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High Temperature Materials Laboratory Second Annual Report (October 1988 through September 1989)

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

ORNL/TM-11418

Metals and Ceramics Division

HIGH TEMPERATURE MATERIALS LABORATORY SECOND ANNUAL REPORT (OCTOBER 1988 THROUGH SEPTEMBER 1989)

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HIGH TEMPERATURE MATERIALS LABORATORY SECOND ANNUAL REPORT FOR PERIOD ENDING OCTOBER 1988 THROUGH SEPTEMBER 1989*

V. J. Tennery and F. M. Foust

ABSTRACT

The High Temperature Materials Laboratory has completed its second year of operation as a designated DOE User Facility at the Oak Ridge National Laboratory. Growth of the user program is evidenced by the number of outside institutions which have executed user agreements since the facility began operation in 1987. Thirty-four institutions (11 universities, 20 industries, and 3 other government facilities) were added in FY 1989; there are now a total of 64 agreements in effect.

Forty-seven nonproprietary research proposals (22 each from universities and industries and 3 other government facilities) and 3 proprietary proposals were approved during this period. The total approved proposals to date is 100. Research projects active in FY 1989 are summarized.

INTRODUCTION

The High Temperature Materials Laboratory (HTML) is a modern research facility which houses an array of special research equipment used to meet research needs in advanced high-temperature materials, including structural ceramics and alloys. The research instruments in the four HTML User Centers provide a comprehensive set of tools for performing state-of-the-art determination of the structure and properties of solids. A key part of the HTML concept includes a staff of highly trained technical personnel who interact with industrial and university researchers in this 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 Oak Ridge complex. Support includes a wide range of involvements with research personnel such as (1) conducting research

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relating materials properties to structure, (2) characterizing of one-of-a-kind specimens, and (3) training qualified users and then providing them access to equipment to perform their own materials research.

User agreements were developed which establish the property and liability rights of the user institution and ORNL. The first official User Agreement was signed on July 15, 1987. Since that time, over 60 agreements have been executed. The first users from the university and industrial community started their research projects in August 1987.

A brief description of the capabilities available in each user center and a listing of the HTML User Center staff are included in Appendix A.

COMMITTEE ACTIVITIES

Three advisory committees assist in the successful operation of the User Centers. These committees are listed below with a brief description of their organization, function, and activities during the past year.

Advisory Committee. This committee has the responsibility of advising the HTML Director on policy for operation of the User Centers. It is composed of six members who represent the industrial and academic communities. The Committee meets at least annually and more frequently when its advice is urgently required on particular matters. A formal report containing conclusions resulting from a meeting of the Committee is submitted to the Associate Director for Physical Sciences and Advanced Materials of ORNL.

User Advisory Committee. The responsibility of this committee is to review nonproprietary research proposals and make recommendations to the HTML Director as to their acceptability. It is composed of six members--two from industry, one from a university, one from DOE, one from the M&C Division staff, and the HTML Director. A more detailed description of this committee's function and responsibilities is given in the brochure, *User Program for the High Temperature Materials Laboratory*. This committee reviewed 47 research proposals in FY 1989 (December, March, May, and July).

HTML User Exchange Group. This group was formed and a first meeting held in the HTML on August 18, 1989. A summary of this meeting is included as Appendix B. This group includes all present and past HTML users, and its purpose is to give advice and recommendations to the HTML User Center staff for improving the HTML User Program operations.

USER AGREEMENTS

There are two user agreements utilized in the HTML User Program. The "Standard Nonproprietary Agreement" is used by both state, and other, educational institutions and industrial organizations. The "Standard Proprietary Agreement" is used for all projects in which the user desires that the data and results be proprietary. Table 1 is a listing of institutions who have executed user agreements to date.

USERS

A discussion and graph showing the 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 report period are shown below.

Type of user[a]	Number of		User days
	institutions	individuals	
Industry (NP)	17	28	581
University (NP)	18	37	248
Other Government	2	2	9
Industry (P)	3	5	31
Martin Marietta Energy Systems	2	86	1427

[a]NP = nonproprietary; P = proprietary.

