincided with the advance of the amorphous grain boundary front. Their mutual growth during the static creep tests ultimately led to stress-induced failure.

AlliedSignal, Inc. Center: MPUC

Project Title: "Evaluation of Tensile Properties of Two Silicon Nitrides," *H. Fang*

In this project, the creep/fatigue behavior of a HIPed silicon nitride (Norton NT154) was evaluated at temperatures of 982, 1149, 1204, 1260, 1315, 1371, and 1400°C. In the range 1204 to 1400°C, creep deformation was reliably measured using high-temperature contact probe extensometry. The stress and temperature sensitivities of the secondary (or minimum) creep rate were estimated using a global regression procedure. This approach provided an assessment of the scatter typical of this class of materials as well as the identification of critical transition temperatures associated with a change in the failure mechanism (as reflected by a change in the stress dependency).

The resulting data were used to verify models for predicting both primary and secondary creep rates and the stress-rupture life. The parameters used for relating failure time to stress at a given temperature were based on those developed previously for metals. In this work, the Dorn parameter was used for the modeling of stress rupture because of its closeness in formulation to the creep rate model. However, it was further modified to accommodate an apparent change in mechanism observed both in the creep rate and stress-rupture behavior of NT154. The applicability of a modified Monkman-Grant expression for describing the stress rupture life was also examined and found to be inappropriate since the rupture life was not uniquely dependent upon creep rate. A modification to the Monkman-Grant expression was necessary to accommodate an additional temperature or stress dependency. In effect, the stratification of the Monkman-Grant curves could be explained on the basic crack growth model. These phenomenological models are expected to be applicable to other silicon nitrides of similar class or chemical composition.

Carborundum Company Center: MPUC

Project Title: "High-Temperature SiC Development Program," *K-Y Chia*

In this study, which was initiated in FY 1991, the mechanical properties of a developmental high-strength and high-toughness SiC, Generation I (SX-G1), were evaluated. The mechanical properties determined included flexural strength, tensile strength, and fracture toughness at room and elevated temperatures. Stress rupture, dynamic fatigue, and creep at elevated temperatures were also evaluated. The strength-limiting factors were identified at room and elevated temperatures. In terms of representative results, a mean flexural strength value of 780 MPa was measured at room temperature. The strength decreased at elevated temperatures, primarily due to a reduction of K_{IC}. The SX-GI material appeared to be susceptible to SCG at 1370°C. Finally, the SX-GI material possessed good creep resistance, despite an oxide second phase in the microstructure.

In summary, the results showed that SiC materials sintered with the addition of yttrium and aluminum compounds can achieve the high level of mechanical properties required for their use in heat engine applications. However, the reaction of the secondary phase with the SiC led to the formation of reaction-phase clusters or pools which increased the variability of the mechanical properties. If this variability can be eliminated or controlled, the relatively

low cost of the raw materials and processing of SX-SiC should greatly facilitate its commercialization.

Applied Materials Center: MPUC

Project Title: "Characterization of the Long-Term Creep Behavior of Aluminum Alloys at High Temperature as a Function of Anodization Thickness," *S. Shamouilian*

The goal of this program is to extend the creep data base for an aluminum alloy that is currently used as a susceptor in a CVD facility. During FY 1993, one tensile test machine was modified to accommodate the aluminum tensile specimen. The major modification involved the fabrication of new high-temperature grips so that water cooling was not required. This, in turn, greatly reduced the temperature gradients in the specimen. Additional modifications, which are planned for FY 1994, involve the replacement of the existing two-zone slot furnace with a clamshell furnace having better temperature stability at lower temperatures.

4.2.3 MPM (Nanoindenter)

Indentation Testing with the Nanoindenter and Interfacial Test System. The nanoindenter is a special microhardness instrument 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 instruments, 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 directly. 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.

The characterization of the micro-mechanical behavior of CFCCs often relies on the use of MPM-based indentation techniques for the measurement of interfacial properties (including debond stress, interfacial shear stress, and residual stress). These techniques typically involve the generation of a load-displacement curve associated with sliding of individual fibers. The corresponding instrumentation is currently limited by its inability to test fibers exhibiting a wide range of diameters (5 to 100 μm). This limitation arises from (1) insufficient load capability, (2) low spatial resolution of stages used to position the fiber with respect to

the indenter due to the air objective optical microscope in the system, and (3) insufficient resolution of the transducers used to measure fiber displacement.

The development of the interfacial test system (ITS) represents a major effort at addressing the aforementioned limitations. The ITS is capable of measuring the load and displacement associated with both fiber push-in and push-through tests. Positioning of the fibers is achieved with an x-y table capable of precise movement to better than 0.5 μm. An optical microscope is used to locate fibers for indentation loading. A large-screen monitor connected to the microscope camera is used for easy viewing of the fibers. The indenter and load cell are mounted on a separate bracket located along the right side of the stages. This bracket is designed such that load cells covering a range of load capabilities can be easily accommodated. The indenter holder also accommodates several WC and diamond flat-bottomed probes as well as a standard Vickers indenter. The specimen is loaded into the indenter using a z stage, which is mounted on top of the x-y table. During testing, the displacement is measured to 0.1 μm using an encoder mounted on the shaft of the z-stage. The operation of the entire system, including data storage and analysis, is controlled by a microcomputer.

During FY 1993, the nanoindenter was used to evaluate the plastic and elastic properties of thin films, ion-implanted surfaces, and metal alloys. Both the nanoindenter and ITS were used to measure the interfacial properties of a variety of fiber-reinforced ceramic-ceramic composites. Examples of these research activities are provided below.

University of Illinois — Center: MPUC

Project Title: **"Investigation of Interfacial Shear Strengths of Fiber-Reinforced Boron Nitride Composites,"** *J. Economy*

Interfacial shear strength measurements were performed on a series of boron nitride composites fabricated with carbon and other inorganic fibers. These fibers included PAN T300, Pitch K1100X, Nicalon, c-Nicalon, Nextel 440, Sumica, and FP alumina. Both the nanoindenter and the ITS were used to perform push-in tests on at least ten fibers in each system. Fiber sliding, indicative of weak interfacial strengths, was observed for the carbon fiber specimens.

Additional investigations are planned for the boron nitride matrix composites. Interfacial testing will be performed on a series of fibers that have been coated with a series of carbon or boron nitride coatings. Wedge-shaped samples will also be prepared in order to complete push-out testing of these composite systems. Further mechanical testing will then be performed in order to correlate first matrix cracking of the systems.

American Superconductor Corporation — Center: MPUC

Project Title: **"Mechanical Properties of Oxide Superconductors,"** *G. N. Riley*

During FY 1993, much progress was made on characterizing the mechanical properties of oxide superconductors based on studies conducted in the HTML. Using the powder in tube (PIT) process, high-temperature superconductor (HTS) composite wires and precursors

were fabricated using oxide powders from the (Bi,Pb)SrCaCuO system. Metallographic techniques were used to prepare these high-Jc, textured samples so that specific orientations of the superconducting grains could be evaluated. Mechanical characterization was done using the nanoindenter in the HTML. Although the grain size of the superconducting phases is small, mechanical characterization of individual grains was performed. The resulting data represent the first measurement of the orientation-dependent, intrinsic mechanical properties of the BSCCO superconductors. In addition to this technologically important data, hysteretic behavior was observed during mechanical cycling. This interesting phenomena may lead to fundamental insight into the mechanisms that contribute to texture during the PIT process. Consequently, this research must continue so that (1) the hysteretic behavior can be better characterized and understood and (2) the relationship between microstructure and properties can be determined.

New Mexico Institute of Technology Center: MPUC

Project Title: "The Effect of Tin Dioxide Interlayer in Alumina/Glass Composites," *K. Chawla*

Oxide fiber/oxide matrix composites, such as alumina fiber/glass matrix, represent an important class of ceramic matrix composites because of their inherent stability in air at high temperatures. Alumina and glass, however, form a very strong chemical bond, which is incompatible with high-fracture toughness. In this study, an interface was introduced between the matrix and the fiber, in order to obtain energy dissipating processes such as interface debonding, crack deflection, and fiber pullout in this system. In initial studies, the role of tin dioxide as a barrier coating between alumina and glass was examined using fiber push-in measurements. An important finding of this work was that, although the SnO_2 coating provided the intended diffusion barrier and the thermal stress distribution was of the desirable kind, a neat and clean fiber pullout was absent because of the roughness of the PRD-16/SnO_2 interface. Some fiber/matrix debonding, crack deflection, and crack bridging were observed. The roughness-induced radial clamping stress was too large to allow fiber pullout. To reduce this radial clamping effect, a relatively smooth fiber, viz., Saphikon, a single-crystal alumina fiber, was examined in a subsequent study. As expected, SnO_2-coated Saphikon fiber/glass composite showed a much larger fiber pullout length than the coated PRD-166 fiber/glass composite. However, fiber push-through tests on both coated and uncoated versions of this composite were unsuccessful in inducing fiber sliding.

In more recent studies, a mullite fiber (Nextel 550), CVD coated with a BN coating, was incorporated in a mullite matrix. The ITS was used to measure the interfacial properties. Using a progressive debonding interface model, the average interfacial shear stress was determined to be 28 MPa, while the range was 1.3 to 64 MPa. This large variation was attributed to variations in the thickness of the BN coating after processing of the composite. Fracture surfaces of these BN-coated composites obtained from flexure testing showed fiber pullout.

Dow Corning Corporation — Center: MPUC

Project Title: "Characterization of alpha-SiC:H Films Using Micro-Indentation Techniques," *M. Loboda*

Amorphous hydrogenated silicon carbide (a-SiC:H) films were grown from two different precursor gases, a methane/silane mixture and silacyclobutane (SiC_3H_8). Plasma-enhanced CVD was used to deposit α-SiC:H films at temperatures of 175 and 600° C. The α-SiC:H films were characterized using the nanoindenter and by scratch testing. Two approaches were used to calculate the hardness and elastic modulus, H and E, respectively. The first approach involved the use of an averaging technique in which H and E were calculated from the slopes of compliance versus inverse plastic depth and load versus area curves, respectively. In the second approach, H and E values, determined from a standard material indented under identical conditions, were used to normalize the experimental data. Both approaches gave similar trends. Measurements on silicon show good agreement with that previously reported. The calculated a-SiC:H film hardness was shown to depend on the precursor gas at 175° C, while the film elastic modulus varied with precursor gas, composition and density, as determined by the plasma source deposition power. The micro-indentation and scratch test data showed similar correlations to plasma source power and film structure and composition. A strong correlation was observed between the relative coefficient of friction of the film surface and the elastic modulus. Although small indentations (<100 nm) were performed, the data and estimation of the spatial extent of the plastic stress zone under the indenter suggest that for the modulus-to-hardness ratio range of the films examined ($5<E/H<9$), the indentation measurements were not influenced by the substrate.

University of Alabama — Center: MPUC

Project Title: "Microstructural Characterization of Composite and Intermetallic Materials by the Double Ligament Tension Test," *J. Berry*

The nanoindenter was used to generate mechanical property data (primarily hardness) required for the finite element modeling of the double ligament tension (DLT) test. The purpose of the modeling effort was to develop the DLT test for application to materials such as metal matrix composites and intermetallics.

Johns Hopkins University — Center: MPUC

Project Title: "Nanoindentation Study of the Mechanical Properties of Granular Metal Thin Films," *R. Cammarata*

Nanoindenter techniques were used to investigate the mechanical properties of Ag-Al_2O_3 and Fe-SiO_2 granular metal films. A discontinuity in the rate of change of hardness as a function of metal volume fraction, p, was observed. The discontinuity occurred at the percolation threshold, p_c, of the metal and appeared to result from a change in the deformation mechanism. A large peak in the unloading compliance was observed near p_c in the Ag-Al_2O_3 films but was absent in the Fe-SiO_2 films. This compliance peak is believed to result from debonding at the metal-ceramic interface and subsequent interfacial sliding.

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

The PPUC is dedicated to measurement of physical properties as a function of temperature and correlation of the thermophysical properties with processing, microstructure, and performance. Current facilities include the following instruments. (The last two instruments were built by the staff to meet specific measurement needs where no commercial instruments exist).

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

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

Mr. W. D. (Wally) Porter	Thermal analysis
Dr. R. B. (Ralph) Dinwiddie	Thermal transport
Dr. S. (Steve) Beecher	Thermal transport
Ms. A. (Amanda) Abeel	Co-op student
Ms. J. (Joy) Kilroy	Group Administrative Support

Research projects performed in this center are summarized below.

McDonnell Douglas Aerospace Center: PPUC

Project Title: "Development of High-Thermal-Conductivity Carbon-Fiber Composites,"
D. A. Bowers

Thermal diffusivity data were obtained on 4 one-dimensional (1-D) carbon-carbon (C/C) composite materials this year as part of a plasma-facing material development program at McDonnell Douglas Aerospace. This fusion energy program's objective is to develop lower cost, high-thermal-conductivity carbon-fiber composites for high-heat-flux diverter surfaces in experimental fusion energy generating systems.

Measurements included samples from Hercules Aerospace; Applied Sciences, Inc.; and Tonen Energy International, Inc., of Japan. Room-temperature conductivities as high as 840 W/m-K (copper = 360 W/m-K) were measured, which compares with the pyrolytic graphite (PG) range of 700 to 1400 W/m-K. Carbon-fiber composites (CFCs) are expected to possess greater mechanical strengths than the inherently low-strength PG, thereby making fusion diverter designs that utilize CFC materials much more robust. Measurement of

elevated-temperature properties are currently being concluded and will also be compared with PG data.

General Electric Aircraft Engines Center: PPUC

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

The xenon flash thermal diffusivity system was used to study the effects of thermal aging on the thermal conductivity of PSZ TBCs. TBCs are currently used to increase component reliability in gas turbine engines. Two types of TBCs were investigated. The first type was fabricated by physical vapor deposition (PVD) and the second type by plasma spraying. The specimens were heat treated at various temperatures, up to 2200° F, for times ranging to 100 h. At room temperature, the PVD TBCs have a thermal conductivity 35% higher than the plasma-sprayed TBCs. However, the PVD TBCs were found to be less susceptible to increases in thermal conductivity due to thermal aging than the plasma-sprayed TBCs.

Virginia Polytechnic Institute and State University Center: PPUC, XRDUC

Project Title: "High-Temperature Crystallography and Phase Transitions of CMZP Ceramics," *W. M. Russ*

Differential Scanning Calorimetry: Hot-pressed $(Ca_{0.6}, Mg_{0.4})Zr_4(PO_4)_6$ [CMZP] that had been ground to a fine powder (-325 mesh) was analyzed utilizing the Stanton Redcroft DSC 1500S. A heating rate of 50° C/min was employed to achieve a maximum temperature of 1000° C. The furnace cooled to room temperature on its own. An Omnitherm software package was used for DSC operation and determination of heat flow with temperature.

Thermogravimetrical Analysis (TGA): The Stanton Redcroft STA was used to measure mass loss as a function of temperature. The specimen analyzed was a fine powder (-325 mesh) that had been milled from a hot-pressed CMZP specimen. A 20° C/min heating rate was utilized to reach a final temperature of 1000° C. Once again, an Omnitherm software package controlled the STA unit and calculated results. The mass change of the specimen was monitored as a function of temperature, and differential thermal analysis measurements produced insight as to the cause of the change in mass.

Results: None of the experiments performed were able to explain or correspond to the reversible transition observed for these new low thermal expansion ceramic materials in the thermal expansion plot as a function of temperature (change in slope of relative expansion versus temperature). The transition was probably due to an ordering of the Ca^{2+} and Mg^{2+} cations in the unit cell of the crystal structure.

Advanced Refractory Technologies, Inc. Center: PPUC

Project Title: "High-Temperature Thermal Conductivity of Aluminum Nitride," *T. Mroz*

Aluminum nitride has very high thermal conductivity while being an electrical insulator. Reduction of oxygen within the sample using secondary phases has a large effect on the thermal conductivity at room temperature. However, the effect of these secondary phases at elevated temperatures is of interest for new applications. The thermal diffusivity of pressureless sintered AlN samples prepared with varying yttria concentration was evaluated over the temperature range of 20 to 1200° C. As expected, the thermal diffusivity decreased with increasing temperature. It was found that the effectiveness of the sintering aid decreases significantly over the experimental range. Confirmation of the second-phase chemistry was made via XRD. High-temperature specific heat data were also obtained and used along with thermal diffusivity data to calculate thermal conductivity as a function of temperature.

Northwestern University Center: PPUC

Project Title: "Investigation of the Thermal Expansion and Heat Capacity of $(Mo,Cr)_2C$," *C. Knepfler and G. Olson*

Recent interest in improving the strength and toughness of ultra high-strength steels has led to an interest in the properties of molybdenum-chromium carbides and molybdenum-chromium-vanadium carbides of the M_2C type. There is currently little information on the thermodynamic properties of the M_2C carbide phase. Thermodynamic information of the steel phases can be used to predict the compositional dependence of the precipitation-driving forces and coarsening-rate constants. Measurements of the physical properties of the molybdenum-chromium and molybdenum-chromium-vanadium carbides were undertaken at the HTML in order to expand the thermodynamic data base of these M_2C carbides, which will allow the system effects of the M_2C carbides to be more completely modeled.

Bulk samples of M_2C (M = Mo,Cr,Fe,V) carbide were synthesized, and the specific heat capacity was determined using the Stanton Redcroft DSC 1500S. The specific heat of the carbides are used to provide information on the free energy of the carbide system.

The lattice thermal expansion behavior of the M_2C carbides was investigated using the Scintag HTXRD. The expansion behavior of the M_2C carbides is important for the understanding of the nucleation and strengthening behavior of these carbides within secondary hardening steels. This information enables the investigation of the coherent precipitation behavior of the carbides within the parent steel matrix, as well as the consideration of the effect of residual stresses on the observed strengthening.

North Carolina State University Center: PPUC, MAUC

Project Title: "Analyses of Ni_3Al, Ni_3Si Intermetallic Alloys," *C. C. Koch and N. Sukidi*

A series of alloying studies has been carried out in recent years at ORNL to optimize the mechanical behavior of Ni_3Si-base alloys. Several of these alloys show great promise

as potential elevated-temperature structural materials with excellent corrosion resistance. The mechanical behavior of these alloys is sensitive to chemistry, which determines phase relationships as well as affecting the environmental embrittlement observed. Fundamental understanding of the alloying/structure property relationships in these alloys is the thrust of the present study. Studies were conducted on Ni-19.4 at. % Si-3.2 at. % Nb and Ni-19.4 at. % Si-3.2 at. % V alloys with and without additions of 0.05 at. % B, using the Stanton Redcroft STA 1500S and the Stanton Redcroft DSC 1500S. Melting points and phase-transition temperatures for nine alloys were determined. The phase relationship/chemistry studies will help the understanding of the effects of alloying on ambient temperature ductility and hot fabricability.

University of Illinois Center: PPUC

Project Title: "Thermal Diffusivity of Carbon-Fiber-Reinforced Boron Nitride Composites," *J. Economy*

Room-temperature thermal diffusivity measurements were made on specimens of boron nitride matrix, unidirectional composites fabricated with K1100X carbon fibers. Measurements were made in the fiber direction.

This work has been submitted for oral presentation at the 18th Annual Conference on Composites and Advanced Ceramics, to be held on January 9-14, 1994.

Georgia Institute of Technology Center: PPUC

Project Title: "Thermal Conductivity of Al_2O_3/Glass Composites," *J. Cochran and R. Gonzalaz*

Synthesis of hollow ceramic spheres is being studied at Georgia Institute of Technology and the spheres are targeted for use as high-temperature insulation. Attempts have been made to reduce thermal conductivity of the hollow shells by incorporating a controlled-pore microstructure into the sphere walls. It is expected that a proper pore structure will effectively impede radiant heat transfer and result in a lower conductivity.

Thermal diffusivity measurements were made on pressed Al_2O_3/glass composites and showed that the introduction of an opacifying phase into the ceramic matrix results in a significant decrease in thermal conductivity. Results of this study are being included in the M.S. thesis of Mr. Gonzalez.

University of Texas/Arlington Center: PPUC, XRDUC

Project Title: "A Phase-Transformation Study in Barium Alumino Silicate (BAS)-Silicon Nitride System Using High-Temperature X-Ray, Thermal Gravimetric Analysis, and Differential Thermal Analysis," *P. B. Aswath and A. Bandyopaddhyay*

Seventy percent Si_3N_4 with 30% BAS and 40% Si_3N_4 with 60% BAS compositions were studied using DSC, STA, dilatometry, room-temperature X-ray diffraction (RTXRD), and HTXRD. Presintered powders of both the compositions, containing $BaCO_3$, Al_2O_3, SiO_2, and a mixture of alpha and beta-Si_3N_4 with predominantly alpha-Si_3N_4, were mixed in an

appropriate proportion and sintered for different sintering times. Studies were conducted at the HTML on both presintered powders and on sintered composites that were sintered for different sintering times. Thermal analysis was first conducted using STA, DSC, and dilatometry. For both DSC and STA, experiments were conducted from room temperature to 1450°C using alumina and platinum pans. It was observed that at high temperature, samples were reacting with Pt pans and resulting in some error in the experimental data. Dilatometry tests were conducted from room temperature to 1000°C at a heating rate of 3°C/min for ten samples with different compositions and different sintering times. XRD studies were conducted after thermal analysis. RTXRD experiments were conducted on both presintered and postsintered powders. HTXRD tests were done on both compositions with presintered and sintered powders. Tests were conducted at several temperatures between room temperature and 1600°C in an He environment and with a 2θ range of 10 to 45°.