Figure 1 shows the number of user days per quarter during FY 1989, including all local (in-house), industrial, and university users. The industrial user days varied from about 30 to 446 during a particular quarter, university user days ranged from 97 to 37, and local user days ranged from 530 to 162. Programs supporting this in-house research included Conservation, Fossil, and Basic Energy Sciences.

Figure 2 shows the number of user days for all industrial and university users in the HTML for each quarter of FY 1989. Industry user days varied from 446 to 30 per quarter, while university user days varied from 97 to 37 per quarter.

A short summary of the research performed by nonproprietary users is given for each user organization. The header information shows the cumulative number of proposals received from that institution, and the number of researchers and user days in the HTML during FY 1989. A listing of publications and presentations resulting from User projects is given in Appendix D.

Table 1. Standard User Agreements executed July 15, 1987 through September 30, 1989

AGREEMENTS EXECUTED JULY 15, 1987, THROUGH SEPTEMBER 1988

UNIVERSITIES

Alfred University
Auburn University
Clemson University
Dartmouth College
Georgia Institute of Technology
New Mexico Institute of Mining and Technology
North Carolina State University
Oklahoma State University
Pennsylvania State University
Southern Illinois University
University of Alabama
University of Illinois
University of Michigan
University of New Mexico
University of Southern California
University of Tennessee
Vanderbilt University
Virginia Polytechnic Institute and State University

INDUSTRIES

Allied Signal
American Matrix, Inc.
Ceramics Process Systems Corp.
Dow Corning Corp./Midland
Great Lakes Research Corp.
Norton Company
Nuclear & Aerospace Materials Corp.
Selee Corp.

PROPRIETARY - 3 Agreements

AGREEMENTS EXECUTED OCTOBER 1988 THROUGH SEPTEMBER 1989

UNIVERSITY

Johns Hopkins University
Marquette University
Rice University
Southern University
University of California at Los Angeles
University of Denver
University of Florida
University of Minnesota
University of Missouri-Rolla
University of Utah

INDUSTRIES

Carborundum Company
Coors Ceramics Company
DG Trim Products
Dow Chemical Company
Energy Conversion Devices, Inc.
Foster-Miller, Inc.
GTE Laboratories, Inc.
IMTech Company
Institute for Defense Analysis
Ionic Atlanta, Inc.
Litton Industries
Sullivan Mining Corp.
Tennessee Center for R&D
Textron Specialty Materials
Thermacore, Inc.
Third Millennium Technologies, Inc.
Universal Energy Systems, Inc.

OTHER GOVERNMENT FACILITIES

Albany Research Center (Bureau of Mines)
EG&G Mound
National Institute of Science and Technology

PROPRIETARY - 6 Agreements

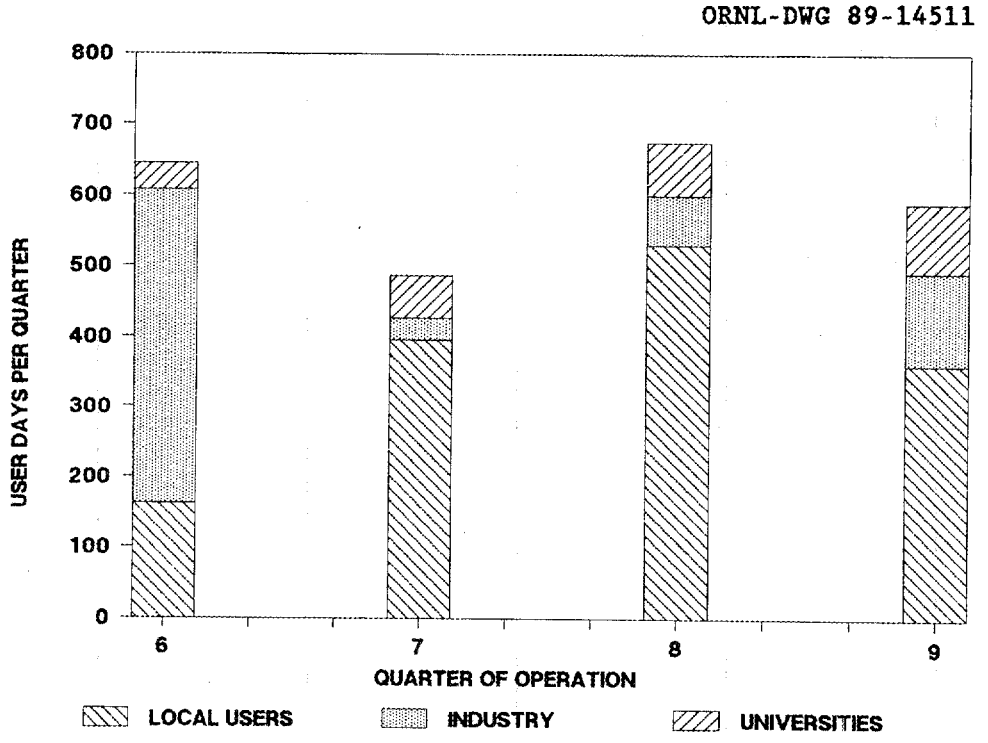

Fig. 1. Total HTML user days in FY 1989.

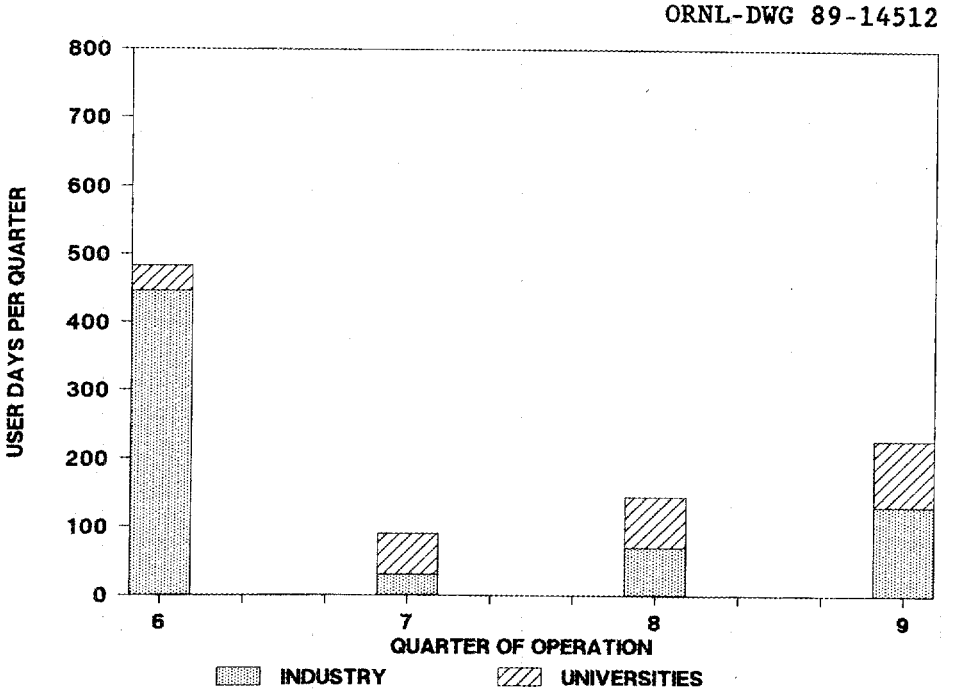

Fig. 2. Total industrial and university user days in the HTML in FY 1989.

INDUSTRIAL USERS

AMERICAN MATRIX, INC.: 5 Proposals 2 Researchers 30 User Days

Project Title: The Microstructure of Alumina Matrix Composites Reinforced with Surface-Modified Silicon Carbide Whiskers

Status: Completed

Alumina reinforced with surface-modified SiC whiskers has shown differences in fracture toughness depending upon surface treatments (including acid washing, carbon deposition, and exposure to reducing atmospheres). Analytical electron microscopy, Auger spectroscopy, and ESCA have been used to characterize whiskers prior to incorporation into composites. Analytical and high resolution electron microscopy have been used to characterize whisker-matrix interfaces after composite fabrication. Interface structures differ depending upon modification treatment, and these differences have been related to fracture toughness.