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

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

There are two major instruments in the XRDUC:

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

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

Mr. O. B. (Burl) Cavin	Mr. R. (Rob) Simpson
Dr. T. R. (Tom) Watkins	Mr. N. (Nathan) McAdams

Mr. Cavin retired at the end of September 1993, and Dr. N. H. (Nick) Packan has joined the group effective October 1, 1993. Mr. McAdams and Mr. Simpson were co-op students from Virginia Polytechnic Institute and State University and Georgia Tech, respectively. Mr. McAdams conducted research under the direction of the Group Leader to expand the analysis capabilities of the User Center. His primary task was to develop Reitreld replacement methods and expertise within the group. From his studies, we can now obtain greater precision lattice parameters and phase analysis than previously possible. Applications of this technique to changes in structure as a function of temperature are continuing to be tested.

Ms. J. Kilroy provides administrative support for this group.

Research projects are summarized below.

University of Tennessee — Center: XRDUC, PPUC

Project Title: "Mechanical and Thermal Expansion Behavior of Sodium Zirconium Phosphate Structure-Type Materials," *J. E. Spruiell*

Room- and high-temperature XRD data were collected on some of the compositions in the barium zirconium phosphate silicate system, which belongs to the sodium zirconium phosphate structure-type materials. These materials are being studied as candidate low (near-zero)-thermal-expansion inserts for low-heat-rejection engine applications. RTXRD data were used to identify the phases present in the compositions, and unit-cell parameters, a and c, were determined using internal standards and least-squares cell refinement methods. HTXRD data were collected, and the axial thermal expansions were then determined. Subsequently, DSC studies were performed in the HTML to observe/confirm phase transitions in some of the compositions.

Virginia Polytechnic Institute and State University — Center: PPUC, XRDUC

Project Title: "High-Temperature Crystallography and Phase Transitions of CMZP Ceramics," *W. M. Russ*

RTXRD: The Scintag RTXRD was used for phase analysis and to set up a room-temperature reference for -325 mesh $(Ca_{0.6}, Mg_{0.4})Zr_4(PO_4)_6$ [CMZP] powder. The powder was obtained from a pulverized hot-pressed disk.

Elevated Temperature X-Ray Diffraction: The Scintag elevated-temperature XRD was used to determine changes in phases and lattice parameters up to 1200° C. One-hour scans (10 to 70° 2θ) were performed at room temperature, 200, 400, 450, 500, 550, 600, 800, 1000, and 1200° C on heating at 20° C/min. On cooling, 600° C, 400° C, and room-temperature data were collected and analyzed. The sample was held at each respective temperature for 2 min to allow for equilibration of the system prior to data collection. Pulverized CMZP powder (similar to above) and a 1-mm-thick hot-pressed CMZP slab were examined in a helium atmosphere.

University of Kentucky — Center: XRDUC

Project Title: "Crystal Structure Identification of Pt-Re-Al_2O_3 Catalysts Using HTXRD," *B. H. Davis and R. Srinivasan*

Four powder samples containing varying amounts of platinum and rhenium impregnated on a silica support were mounted on a single crystal substrate atop the Pt heater strip in the HTXRD furnace. The catalyst containing 0.6 wt % Pt/SiO_2 did not exhibit metallic platinum formation after reduction in H_2 at 500° C for 15 h as determined by HTXRD. The catalyst containing 1.2 wt % Pt/0.6 wt % Re/SiO_2, however, exhibited the metallic platinum phase after reduction at 500° C for 2 h. However, with increase in temperature to 750° C, the platinum peaks broaden and disappear, and Pt_3Si peaks appeared. This phenomenon was observed for all the other samples. To verify this phenomenon, a few milligrams of platinum powder

(-325 mesh) were mounted and heated in hydrogen. At 500° C, metallic platinum was formed and at 750° C, peaks corresponding to $Pt_{12}Si_5$ and Pt_3Si phases appeared. Hence, it has been verified that the platinum in the catalyst reacts with silicon in the silicon substrate on which the catalyst was mounted in the furnace attachment. Hence, a different kind of substrate needs to be used, and a future research visit is scheduled.

Clemson University Center: XRDUC

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

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

The experimental portion of this program is essentially complete. A presentation of the results was made at the Denver Conference on X-Ray Analysis Methods. Efforts are currently being focused upon writing of the final paper.

University of Cincinnati Center: XRDUC, MAUC

Project Title: "Characterization of Doped Titania," *S. E. Pratsinis*

HTXRD: The aim of the work was to study the phase evolution of alumina- and silica-doped titanium dioxide powders made via the gas-phase oxidation of the respective metal chlorides. The qualitative results obtained from the high-temperature diffraction study confirmed the room-temperature observations that the presence of alumina enhanced the formation of rutile titania, while the presence of silica retarded rutile titania formation.

High-temperature scans between 600 and 1000° C under flowing helium shed some light on the mechanism of phase transformation. In the case of silica-doped titania, the high-temperature scans revealed a progressive increase in rutile content and a corresponding decrease in anatase content as a function of time and temperature. This clearly confirms that the silica interferes with and hinders the phase transformation mechanism in titania. Only on increasing the temperature are the titania crystallites able to transform to the rutile form in the presence of silica, suggesting an increasing activation energy for the transformation which, in the absence of silica could occur at lower energy levels.

In the case of alumina-doped titania, the powders were primarily rutile initially while high-temperature measurements invesigated all rutile. At room temperature, the alumina dopant was amorphous, but heating during the high-temperature scans resulted in transformation into alpha-alumina. This was clearly observed in samples doped with greater than 20% alumina. Only qualitative X-ray analysis was done on the high-temperature unit as the LN_2-cooled detector was not operational at the time.

Simultaneously with the high-temperature work, room-temperature analyses were obtained for silica-doped titania and for titania made in the presence of sodium, potassium, and cesium chloride. Profile fitting and silicon internal standard were used for accurate determination of lattice parameters. For silica contents up to 5%, and for all the titania powders made in the presence of ionic additives, there was no increase in the anatase titania lattice parameters.

TEM: TEM in conjunction with EDS was used to study particle morphology and chemical composition of titania doped with alumina. The use of the HF-2000 enabled the mapping of chemical composition variation across the cross section of particles at resolutions as high as 10 nm.

At high alumina content (40%), two distinct morphologies were observed: large, round particles and small, polyhedral ones. The larger particles were between 100 and 200 nm in diameter, and the chemical composition varied from mainly Ti on the surface to predominantly Al in the interior. The smaller particles were more uniform in chemical composition, and the surface was richer in Al. These particles were better crystallized as evidenced from the distinct lattice fringes observed.

At lower alumina content (7%), the particles were polyhedral with round edges. A few of the larger particles were also observed. In all cases, the particle surface was richer in Al than the particle interior. For the small particles a large variation in the Ti/Al ratio was observed; in some, the Al content was as low as 2%, while in others, it was as high as 11%.

The results discussed are from three samples, and more samples need to be studied before drawing any conclusions. These results are very interesting, and the dopant-crystallinity-chemical composition relation will provide some insight into multicomponent particle formation once the analytical TEM results are combined with XPS and Auger data.

University of Tennessee Center: XRDUC

Project Title: "Determination of Pair Distribution Function for Silica Processed Using 2.45-GHz Radiation," *T. Meek and S. S. Park*

Experiments have continued in the study of mechanisms of microwave joining of ceramics by comparing these methods with conventional high-temperature furnace joining. The aim is to explore possible procedures for more economical and efficient joining of ceramics and ultimately to handle some ceramic materials otherwise difficult to join. Room-temperature X-ray powder diffraction studies were carried out comparing SiO_2 specimens fabricated using conventional means with samples fabricated using 2.45-GHz microwave radiation. Data sets were collected with Mo Kα radiation over the accessible 2θ range. Data processing and analysis are currently under way at the University of Tennessee.

Alfred University Center: XRDUC

Project Title: "Isothermal Melt Processing of the Ba-Y-Cu Superconductor," *J. Taylor and D. Carnahan*

High- and room-temperature XRD techniques were applied to better understand the development of phases in BYC213 superconductor material during sintering and annealing. Melt processing of BYC213 produces an oriented microstructure that has very high current-carrying capacity. The controlled-atmosphere, high-temperature experiments using the HTML's XRDUC facilities were aimed at delineating the appearance of phases and texture in the oriented surface layer. They were also used to evaluate parameters that affect the melt-processing procedure.

4.5 CERAMIC MACHINING USER CENTER (CMUC) - B. L. COX

The name of this user center will be changed in January from the Ceramic Specimen Preparation User Center (CSPUC) to better describe the broader role of this center which has been developed in the past year. The CMUC provides basic facilities for (1) investigation of grinding processes for high-performance ceramics, (2) design and fabrication of mechanical property test specimens, and (3) dimensional characterization of test specimens and ceramic and metal components including roughness, form, orientation, and subsurface structure. Specific equipment includes an instrumented CNC grinder, a grinder with open-loop control and; a CNC four-axis cylindrical grinder; a computer-controlled profilometer and non-contact laser topography system; a form-measuring instrument (cylindricity); a resonant ultrasound spectroscopy system; and a computer workstation with design, analysis, and programming software. Major focus of this center is on ceramic machining research using instrumented machine tools along with the dimensional measurement and surface characterization tools.

The group leader of the CMUC through December is Mr. B. L. Cox. Mr. V. T. (Tyler) Jenkins is responsible for the ceramic machining. Ms. D. (Dana) Green provides administrative support for this group.

During late FY 1992 and early FY 1993, a SiliconGraphics Crimson workstation was procured and installed in a specially designed computer room. The workstation has been linked to an ethernet network that provides easy access to other computers throughout the HTML. Software available on the workstation includes Pro/Engineer (solid modeling), COSMOS/M (finite element analysis), ICAM (numerical control programming), CimStation (coordinate measuring machine programming and simulation), and CARES (probability life prediction for brittle materials). The CARES software and a direct interface to the COSMOS/M software were developed by the NASA-Lewis Research Center and installed on the workstation by Ms. Lynn Powers, of Cleveland State University working with NASA-Lewis Research Center.

The Harig CNC grinder was instrumented by the Institute of Advanced Manufacturing Sciences, Inc. The instrumentation includes force, vibration, horsepower, and acoustic emission, all recorded in real time and referenced by an optical encoder pulse-indicating

wheel rotation. The slicer/grinder is used for grinding studies on new ceramic materials and for grinding wheel evaluations.

Over 300 flexure specimens and over 100 cylindrical compression specimens were prepared for a project with the Naval Command, Control, and Ocean Surveillance Center. The materials involved were AL-600 alumina oxide from Wesgo, Inc., and AD-94 alumina from Coors Technical Ceramics. A presentation on the results was given at the International Grinding Conference on Ceramic Materials held at the National Institute of Standards and Technology (NIST) in July 1993.