CERAMICS PROCESS SYSTEMS: 1 Proposal 2 Researchers 13 User Days

Project Title: Highly Oriented Fiber Reinforced AlN-SiC Ceramic Composites

Status: Completed. Follow-up research will be submitted as a new project.

Highly dense SiC-AlN ceramic composites with unique microstructure and properties have been successfully produced by pressureless sintering and optimum heat treatment. Appropriate sintering aids, sintering temperature, sintering period and sintering conditions were identified. The sintered SiC-AlN alloys can achieve a single phase solid solution after annealing at temperatures higher than 2225°C. The lattice constant of the solid solution varied linearly with AlN/SiC ratio. Most of the phase for assisting liquid phase sintering was crystallized and some of it was trapped inside the SiAlCN grains. Further annealing at 1860°C yielded decomposition of solid solution and form a unique microstructure, which was composed of an equiaxed grain with modulated features, elongated grains, and clean grain boundaries. The ratio of the equiaxed grains and elongated grains in the alloys can be controlled by the ratio of AlN and SiC. Preliminary mechanical testing showed that the alloys with more elongated grains have high toughness. Phase II of this research will concentrate on the mechanical properties of these ceramic alloys and the process window to achieve desired microstructure and properties.

CERAMIC PROCESS SUPERCONDUCTOR: 2 Proposals 2 Researchers 10 User Days

Project Title: Effect of Starting Raw Materials on the Formation and Properties of the Resultant $YBa_2Cu_3O_x$ Ceramic Superconductor

Status: Continuing

Project Title: Organic Additive Reactions and Ceramic Superconductor Phase Formation as a Function of Oxygen Content of the Atmosphere

Status: Continuing

CARBORUNDUM COMPANY: 1 Proposal 1 Researcher 9 User Days

Project Title: Microstructural Characterization of Silicon Nitride Ceramics Processed by Pressureless Sintering, Overpressure Sintering, and Sinter/HIP Cycles

Status: Completed

Silicon nitride ceramics of the same nominal composition have been sintered under different conditions including atmospheric sintering, overpressure sintering, and sinter/HIP cycles. The sintered ceramics, which exhibited dramatic differences in fracture toughness, have been characterized by X-ray diffraction, SEM, AEM, and image analysis techniques. Microstructural and chemical data of the grain boundary phases have been correlated with the fracture toughness data.

COORS CERAMICS: 1 Proposal 2 Researchers 9 User Days

Project Title: Investigation of Transparent Spinel Ceramics for Residual Sulfur or Other Grain Boundary Contaminants

Status: Continuing

DG TRIM: 1 Proposal 1 Researcher 5 User Days

Project Title: Determination of Mechanical Properties of Automobile Interior Trim Material

Status: Completed

New products have recently been introduced to be used in the automobile interior trim market. Various companies have developed these materials and, because of their so-far limited or non-utilization, little is known about the products, particularly their weaknesses. As a consumer of these materials, DG Trim has undertaken the task of determining their attributes such as yield and ultimate tensile strength, acoustic performance, effects of thermal and humidity cycling, and flammability. Tensile, elastic, and percent elongation were determined in the HTML on 12 materials. Most of these materials were combinations of various composite materials, such as fiberglass, polyester/polypropylene sheathed fibers, and thermosettable polyurethane foam. Change to these materials is being driven by the possibility of industry regulations and federal legislation against presently used materials. As more products are developed and used in this market, DG Trim plans to continue characterizing the materials.