New instruments being added to the CMUC include a form-measuring instrument to provide rapid feedback on the cyindricity of axially symmetric components. A noncontact laser topography system is being added to complement the existing surface profilometer. This new instrument has been demonstrated to detect surface cracks in structural ceramics and other machined material surfaces that were previously undetected by other means. A resonant ultrasound spectroscopy system has also been procured. This instrument is used for dimensional characterization and subsurface structure determination.

During the summer of 1993, the CMUC hosted both a student on a summer research participation program and a professor and student on a fellowship program. The summer research participation student was from the University of Tennessee, and his project consisted of becoming familiar with the COSMOS/M finite element analysis software, the CARES life prediction for brittle materials software, and the Pro/Engineer solid modeling software. The student modeled several ceramic components and prepared detailed instructions for performing the life prediction calculations. An ORNL internal report will be published on this project.

The fellowship program was sponsored by the DOE Office of Environmental Restoration and Waste Management and the Historically Black Colleges and Universities (HBCU)/Minority Institutions (MI) Environmental Technology and Waste Management Consortium under the Environmental Management Career Opportunities for Minorities (EMCOM) program. The EMCOM fellowship was with North Carolina A&T State University. The purpose of the project was to develop computer models for families of ceramic parts, to develop machining programs for those parts, and to link the computer modeling system to the grinders in the CMUC.

In addition to research performed by internal users, the following research was done under a university user proposal.

Alfred University Center: CSPUC

Project Title: "Characterization of Tin Oxide Films on Glass," *J. Taylor*

The surface profilometer was used to measure the thickness of tin oxide coatings on glass. This material is commercially used as a coating on aircraft windows that can be electrically defrosted, and for infrared reflecting glazing such as for solar cells and buildings.

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

The RSUC has completed 16 months of beneficial use of a unique, state-of-the-art instrument providing high intensity X-rays along with very high precision measurements. The instrument consists of two components:

Scintag PTS 4-axis goniometer
MAC Science 18-kW rotating-anode generator

The system has been shown to have high sensitivity (~10 ppm) to strain and is very stable over a period of several days. These attributes permit measurement of strain in stiff materials such as structural ceramics as well as materials with more typical elastic properties. To date, over 20 user proposals have been approved by the HTML user advisory committee. This extensive demand that could not be met has led to initiating procurement of accessories to speed up data collection. The major accessories on order are (1) a laser-positioning sensor and (2) a position-sensitive X-ray detector.

The laser-positioning sensor will permit locating the specimen at the correct height quickly and reproducibly. This will be particularly valuable for studies involving multiple electropolishing steps to determine stress gradient as a function of depth. The position-sensitive detector (PSD) will speed data collection by a factor of five to ten, enabling user projects to collect more accurate data, faster data, or study more samples.

A number of requests for texture and orientation distribution function measurements, along with more availability for residual stress measurement, has led to the procurement of a second PTS goniometer. This instrument, using a sealed-beam X-ray tube, will be operational by early CY 1994.

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

Dr. C. R. (Cam) Hubbard Dr. X.-L. (Xun-Lee) Wang
Mr. O. B. (Burl) Cavin Dr. T. R. (Tom) Watkins
Dr. K. J. (Kris) Kozaczek

Research projects performed in this center are summarized below.

Saint Gobain-Norton Industrial Ceramics Center: RSUC

Project Title: **"Residual Stress Analysis of Si_3N_4 Cylindrical Tensile Specimens,"** *M. Foley*

The objective of this study was to determine the effect of machining-induced residual stresses (damage) on strength of tensile specimens made of 4% Y_2O_3-doped Si_3N_4 and thereby increase Norton's understanding and control of their ceramic grinding procedures. As-machined and machined-heat treated samples were examined. The results from the first visit indicated hoop compression and no axial residual stress in the samples. This conflicted with earlier work done independently upon similar samples. Major equipment problems and scheduling conflicts prohibited any further work from being done until recently.

Consequently, this study has been reduced to trying to confirm some of the results from the previous study and is still ongoing.

University of Florida Center: RSUC

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

The objective of this study was to determine the residual stresses and texture of diamond coatings on Si substrates as part of an overall project to characterize the mechanical properties of these materials. All diamond coatings showed a weak (110) texture. The in-plane residual stress in the coatings was found to be ~300 MPa (tension). The quantitative X-ray results obtained at the HTML confirmed the results from a qualitative polishing/curvature technique. Here, the substrate of one sample was carefully polished away allowing the remaining sample to bend. The sample curved concave up due to coating contraction, indicating that the coatings were in tension.

University of Missouri-Columbia Center: RSUC

Project Title: **"Measurement of Residual Stress Distribution of Stainless Steel Workpiece Machined by CNC Turning Center,"** *D. Y. Jang*

The objective of this study was to determine the effect various machining parameters have on residual stress in 304 stainless steel and then to test the validity of a finite element model of machining. The full-stress tensor was determined for eight stainless steel (SS304) samples representing different machining conditions. The residual stresses were measured using a back-reflection XRD technique and calculated using the multiple $\sin^2\psi$ method. The residual strains were measured using CrK_β radiation and (311) crystallographic reflection ($2\theta = 147.68°$). The residual stresses were calculated from the measured strains using XRD-effective elastic moduli derived from single-crystal elastic constants for 304 stainless steel using generally accepted averaging schemes. The measured strains were normalized against a stress-free standard (SS304 polycrystalline sample annealed at 2000°F for 2 h).

Idaho National Engineering Laboratory (INEL) Center: RSUC

Project Title: **"Residual Stress Distribution in Graded Ceramic-to-Metal Joints,"** *B. Rabin*

The goal of this collaborative research effort is to understand the effects of microstructure and specimen geometry on the distribution of residual stresses in ceramic-metal gradient materials using a combination of experimental and modeling techniques. XRD residual stress-mapping measurements were made at the HTML RSUC at the ORNL and at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory on a graded Ni-Al_2O_3 specimen that was fabricated at the INEL by powder-processing techniques. Surface residual strains were measured at several locations along the length of the specimen using X-ray spot sizes of 0.5 to 1.0 mm. The experimental data are currently being analyzed, and the results will be compared with finite element modeling predictions. One or more peer-reviewed publications will be generated from this work. Additional experiments are planned

for the future, including X-ray analysis of Ni-Al_2O_3 specimens having other geometries and microstructures, and neutron diffraction mapping of interior residual strain distributions.

University of Florida Center: RSUC

Project Title: "X-Ray Analysis of Residual Stress in Metals and Ceramics," *F. Baldwin and P. H. Holloway*

The objective of this study was to determine the residual stresses and stress relaxation rates in thin films of an Al-Si-Cu alloy on Si substrates in order to predict the mechanisms and times of failure. These films were found to possess an extreme (111) texture that limited the number of tilt angles (Ψ) to three. The "unpatterned" samples consisted of a uniform thin film of alloy on an Si substrate. The "patterned" samples consisted of 2.5- or 1.5-μm-wide conductor lines of alloy on a Si substrate. The stress states were found to be biaxial. The unpatterned samples possessed less residual stress than the patterned. As the line width (cross section) of the patterned samples decreased, the tensile residual stress in the alloy increased. The residual stresses in the samples containing voids in the lines were less than those without voids. These results will now be correlated with residual stresses from optical measurements of the radius of curvature.

Alpha Optical Systems, Inc. Center: RSUC

Project Title: "Residual Stress in Transparent Polycrystalline $MgAl_2O_4$ Spinel," *S. H. Evans, Jr.*

The objective of this study is to determine the state of residual stress within $MgAl_2O_4$ spinel. The samples had been subjected to different combinations of hot pressing, HIPing, and optical fabrication (grinding and polishing). Additionally, if residual stresses are present, the next objective is to determine the optimum sequence of processing steps for maximum strength and optical quality. Unfortunately, upon examination with X rays at the HTML, these samples were found to possess a large grain size (30 to 100μm) which precludes obtaining meaningful residual stress data.

AlliedSignal, Inc. Center: RSUC

Project Title: "Residual Stress and Indentation as Machining Damage Discriminators," *R. D. Silvers, R. G. Rateick, Jr., P. J. Whalen, and F. Reidinger*

The objective of this study is to determine if the machining-induced residual stresses (damage) scale with the flexural strength distribution of Si_3N_4 flexure specimens. If so, then residual stress measurements may be appropriate for part inspection purposes. To date, the results have shown that machining aggressiveness, which affects the strength of silicon nitride (AS-44), also affects the XRD peak positions. However, significant peak anisotropy precludes quantitation of the results using standard profile-fitting methods. The use of peak centroids will be attempted pending establishment of a working internet link. Once an approach for quantitative analysis of the XRD data is identified, a follow-on project may be proposed to develop the technique.

Caterpillar, Inc. Center: RSUC

Project Title: **"Residual Stress Determination of Thick Thermal Barrier Coating Systems,"** *J. L. Bjerke and C. J. Anderson*

The objective of this study is to determine elastic constants of thick thermal barrier coatings for use in current finite element models and experimental stress determinations by Caterpillar. Cylinders of plasma-sprayed ceria- and yttria-stabilized zirconia were loaded in compression and examined with X rays to determine the Young's moduli of the (213) and the (331) reflections in tetragonal and cubic polymorphs, respectively. The loading fixture, developed by Caterpillar, contained a calibrated load cell with an applied load display. This fixture was mounted on a goniometer in the "psi-geometry." The Young's moduli were determined using a $\sin^2\Psi$ analysis at various loads. The average Young's moduli for the (213) and (331) reflections were found to be 200 and 160 GPa, respectively, which are in reasonable agreement with literature values for dense zirconia. The stabilization phase appears to have only a small effect on Young's modulus. This study will continue by examining the stress-strain behavior of a Y_2O_3-stabilized ZrO_2-NiCrAlY cylinder.

In addition to supporting industrial and academic research via the User Program, the RSUC instrument has been employed on two major and several small internal projects as well.

Navy Center: RSUC

Project Title: **"Residual Stresses Due to Grinding Parameters in Al_2O_3,"** T. Watkins and C. R. Hubbard

The role of grinding parameters with respect to residual stress was investigated under Navy sponsorship. This "Navy project" was a team research effort to eliminate/reduce a persistent cracking phenomenon that occurs in ceramics utilized in the development of deep-submergence vessels. Small cracks are thought to originate from the machining processes applied to the ceramic. These cracks then grow under the cyclical loading of emergence-submergence cycles. Toward this end, the residual stresses in the ceramic were measured and related to grinding damage and the various grinding conditions. The results of the research team were pooled, and an understanding of the fracture behavior of the selected ceramic (Al_2O_3) was presented.

Laboratory-Directed Research and Development (R&D) Center: RSUC

Project Title: **"Residual Stress Mapping of a Metal/Ceramic Joint,"** *X. L. Wang, T. D. Watkins, S. A. David (Metals and Ceramics Division), S. Spooner (Solid State Division), and B. Rabin (INEL)*

This project is part of a large Laboratory-Directed R&D project on mapping residual stresses by neutron and XRD methods. In this project, the X-ray measurements will provide surface residual stress data to complement neutron residual stress-mapping data. Modeling of the joints by finite-element analysis (FEA) methods had been completed by B. Rabin of INEL, and assessment of experimental opportunities for use of synchrotron radiation have

been completed at the NSLS. High-strain gradients are predicted by the FEA codes and indicate the need for small spot sizes (<0.3 mm diam). The preliminary results obtained at NSLS indicate compressive residual stresses in the surface of the metallic part of the brazed ceramic to metal cylinder but do not agree quantitatively with the FEA results. Work is continuing to resolve this discrepancy.

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

This year has been one of continued creation and startup for the CMC. The CMC had evolved from a partnership of the Energy Efficiency (EE), Energy Research (ER), and Defense Programs (DP) programs of DOE. This partnership program, called "Cost-Effective Machining of Ceramics," has continued to gain momentum. ORNL and EE made laboratory and office space available in the HTML for the CMC, and EE provided capital funds for the purchase of a creep feed grinding machine. ER funds support a CRADA with Brown and Sharpe that provides a state-of-the-art coordinate measuring machine (CMM) for use in the CMC. DP provided ceramic CRADA support funds for seven CRADAs signed so far with three more approaching final approval. The equipment from the last of the ten laboratories to be moved was relocated in 1993. The CMM was delivered and installed in February 1993, and the creep feed grinder was delivered and installed in May 1993.

Since the stated objective of the CMC is "to develop, in conjunction with U.S. industry, advanced technologies and associated scientific concepts necessary to significantly reduce the cost of machining structural ceramics, with an initial focus on heat engine components," the efforts undertaken in the CMC will be very much industry driven. This cooperative effort with industry will influence the types of equipment installed in the CMC at any given time. This industry influence is a major factor in the forthcoming installation of a new centerless grinder provided as an in-kind CRADA contribution by Cincinnati Milacron. This state-of-the-art grinder will be utilized in developing cost-effective machining processes for ceramic heat engine components having axial symmetry, such as engine valves.

Computerization of the CMC was initiated early in 1992 with the procurement of computer aided design (CAD)/computer aided manufacturing (CAM) equipment and software. The facility also provides extensive computer modeling and graphical capabilities utilizing a SiliconGraphics workstation, Macintosh computers, and IBM-compatible computers. All of the capabilities and equipment are linked together through a DECNet/EtherNet network such that a true "Art-to-Part" process can be followed in the development of manufacturing processes for lower cost ceramic components.

Presentations on the mission and creation of the CMC have been made to numerous potential industrial CRADA partners, to the industrial Diamond Association, at a Society of Manufacturing Engineers Seminar, and to various DOE representatives.

Other personnel in the CMC are Mr. L. (Lawrence) O'Rourke (Y-12), Mr. R. (Randy) Parten, and Mr. E. (Earl) Shelton.

Voelkl, HTML staff members. Compelling evidence was obtained from the reconstructed holograms for the presence of voids in these nanoparticles. It is the first observation where nanometer-sized single crystals have been reported to have an internal void. The work has been submitted for publication.

Deactivation Mechanisms in Automotive Catalysts: Since federal regulations will soon mandate lifetimes of 100,000 miles for auto exhaust catalysts, there is renewed interest in understanding and controlling catalyst deactivation. In collaboration with Dr. Heinz Robota at AlliedSignal in Des Plaines, a series of Pt and Rh/Al_2O_3 monoliths were studied in the S-5000 high resolution SEM. The objective was to determine the size and distribution of the precious metal and to follow its evolution as the catalyst was aged at elevated temperatures in air. The samples were sections of monoliths that were washcoated with alumina in a manner similar to that for commercial auto catalysts. We were successful in showing that the same region of the sample could be imaged by SEM after successive treatments at temperatures ranging from 800 to 1100°C. Small Pt and Rh particles of nm-size could be detected with the SEM. The lack of analytical capabilities and backscatter detection on the S-5000 made it difficult to unambiguously identify the smallest particles. The low catalyst loading and the nature of the alumina made particle detection even more difficult. We plan to continue this work in future with the objective of pinpointing factors that control sintering of the precious metal under the harsh conditions encountered during auto exhaust catalysis.

Hysterisis in Chemisorption on Pd/SiO_2 Catalysts: In collaboration with Dr. Ignatius Chan at Chevron Research, Richmond, California, we have been studying the morphology of Pd particle son silica supports. These Pd particles exhibit unusual hysterisisi in the strong chemisorption of H_2 as they are cycled under oxidizing and reducing conditions. Therefore, we prepared a series of Pd catalysts on model silica supports and subjected them to high temperature treatments under oxidizing and reducing conditions. We were able to reproduce the behavior of commercial catalysts using these model supports. High resolution TEM was used to study the structures of the Pd particles to determine the factors that might explain the observed chemisorption results. This work will be submitted for publication shortly. As an extension of this work to investigate Pd/Al_2O_3 interactions in automotive catalysts is being planned with Dr. Klaus Otto at Ford Motor Company, Scientific Research Labs.

Nature of CeO_2 in CeO_2-Al_2O_3 Automotive Catalyst Supports: Ceria is an essential component in automotive catalysts. Besides providing oxygen storage capability, it appears to provide bifunctional pathways for catalysis, possibly via sites at the metal-support interface. As part of our continuing studies on ceria-alumina catalysts, we used the microanalytical capabilities of the HF-2000 to study the nature of ceria. We found evidence for an amorphous thin film of ceria on the alumina in catalysts prepared from the nitrate. When colloidal ceria was used, no such amorphous thin films could be detected. We will report these observations in a short communication to Catalysis Letters. The catalytic behavior of the two forms of ceria will be investigated in future work.

Interaction of Rh with CeO_2 in Auto Exhaust Catalysts: In previous work we found that high temperature reduction of Rh/CeO_2 leads to a several order-of-magnitude drop in hydrogenolysis activity. There is no evidence for migration of cerium suboxides on the Rh, hence the nature of these Rh-ceria interactions remain a mystery. We studied, using the 4000 EX, a series of Rh/Al_2O_3 and Rh/CeO_2-Al_2O_3 catalysts. Catalytic tests on these materials

are presently underway in my laboratory at the University of New Mexico. The results will be submitted for publication in the Journal of Catalysis.

Microstructure of MoS_2/TiO_2-SiO2 Hydrotreating Catalysis: The nature of molybdenum sulfide islands on titania-silica was investigated. In previous work we have shown that the molybdenum preferentially wets the titania component. By depositing small crystallites of titania on silica, we are able to control the structure of the catalytic sites. The high resolution TEM results will be reported along with catalytic reactivity data in a publication under preparation.

Coke Formation in Hydrotreating Catalysts: The active phase in hydrotreating catalysts contains MoS_2 promoted by Co or Ni and supported on alumina. It is known that these catalysts build up carbonaceous deposits (coke) during use which eventually degrade catalyst activity. The location of the coke on the catalyst surface, whether it is deposited on the MoS_2 or present in dose proximity on the alumina, is not well understood. We studied a series of hydrotreating catalysts in collaboration with Dr. Jeff Weisman from Texaco. The results are very interesting and will be communicated shortly for publication. It appears that two different forms of carbon are present on the catalyst surface, one is amorphous and present as a thin film while the other is quite crystalline and rests at considerable distances from he $Ni\text{-}Mo/Al_2O_3$.

The Structure of TiO_2 Photocatalyses: Titania is a semiconductor with a band gap in the uv region. The electrons and holes that are generated when TiO_2 is irradiated can serve as powerful oxidizing agents. for instance, organic compounds such as benzene can be converted to CO_2 and H_2O at room temperature. Even chlorinated compounds, such as TCE and PCE which are common constituents of contaminated waste water can be effectively mineralized. There is however very little understanding of the morphology of the titania that makes the most effective photocatalyst. Our work at New Mexico is focused on investigating the effects of particle size and morphology on photocatalytic activity. Using the 4000 EX we studied one type of TiO_2 powder to determine the nature of the primary particles, their crystallinity as well as structure. A short note to J. Solid State Chem is under preparation based on this work.

Hydrous Titanium Oxide (HTO) Catalyst Supports: HTO supports were developed at Sandia National Laboratories as efficient ion exchange materials. Originally intended to immobilize radioactive wastes, they were also found useful for stabilizing metals in a highly dispersed form. We have been investigating the catalytic properties of HTO-supported catalysts over the past several years. At the HTML, we used the S-5000 SEM to investigate thin film HTO based catalysts. Also, the 4000 EX was used to determine the mechanisms of deactivation in these catalysts and the particle size distribution of Rh as a function of hydrothermal treatment. We will report these observations in the catalysis literature.

Graduate Fellowships

The first Graduate Fellow, Mr. Alex Cozzi of the University of Florida, performed the research described here.

Microwave Joining of Alumina Using Sol-Gel Derived Adhesives

Gel Processing: The forming of gels by the sol-gel process is a time consuming procedure. The sol-gel approach includes chemical reactions that often occur slowly. However, once the finer points of sol-gel processing were mastered, gels could be manufactured on a continuous basis--that is, a finished 20 g batch of gel every two weeks. Initially, gels of two differing compositions were attempted. Produced were an all alumina gel and one containing 4 mol % chromia. The chromia was introduced as the peptizing agent in the hydrolysis of the aluminum alkoxide in the form of chromium nitrate nonahydrate.

The yield of alumina from the alumina sol was calculated so that known ratios of other materials may be introduced to alter the dielectric properties of the finished gels.

Several known Microwave Absorbing Materials (MAM) were than added to sols in a 1:1 Al_2O_3:MAM ratio. The microwave absorbing materials that were employed were Fe_2O_3, NiO, SiC and Fe_3O_4. After the powder was added to the correct volume of sol, the mixture was sonicated to disperse the powder and break up any agglomerates. A stir bar was added to the sonicated sol to maintain homogeneity and the mixture was heated on a stir/hot plate to speed the gelation. The dried gels were then crushed into a fine powder using a mortar and pestle.

As work progressed, compositions made with 10 mol % ferric nitrate nonahydrate, $Fe(NO_3)_3 \bullet 9H_2O$, and 10 mol % chromic nitrate nonahydrate, $Cr(NO_3)_3 \bullet 9H_2O$, were produced. Additions of Fe_3O_4 in varying amounts were also made to the all alumina sol.

Microwave Preparation: A 2.45 GHz microwave oven was fitted with an Omega on/off temperature controller. An inconel shielded Type K thermocouple was attached for temperature measurement of specimens within the microwave. Low dielectric loss refractory brick was machined to accommodate a specimen and thermocouple.