ENERGY CONVERSION DEVICES: 1 Proposal 2 Researchers 18 User Days

Project Title: Orienting Superconducting YBaCuO(F) Film on Sapphire

Status: Continuing

The electrical transport properties of nonepitaxial grown $YBaCuO_x$ superconducting film on sapphire are found to be improved considerably by adding a small amount of fluorine into the film during growth. An in situ superconducting film of YBaCuO(F) has been deposited on sapphire by laser ablation from a multiphase target with a nominal composition of $YBa_2Cu_3O_6F$. A 0.5-μm as-deposited film has a zero resistance temperature of 86 K and a critical current density of 5.2×10^3 A/cm^2 at 77 K. Cross-sectional transmission electron microscopy studies indicate that the film has a columnar grain structure with the c-axis normal to the substrate. A thin layer of chemically reacted barium aluminum oxide (7-15 nm thick) is observed between the superconducting film and sapphire substrate. As a result of this chemical reaction, small amounts of impurity phases such as yttrium copper oxide and copper oxide are also present in the film, located primarily in regions between columnar grains.

GTE LABORATORIES: 2 Proposals 2 Researchers 3 User Days

Project Title: Fracture Behavior of PY6 SiN Specimens in Tension

Status: Continuing

This study is to determine a correlation between the failure stress and the state of machining damage in the critical surfaces.

INSTITUTE FOR DEFENSE ANALYSIS: 1 Proposal 2 Researchers 5 User Days

Project Title: Standardization of High-Temperature Fiber Testing

Status: Continuing

Preliminary work has started on a series of tests which will further the effort to develop a more standardized approach for high-temperature fiber testing.

IONIC ATLANTA, INC.: 1 Proposal 1 Researcher 6 User Days

Project Title: Hydroxyapatite Deposition and Analysis

Status: Continuing

Ionic Atlanta, Inc. has pioneered in the use of ion beam and plasma technology for the production of highly adherent films of hydroxyapatite. The films can be produced in both an amorphous state and a crystalline state. Adhesion was tested using a controlled scratch test and optical

microscopy. Infrared absorption spectroscopy was used to determine the presence of phosphate groups. Electron spectroscopy for chemical analysis (ESCA) showed the films to be free of contamination and Ca:P:O ratios to be characteristic of hydroxyapatite. Reflection high energy electron diffraction was used to study the crystallinity.

The unique facilities at the HTML for grazing incidence X-ray scattering provided another analytical tool to characterize the crystal structure. Preliminary results were reported at the HTML User's Meeting and the work is continuing.

LITTON INDUSTRIES: 1 Proposal 1 Researcher 14 User Days

Project Title: Characterization of Beryllium Surfaces Modified by Anodization, Ion Implantation, or an Alternate Process

Status: Continuing

NUCLEAR AND AEROSPACE MATERIALS CORPORATION: 1 Proposal 1 Researcher (NAMCO) 10 User Days

Project Title: High Conductivity, High Strength Carbon-Carbon Composites

Status: Completed. Follow-up research will be submitted as a new project.

NAMCO developed a series of highly crystalline carbon-carbon composites using high modulus mesophase derived pitch carbon fibers. These carbon-carbon composites were expected to have high thermal conductivities along with excellent mechanical properties. Thermal diffusivity was measured in-plane and cross-plane in the range 100 to 1200°C at the HTML. The thermal conductivities of the carbon-carbon composites were calculated from the thermal diffusivity values and the bulk density and heat capacity values. The optimum specimen thickness was determined prior to measuring the carbon-carbon specimens. A total of 21 specimens were measured. The thermal conductivities of the composites, as derived from the thermal diffusivity values, ranged from 108 to 164 W/m•K, when measured at 100°C. The thermal conductivity depended upon the fiber, matrix carbon, and processing used during fabrication of the composites. It is expected that carbon-carbon composites fabricated with carbon fibers of even higher thermal conductivity will be available for additional thermal diffusivity measurements in the period 1990 through 1991.