Initial results may have been influenced by arcing between the thermocouple and the specimen. Arcing is the release of a charge build up between two points. This can cause localized heating and even melting of the thermocouple and the specimen. Inaccurate, widely varied temperature readings is another problem associated with arcing. One way in which arcing can be reduced by increasing the surface area of the tip of the thermocouple. In this case, a thin nickel disc was placed between the thermocouple tip and the specimen. This significantly reduced the amount of arcing experienced.

Specimens were heated at full power (800 W) and temperature readings were taken periodically. Temperature as a function of time for all of the specimens produced were plotted.

The temperature of all of the gel compositions rose quickly due to the presence of free water. The rise in temperature slowed as the free water and then the bound water was eliminated. During the elimination of the bound water, gamma-alumina was formed. The composition containing 10 mol % Fe_3O_4 reached the highest temperature, 841°C. This specimen also experienced a reduction in temperature after the maximum was attained. This

may be due to a decrease in the microwave absorption of the specimen caused by the further oxidation of the iron (II,III) oxide. Visual inspection of the specimen after heating showed it to be red, supporting the idea of additional oxidation. The gel made with 10 mol % $Cr(NO_3)_3 \bullet H_2O$ also heated well by itself. Future work will include using an optical pyrometer to measure the temperature of the specimen, thus avoiding the possibility of arcing.

Differential Scanning Calorimetry (DSC): Several of the gel powders were analyzed at the HTML. Differential scanning calorimetry was performed on a Stanton Redcroft DSC 1500S under the supervision of Wally Porter. The use of the DSC will provide information involving phase changes within the gel that occur during heating. This data will be used later to determine the expected reactions that will arise during the joining process and what phases may possibly develop. The gels were heated from room temperature to 1400° C at 10 degrees per minute in flowing air. A piece of sapphire was used as a reference material to help maintain a level baseline. The data for all of the gels analyzed have been normalized for the weight of each of the gels.

All of the gels exhibited an exotherm between 100° C and 200° C. This was due to the removal of residual water from the gel. The next reaction to appear is most pronounced in the gel containing 4 mol % chromia. This occurs slightly above 200° C and has been attributed in the literature to be from pore water. The endotherm that appears between 400° C and 450° C is the removal of the structural water. This is accompanied by the formation of gamma-alumina. The gel containing 50 mol % silicon carbide has an exotherm at the end of the structural water endotherm. This is most likely due to the formation of the gamma-alumina. This peak is probably overshadowed in the other gels by the preceding endotherm. The alumina gel and the gel with 50 mol % nickel oxide added both had an exotherm of 1157° C. This was due to the formation of alpha-alumina. In the gel made with 4 mol % chromia, the alpha-alumina crystallization peak appeared at 1207° C. This increase in crystallization temperature is understandable because the chromium replaces aluminum and slightly distorts the crystal structure making it more complex and thus, more difficult to crystallize. The crystallization peak in the gel containing 50 mol % Fe_3O_4 was actually two peaks. This was followed by a sharp endotherm near 1350° C. The origins of these peaks cannot be determined without x-ray diffraction analysis and the Fe-Al-O phase diagram. One assumption that is probable is that the Fe_3O_4 has oxidized more completely during heating.

X-ray Diffraction Analysis: The ceramic powders that resulted from the heating in the DSC were subsequently analyzed using a Scintag x-ray diffraction analyzer under the supervision of Burl Cavin. The X-rays were copper k-alpha at 45 kilovolts and 40 milliamps at a wavelength of 1,54060 nanometers. The powders were scanned at one degree 2θ per minute from 20 to 40° 2-θ. A compilation of the X-ray scans for all of the powders was superimposed so that peak positions could be compared.

The powder made solely of alumina precursor had x-ray peaks that corresponded well to those of high-purity alumina. There were no extraneous peaks present. The powder containing NiO displayed peaks that can be attributed to alumina, nickel oxide and a nickel-aluminate spinel. The presence of the alumina and nickel oxide are due to their incomplete reaction in the DSC. This is understandable because of the relatively rapid heating rate used combined with no hold time at the maximum temperature. The X-ray scan for the powder

containing chromia had peaks relating to alumina but the 2θ was shifted to a smaller angle. This corresponds well o the chromia entering into the alumina as a solid solution. The larger chromium atoms will make the atomic lattice slightly larger, producing a smaller d-spacing, thus a smaller 2θ. The composition containing SiC was comprised of peaks that were related to alumina and SiC. This signified that there was no reaction between the gel and the SiC powder. The powder made with iron oxide provided two differing x-ray patterns for the powders heated to different temperatures. For the one heated to only 1200°C, the phases present were the iron oxide/alumina spinel, $FeO \bullet Al_2O_3$, alumina and iron (III) oxide, Fe_2O_3. When a powder of identical composition was heated to 1400°C, the phases present were alumina and iron (III) oxide, Fe_2O_3. The alumina peaks appeared at different 2θ values because some of the iron was dissolved as a solid solution. These values were larger than the aluminum atom and atomic lattice is smaller.

This information, coupled with dielectric data for the compositions tested, was submitted in an abstract to be presented as a poster paper at the ACerS Engineering and Ceramics Division meeting in January, 1994, with W. Porter and B. Cavin of the HTML staff, as co-authors.

Other Work: Other work being performed for this project includes consulting with M. K. Ferber regarding the direction to take for the initial testing of specimens. He has provided me with some high-purity alumina to prepare specimens for flexure strength measurements. Working with B. L. Cox and his group, we devised a configuration for machining specimens for joining and subsequent strength measurements that would include the latitude ability to compensate for mismatches that may occur during the joining process.

Three new Graduate Fellows were appointed and will start research in the HTML in early FY 1994.

Mr. A. Haynes, University of Alabama
Mr. R. Ott, University of Alabama
Mr. D. Taylor, University of Arizona

7. ACKNOWLEDGMENTS

The contributions of each staff member of the HTML and each researcher who utilized the capabilities offered by the HTML User Program are gratefully acknowledged for their role in the success of the program. Appreciation is also expressed for the support of Mr. T. J. Gross, Acting Deputy Assistant Secretary, Office of Transportation Technologies, and his staff members, Dr. J. Eberhardt, Director, Office of Advanced Transportation Materials; Mr. R. B. Schulz; and Dr. S. Diamond. The authors also thank the Metals and Ceramics Division Reports Office for final report preparation and K. Spence for editing. Special thanks goes to B. J. Russell and S. R. Odom for time spent on report preparation.

APPENDIX A

HTML ADVISORY COMMITTEES

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

Members of this committee include Dr. Maxine Savitz (AlliedSignal), Dr. James Patten (Cummins Engine Company), Mr. Ron Chand (Chand Kare Technical Ceramics), Mr. Al Chesnes (Retired/DOE), and Dr. Robert Snyder (University of Tennessee).

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

Members of this committee include Mr. Lance Groseclose (Allison Gas Turbine Division/GM), Dr. Norn Hecht (University of Dayton Research Center), Dr. Tom Whalen (Ford Motor Company), Ms. Mary Harris (DOE-ORO), Dr. Linda Horton (M&C Division/ORNL), and Dr. Vic Tennery (HTML/ORNL).

HTML User Exchange Group. Present, past, and potential HTML users are invited to attend, which provides participants an opportunity to give advice on how the User Program can be improved and made more effective.

On September 23, 1993, the fourth meeting of this group was held. Thirty researchers from the university and industrial community, the HTML staff members, and other researchers from ORNL attended this meeting. This number included 18 researchers from 5 universities and 12 researchers from 8 companies. The following technical presentations were made:

Creep Rate and Stress Rupture Properties of NT154 Si_3N_4
M. N. Menon, AlliedSignal Engines

Use of Cobalt and Nickel as a Barrier to Diffusion of Nickel Through Gold, W. K. Baxter, Coors Electronic Package Company

Ermophysical Properties and Applications of [NZP] Materials,
T. B. Jackson, LoTEC, Inc.

Measurement of an Elastic Constant in Plasma-Sprayed Zirconia Coatings Using X-Ray Diffraction, J. L. Bjerke, Caterpillar, Inc./T. Watkins, HTML

APPENDIX B

Standard Nonproprietary User Agreements Executed with Universities--July 15, 1987, through September 30, 1993

Alabama A&M University
Alfred University
Auburn University
Berea College
Brown University
Carnegie Mellon University
Case Western University
Clarkson University
Clemson University
Cornell University
Dartmouth College
Florida International University
Florida State University
Georgia Institute of Technology
Harvard University
Illinois Institute of Technology
Iowa State University
John Carroll University
Johns Hopkins University
Kent State University
Lehigh University
Louisiana State Univ. and A&M College
Marquette University
Massachusetts Institute of Technology
Michigan State University
Michigan Technological University
Mississippi College
Mount Holyoke College
New Mexico Inst. of Mining & Technol.
North Carolina State University
North Carolina State A&T University
Northwestern University
Ohio State University
Oklahoma State University
Oregon State University
Pennsylvania State University
Princeton University
Rensselaer Polytechnic Institute
Rice University
Rochester Institute of Technology
Rutgers University
South Dakota State University
Southern Illinois University
Southern University
Stanford University
Stevens Institute of Technology
Tennessee Technological University
Texas A&M University
Tuskegee University
University of Akron
University of Alabama
University of Arizona
University of California/Los Angeles
University of California/San Diego
University of California/Santa Cruz
University of Cincinnati
University of Connecticut
University of Dayton
University of Delaware
University of Denver
University of Florida
University of Houston
University of Illinois
University of Kentucky
University of Maryland
University of Massachusetts
University of Michigan
University of Minnesota
University of Missouri-Columbia
University of Missouri-Rolla
University of New Mexico
University of North Carolina/Chapel Hill
University of Notre Dame
University of Oklahoma
University of Pennsylvania
University of Pittsburgh
University of Puerto Rico
University of Southern California
University of South Carolina
University of Tennessee
University of Texas-Arlington
University of Utah
University of Washington
University of Wisconisn
Vanderbilt University
VPI & State University
Washington State University
Washington University
Wright State University

Standard Nonproprietary User Agreements Executed with Industrial Companies--July 15, 1987, through September 30, 1993

Advanced Refractory Tech., Inc.
Alcoa Technology Center
AlliedSignal
Allison Gas Turbine Div./GM
Alpha Optical Systems
Aluminum Company of America
Alzeda Corporation
Americom Inc.
American Matrix, Inc.
American Superconductor Corporation
AT&T Bell Laboratories
Babcock & Wilcox
CarboMedics, Inc.
Carborundum Company
Caterpillar, Inc.
Ceramic Magnetics, Inc.
Ceramics Process Systems Corp.
Certainteed Corporation
Chand Kare Technical Ceramics
Chrysler Corporation
Church & Dwight Company, Inc.
Concurrent Technologies Corp.
Coors Ceramics Company
CTI, Inc.
Cummins Engine Company
Detroit Diesel Corporation
DG Trim Products
Dow Chemical Company
Dow Corning Corp./Midland
Eaton Corporation
Eastman Chemical Company
E. I. du Pont de Nemours
E. I. du Pont de Nemours (Fluorochems)
Energy Conversion Devices, Inc.
Engelhard Corporation
Exxon Chemical Company
Florida Solar Energy Center
Florida Tile Industries
FMC Naval Systems Division
Ford Motor Company
Foster-Miller, Inc.
General Electric Aircraft Engines
Goodyear Tire & Rubber Company
Great Lakes Research
GTE Laboratories, Inc.
IBM Almaden Research Center
ImTech Company
INRAD, Inc.
Institute for Defense Analyses
Ionic Atlanta, Inc.
Litton Industries
LoTEC, Inc.
McDonnell Douglas Corporation
Membrane Technology Research
Miniature Precision Bearings
Monarch Tile, Inc.
Northrop Corporation
Norton Company
Norton/TRW Ceramics
Nuclear & Aerospace Materials Corporation
Pratt & Whitney
Proctor & Gamble Co.
Quadrax Corporation
Refractory Testing 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.
Torrington Company
Tosoh SMD, Inc.
Ultramet
United Technologies Research Center
Universal Energy Systems, Inc.
Westinghouse Electric Corporation

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 25 quarters of operation to date. Approximately 56% of the cumulative user days have been from local researchers, while about 34% have been from industry and 10% have been from universities. (For FY 1992, use by local researchers is 50%, by industry researchers 40%, and by university researchers 10%.)