NORTON COMPANY: 4 Proposals 4 Researchers 423 User Days

Project Title: STEM Analysis of Composite Interfaces

Status: Continuing

The research started under this proposal was to characterize the stress rupture behavior of Si_3N_4-Si_3N_4 joints developed for the DOE Ceramic Technology for Advanced Heat Engines program. The studies are about 50% complete; completion of this project is uncertain at this time because the Norton user is on a leave of absence.

Project Title: Thermal Expansion Measurements of Y_2O_3-SiO_2 Phases

Status: Completed

This project was to characterize the high-temperature behavior of a hot isostatic pressed silicon nitride containing 4% yttria as a sintering aid. The high-temperature X-ray diffraction facility and the Differential Scanning Calorimeter were utilized to determine the anisotropic thermal expansion and the high-temperature instability of two yttrium silicate phase which occur in Norton Si_3N_4 ceramics. The information gained in this project allowed us to better understand the magnitude of residual stresses in Si_3N_4 matrix composites.

Project Title: Auger Analysis of Interfaces in Si_3N_4/SiCw Composites

Status: Continuing

Auger analyses of the composite interfaces were completed. Ray Padgett of the HTML staff has developed a technique for fracturing specimens in vacuum and analyzing exposed whisker surfaces which was crucial to this research. This effort is about 70% completed and remaining studies will involve the analysis of standard materials for calibrations and two additional composites.

Project Title: Analytical and Experimental Evaluation of Joining Silicon Carbide to Silicon Carbide and Silicon Nitride to Silicon Nitride for Advanced Heat Engine Applications

Status: Completed

Tests to evaluate the strain response as a function of time and stress at elevated temperature of silicon nitride joins developed at Norton Company were conducted. The joined silicon nitride was observed to survive 200 h at 1370°C under a stress of 250 MPa constant flexural stress with a maximum strain of 0.006. A comparison of strength, stress rupture, and creep of the various silicon nitride joins was the subject of a presentation at the meeting of the Engineering Ceramics Division of the American Ceramic Society in early 1989.

SULLIVAN MINING COMPANY: 1 Proposal 2 Researchers 8 User Days

Project Title: Aluminum Oxide Coatings on Cobalt Alloy Substrates

Status: Continuing

The Nanoindenter and the Auger spectrometer were used to conduct research on thin film aluminum oxide coatings deposited by ion beam assisted deposition onto cobalt alloy substrates. Some further research is planned before the results are reported.

TEXTRON SPECIALTY: 1 Proposal 1 Researcher 8 User Days

Project Title: Titanium Diboride Fibers

Status: Completed

Textron Specialty Materials utilized the mechanical properties Nanoindentor in an effort to obtain values for the modulus of experimental titanium diboride fibers produced at Textron. A standard of known modulus was also used to verify calibration of the instrument (SCS-6 SiC). Although no presentations or publications resulted from these studies, valuable experience was gained in the operation and requirements for obtaining data with the Nanoindenter instrument.

THERMACORE INC.: 1 Proposal 1 Researcher 5 User Days

Project Title: Refractory Metal Heat Pipe Compatibility with Lithium at Elevated Temperatures

Status: Continuing

The objective is to determine the compatibility of lithium with several refractory metals in order to develop a process to fabricate long-life heat pipes. The refractory metals include tantalum and columbium alloys. The heat pipes are operated in vacuum for 1000 h at temperatures up to 1150°C. Cross-sectional electron microprobe and scanning electron microscope analyses indicate that the tantalum heat pipe test specimens did not exhibit major degradation due to the corrosive lithium environment. Conclusions of this study show that the processing techniques developed by Thermacore are capable of producing long life (>1000 h) lithium heat pipes to be operated up to 1150°C in vacuum. This study is continuing and includes tantalum heat pipes with an external protective coating.