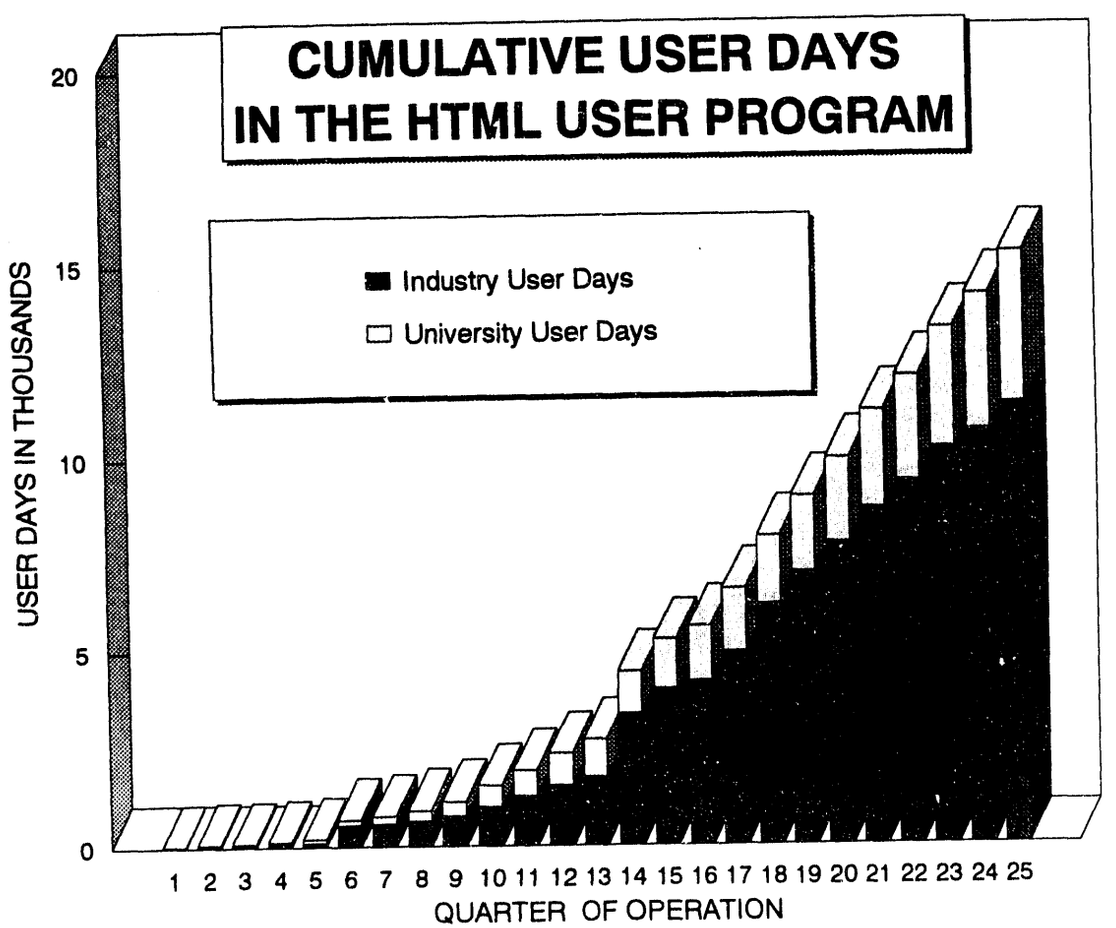

Fig. C.1. Cumulative user days in the HTML User Program.

APPENDIX D

PUBLICATIONS AND PRESENTATIONS

1. INDUSTRY USERS PUBLICATIONS AND PRESENTATIONS

L. F. Allard, E. Volkl, T. A. Nolan, C. M. Sung, K. J. Ostreicher, and B. Elman, "Electron Holography and High Resolution Electron Microscopy of NiGaAs/InP Quantum Wells Grown by Gas Source MBE," presented at John M. Cowley Symposium, Tempe, Arizona, January 5-8, 1993. GTE LABORATORIES INC

L. F. Allard, W. E. Hollar, D. W. Coffey, and R. A. Lowden,"The Ultrastructure of Bulk SiC Fibrils Grown by VLS Methods," presented at The American Society Ceramic Society (ACerS) 95th Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. CARBORUNDUM COMPANY

L. F. Allard, W. E. Hollar, D. W. Coffey, and R. A. Lowden, "The Ultrastructure of Bulk SiC Fibrils Grown by VLS Methods," submitted to *Ceramic Matrix Composites*, ed. N. P. Bansal, American Ceramic Society publishers. CARBORUNDUM COMPANY

R. B. Dinwiddie, Jr., S. C. Beecher, D. A. Bowers, and J. W. Sapp, "Thermal Conductivity of 1-D Carbon Composite Constituents After Processing," presented at the 95th American Ceramic Society (ACerS) Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. MCDONNELL DOUGLAS

R. B. Dinwiddie, Jr., S. C. Beecher, D. A. Bowers, and J. W. Sapp, "Thermal Conductivity of 1-D Carbon Composite Constituents After Processing," presented at the 95th American Ceramic Society (ACerS) Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. MCDONNELL DOUGLAS

R. B. Dinwiddie, Jr., H. Eaton, and J. Linsey, "The Effect of Thermal Aging on the Thermal Conductivity of Plasma Sprayed Fully Stabilized Zirconia," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993. UNITED TECHNOLOGY RESEARCH CENTER

C. R. Hubbard, X.-L. Wang, S. Spooner, and J. Jo, "Comparison of Residual Stresses Within HY-100 Weldments Prepared With and Without Vibratory Stress Relief", presented at the 42nd Annual Denver X-Ray Conference, Denver, Colorado, August 2-6, 1993. CONCURRENT TECHNOLOGIES

T. A. Nolan, L. F. Allard, D. W. Coffey, M. K. Ferber, and R. L. Yeckley, "Microstructural and Chemical Changes Prior to Failure in Creep-Deformed Si_3N_4 Ceramics," presented at The American Society (ACerS) 95th Annual Meeting, Cincinnati, Ohio, April 18-22, 1993. NORTON COMPANY

C. M. Sung, K. J. Ostreicher, D. Kenneson, B. Elman, L. F. Allard, and T. A. Nolan, "Electron Holography and High-Resolution Electron Microscopy of InGaAs/InP Quantum Wells Grown

By Gas Source MBE," presented at the 1992 Materials Research Society Fall Meeting, Boston, Massachusetts, November 30 - December 4, 1992. GTE LABORATORIES INC.

T. R. Watkins, C. R. Hubbard, and J. L. Bjerke, "Measurement of an Elastic Constant in Plasma Sprayed Zirconia Coatings Using X-Ray Diffraction," presented at the 42nd Annual Denver X-Ray Conference, Denver, Colorado, August 2-6, 1993. CATERPILLAR

2. UNIVERSITY USERS PUBLICATIONS AND PRESENTATIONS

L. F. Allard, E. Volkl, and A. K. Datye, "High Resolution SEM Imaging of Model Catalytic Materials," presented at the Joint UT/ORNL Ultra-High Resolution Scanning Electron Microscopy (SEM) Workshop, Knoxville, Tennessee, September 23-24, 1993. UNIVERSITY OF NEW MEXICO

L. F. Allard, E. Voelkl, D. S. Kalakkad, and A. K. Datye, "Hollow Nanocrystals" of Catalytic Palladium," submitted to *Nature*.

L. F. Allard, E.Volkl, and A. K. Datye, "High Resolution SEM Imaging of Model Catalytic Materials," Joint UT/ORNL Ultra-High Resolution Scanning Electron Microscopy (SEM) Workshop, Knoxville, Tennessee, September 23-24, 1993. UNIVERSITY OF NEW MEXICO

G. Carrasquillo, S. C. Danforth, L. F. Allard, T. A. Nolan, W. Chen, S. J. Dapkunas, G. J. Piermarini, S. G. Malghan, and A. Pechenik, "High Pressure Consolidation of Laser-Derived Nanosized Silicon Nitride Powder," presented at The American Ceramic Society 1993 PAC RIM Meeting, Honolulu, Hawaii, November 7-10, 1993. RUTGERS

Narendra B. Dahotre, Mary Helen McCay, T. Dwayne McCay, Camden R. Hubbard, Wallace D. Porter, and O. B. Cavin, "Effect of Grain Structure on Phase Transformation Events in the Iconel 718," published in *Scripa Metallurgica et Materialia*, Vol. 28, pp. 1359-1364, 1993. UNIVERSITY OF TENNESSEE SPACE INSTITUTE

K. J. Kozachek, C. O. Ruud, and J. D. Fitting, "Measurement of Residual Stresses on Ceramic Materials with High Spatial Resolution," to be published in *NDC of Materials VI*. PENNSYLVANIA STATE UNIVERSITY

K. J. Kozachek, B. G. Petrovic, C. O. Ruud, S. K. Kurtz, A. R. McIlree, "Microstructural Modeling of Grain Boundary Stresses in Alloy 600," submitted to the *Journal of Materials Science*. PENNSYLVANIA STATE UNIVERSITY

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

Ram Srinivasen, Camden R. Hubbard, O. Burl Cavin, and Burtron H. Davis, "Factors Determining the Crystal Phases of Zirconia Powders: A New Outlook," pubiished in *American Chemical Society*. UNIVERSITY OF KENTUCKY

3. HTML AND LOCAL USERS PUBLICATIONS AND PRESENTATIONS

Kathleen B. Alexander, Paul F. Becher, Xun-Li Wang, and Chun-Hway Hsueh, "Internal Stresses and the Martensite Start Temperature in Alumina-Zirconia Composites: Effects of Composition and Microstructure," submitted to *J. Am. Cer. Soc.*

L. F. Allard, "Electron Holography: Techniques and Perspectives for Materials Science," presented at the Technical Seminar, Penn State University, University Park, Pennsylvania, October 29, 1992.

L. F. Allard, E. Volkl, and T. A. Nolan, "Optical Characteristics of the Hitachi HF-2000 Cold Field Emission TEM", presented at the 51st Annual Meeting of the Microscopy Society of America (MSA), Cincinnati, Ohio, August 1-6, 1993.

L. F. Allard, E. Volkl, and T. A. Nolan, "Optical Characteristics of the Hitachi HF-2000 Cold Field Emission TEM," presented at the 51st Annual Meeting of the Microscopy Society of America (MSA), Cincinnati, Ohio, August 1-6, 1993.

S. C. Beecher and R. B. Dinwiddie, Jr., "The Thermal Conductivity of Fiber-Reinforced Ceramic Composites," presented at the 9th American Ceramic Society (ACerS) Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., A. M. Abeel, and R. A. Lowden, "The Thermal Conductivity of Silicon Nitride with Molybdenum Disilicide Additions," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., and R. A. Lowden, "The Thermal Conductivity of Carbon Coated Silicon Carbide Fibers Embedded in a Silicon Carbide Matrix," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., and A. M. Abeel, "An Automated Guarded Longitudinal Heat Flow Instrument to Measure Thermal Conductivity in the Temperature Range 85 to 500 K," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

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

S. C. Beecher, R. B. Dinwiddie, Jr., and P. Arya, "The Thermal Conductivity of Fiber-Reinforced Copper and Aluminum Matrix Composites," published in the proceedings of the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

S. C. Beecher and R. B. Dinwiddie, Jr., "The Thermal Conductivity of Fiber-Reinforced Ceramic Composites," presented at the 9th American Ceramic Society (ACerS) Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., A. M. Abeel, and R. A. Lowden, "The Thermal Conductivity of Silicon Nitride with Molybdenum Disilicide Additions," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., and R. A. Lowden, "The Thermal Conductivity of Carbon Coated Silicon Carbide Fibers Embedded in a Silicon Carbide Matrix," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

S. C. Beecher, R. B. Dinwiddie, Jr., and A. M. Abeel, "An Automated Guarded Longitudinal Heat Flow Instrument to Measure Thermal Conductivity in the Temperature Range 85 to 500 K," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

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

D. N. Braski, A. D. Underwood, L. Riester, and R. L. Jackson, "The Use of Atomic Force Microscopy in Materials Science," presented at the American Society for Information Science (ASIS), Knoxville, Tennessee, May 21-27, 1993.

D. N. Braski and K. B. Alexander, "Auger Analyses of SiC Whisker/Alumina Matrix Interfaces," presented at the 40th National American Vacuum Society (AVS) Symposium and Topical Conferences, Orlando, Florida, November 15-19, 1993.

D. N. Braski, A. D. Underwood, L. Riester, and R. L. Jackson, "Atomic Force Microscopy of Ceramic Surfaces," presented at the 95th American Ceramic Society (ACerS) Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

R. B. Dinwiddie, Jr., S. C. Beecher, and P. Arya, "The Thermal Conductivity of Carbon-Fiber-Reinforced Metal Matrix Composites," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

R. B. Dinwiddie, Jr., "Microscopic Thermal Conductivity Measurement," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

R. B. Dinwiddie, Jr., S. C. Beecher, and P. Arya, "The Thermal Conductivity of Carbon-Fiber-Reinforced Metal Matrix Composites," presented at the 22nd International Thermal Conductivity Conference, Tempe, Arizona, November 7-10, 1993.

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

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

M. K. Ferber and M. G. Jenkins, "Creep and Fatigue Behavior of Silicon Nitride Ceramics," presented at the American Ceramic Society 1993 Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

C. R. Hubbard, X.-L. Wang, S. Spooner, S. David, and T. A. Dodson, "Residual Stress Mapping Throughout a Brazed Fe-ZrO_2 Sample by Neutron and X-Ray Diffraction," presented at the American Ceramic Society (ACerS) 95th Annual Meeting, Cincinnati, Ohio, April 18-22, 1993.

C. R. Hubbard, T. A. Dodson, X.-L. Wang, S. Spooner, O. B. Cavin, and T. R. Watkins, "Non-Destructive Residual Stress Mapping Facilities by Neutron and X-Ray Powder Diffraction", presented at the 1993 American Crystallographic Association Annual Meeting, Albuquerque, New Mexico, May 23-28, 1993.

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C. R. Hubbard, X.-L. Wang, T. A. Dodson, T. Watkins, O. B. Cavin, and S. Spooner, "Nondestructive Residual Stress Mapping by X-Ray and Neutron Diffraction," presented at The Residual Stress Workshop, Oak Ridge National Laboratory, Oak Ridge, Tennessee, August 26-27, 1993.

C. R. Hubbard, "X-Ray Safety Instruction - Resources, Methods, and Responsibilities with a Goverment Laboratory Perspective", presented at The Residual Stress Workshop, Oak Ridge National Laboratory, Oak Ridge, Tennessee, August 26-27, 1993.

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C. J. Yu, J. C. Conway, Jr., J. Hirsch, C. O. Ruud, and K. J. Kozachek, "Application of Nondestructive Techniques for the Prediction of Elastic Anistropy of a Textured Polycrystalline Material," published in *Journal of Nondestructive Evaluation*, Vol. 12, No. 1, 1993.

X. Zhang, D. C. Joy, L. F. Allard, and T. A. Nolan, "Study of Ferroelectric Domain Wall Structures Using Electron Holographic Techniques," presented at the 8th International Meeting of Ferroelectrics- NIST, Gaithersburg, Maryland, August 8-13, 1993.

4. PENDING PUBLICATIONS AND PRESENTATIONS

S. C. Beecher, R. B. Dinwiddie, Jr., and P. Arya, "The Thermal Conductivity of Fiber-Reinforced Copper and Aluminum Matrix Composites," to be presented at the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

S. C. Beecher and R. B. Dinwiddie, Jr., "Modeling the Thermal Conductivity of Fiber-Reinforced Composites," will be submitted to *J. Am. Ceram. Soc. Bull.*

S. C. Beecher, R. B. Dinwiddie, Jr., and P. Arya, "The Thermal Conductivity of Fiber-Reinforced Copper and Aluminum Matrix Composites," to be presented at the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

S. C. Beecher, R. B. Dinwiddie, Jr., and P. Arya, "The Thermal Conductivity of Fiber-Reinforced Copper and Aluminum Matrix Composites," will be published in the proceedings of the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

D. N. Braski and M. C. Osborne, "The In-Situ Fracture and Auger Analysis of SiC Fibers," to be presented at the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

R. B. Dinwiddie, Jr., "The Scanning Thermal Conductivity Microprobe: Experiment Results," to be presented at the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

R. B. Dinwiddie, Jr., "The Scanning Thermal Conductivity Microprobe: Experiment Results," will be published in the proceedings of the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994.

M. Grujicic, C. R. Hubbard, S. Tangrila, W. D. Porter, and O. B. Cavin, "Effect of Iron Additions on Structure of Laves Phases in Nb-Cr-Fe Alloys", will be submitted to *Mater. Sci. Eng.* CLEMSON UNIVERSITY

L. Hehn, C. R. Hubbard, C. Zheng, and J. J. Mecholsky, Jr., "Measurement of Residual Stresses in Al_2O_3/Ni Laminated Composites Using an X-Ray Diffraction Techniques", will be submitted to *J. Am. Ceram. Soc.* UNIVERSITY OF FLORIDA

C. R. Hubbard, S. Subramaniam, O. B. Cavin, X.-L. Wang, and N. McAdams, "Anisotropic Thermal Expansion and Atomic Structure of $BaZr_4P_6O_{24}$ by HTXRD Methods", to be presented at the 18th Annual American Ceramic Society (ACerS) Meeting, Cocoa Beach, Florida, January 9-14, 1994. UNIVERSITY OF TENNESSEE

C. R. Hubbard, X.-L. Wang, S. Spooner, O. B. Cavin, and T. Watkins, "Research Opportunities and Facilities at ORNL's Residual Stress User Center", will be presented at the Fourth International Conference on Residual Stresses, Baltimore, Maryland, June 8-10, 1994.

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C. R. Hubbard, X.-L. Wang, S. Spooner, "Residual Stress Measurements within Components by Neutron Diffraction Methods," to be presented at the American Ceramic Society (ACeRS) 96th Annual Meeting & Exposition, Indianapolis, Indiana, April 24-28, 1994. (invited talk)

J. Jo, X.-L. Wang, M. J. Kleinosky, R. S. Green, C. R. Hubbard, and S. Spooner, "Evaluation of Stress Relief Treatment by Neutron and X-Ray Diffraction Methods," to be published in Proceedings of the 4th International Conference on Residual Stresses, Baltimore, June 8-10, 1994.

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X.-L. Wang, C. R. Hubbard, K. B. Alexander, P. F. Becher, J. A. Fernandez-Baca, and S. Spooner, "Neutron Diffraction Study of the Transformation Behavior in Alumina-Zirconia Ceramic Composites," will be presented at the Fourth International Conference on Residual Stresses, Baltimore, Maryland, June 8-10, 1994.

X.-L. Wang, C. R. Hubbard, S. Spooner, and S. A. David, "Residual Stress Determination in Ceramic-Metal Joints", will be presented at the 75th Annual American Welding Society (AWS), Philadelphia, Pennsylvania, April 10-14, 1994.

X.-L. Wang, C. R. Hubbard, K. B. Alexander, P. F. Becher, J. A. Fernandez-Baca, and S. Spooner, "Neutron Diffraction Measurements of the Residual Stresses in Al_2O_3-ZrO_2CeO_2 Ceramic Composites", will be submitted to *J. Am. Ceramc. Soc.*

X.-L. Wang, J. A. Fernandez-Baca, C. R. Hubbard, K. B. Alexander, and P. F. Becher, "Neutron Diffraction Study of the Tetragonal to Monoclinic Phase Transformation in Al_2O_3-$ZrO_2(CeO_2)$ Ceramic Composites," (in preparation).

T. R. Watkins, X.-L. Wang, and C. R. Hubbard, "Mapping of Residual Stresses of a Cermic-to-Metal Joint Using X-Ray Diffraction," will be presented at the Fourth International Conference on Residual Stresses, Baltimore, Maryland, June 8-10, 1994.

X. Zhang, D. C. Joy, Y. S. Zhang, T. Hashimoto, L. F. Allard, and T. A. Nolan, "Electron Holography Techniques for Study of Ferroelectric Domain Walls," will be submitted to *Ultramicroscopy*.

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