UNIVERSAL ENERGY SYSTEMS: 1 Proposal 1 Researcher 5 User Days

Project Title: SiC and Si_3N_4 Ceramics Co-implanted with Ti and C, and Cr and C Followed by High-Temperature Annealing

Status: Continuing

UNIVERSITY USERS

CLEMSON UNIVERSITY: 3 Proposals 2 Researchers 24 User Days

Project Title: Elevated Temperature Gas-Metal Reactions (2 projects)

Status: Continuing

When metals or alloys are exposed to oxidizing gases at elevated temperatures, compounds form upon their surfaces which serve to act as barriers to further reaction. The degree of protection afforded by such barriers is a function of their quality which, in turn, depends upon the structural details of the ceramic compounds formed including: defect and crystal structure, microstructure, and macroscopic structure. The evolution of these structural details will determine the serviceability of metallic materials in aggressive atmospheres.

Aspects of our ongoing research involving the HTML included following structural evolution of barrier layers formed on type 310S stainless steel and on unalloyed nickel. In situ barrier formation was studied using the X-ray Diffraction Facility of the HTML. The data collected on the high-temperature X-ray diffractometer have been interpreted in terms of diffusional models and development of stress in the protective layers.

DARTMOUTH COLLEGE: 3 Proposals 1 Researcher 10 User Days

Project Title: Chemistry of Grain Boundaries in Intermetallic Compounds

Status: Completed

The aim of the research over the past two years at the HTML has been to examine the chemistry of grain boundaries in some intermetallic compounds, viz Ni_3Al, Ni_3Si, NiAl, FeAl, and some B2 Ni-Al-Fe alloys, using the PHI 660 scanning Auger microprobe. The work on Ni_3Al, Ni_3Si is now complete. It has been shown that grain boundaries in nickel-rich Ni_3Al, Ni_3Si are nickel-enriched and that boron segregates to the grain boundaries in both these compounds irrespective of the matrix stoichiometry.

Project Title: Grain Boundary Chemistry in B2 Alloys

Status: Continuing

GEORGIA INSTITUTE OF TECHNOLOGY: 4 Proposals 4 Researchers 34 User Days

Project Title: Tension Testing of LUCALOX as a Function of Grain Size

Status: Continuing

Preliminary planning has begun for studying microcracking and microcrack localization/instability in tension bars as a function of grain size (10-150 μm). To perform this study, an Instron load frame is being modified to displacement controlled loading. Heat treatments of LUCALOX have begun at General Electric, Lighting Products Division to produce samples with 20-, 50-, 70-, and 120-μm (average) grain sizes. A preliminary visit was made to the HTML to inspect the Instron load frames for suitability of attaching a small portable Moire interferometer to study localization effects. It is expected that actual testing of the Moire interferometry optics instrumentation will begin with aluminum tension bars to ascertain uniformity of the tensile field and gain experience before testing the LUCALOX samples.

Project Title: Fiber Composition and Surface Modifications

Status: Continuing

This research determined the effects of fiber composition and surface modifications on the strength of the fiber to matrix bond in slip-cast fused silica composite materials. The composites were fabricated at the Georgia Tech Research Institute, with two compositions of fibers (3M's Nextel 312 and 440 fibers) and with three types of fiber surface modification (untreated, passivated, and boron nitride coated). Controlled indentation fiber push-out testing was performed with the Nanoindenter in the Mechanical Properties User Center. Interfacial strengths were correlated with the bulk composite mechanical properties, and provided critical property data for input to a finite element model of crack deflection about short fiber reinforcements.

Project Title: AlN-BN Nanocomposites

Status: Completed

The goal of this project was to study the phases and microstructures present in AlN-BN nanocomposites deposited by chemical vapor deposition. Samples deposited at Georgia Tech were prepared for analytical electron microscopy by the User Center staff. The samples were characterized using the analytical electron microscope, the ultrahigh resolution transmission microscope, and the electron microprobe. Electron diffraction verified the presence of both the AlN and BN phases; the electron microprobe was used to quantify the phases. The ultrahigh resolution TEM showed turbostratic BN layers with interlayer spacings of 0.333 nm surrounding hexagonal AlN fibers for certain processing conditions.

JOHNS HOPKINS UNIVERSITY: 1 Proposal 1 Researcher 5 User Days

Project Title: Plastic and Elastic Properties of Multilayer Thin Films

Status: Continuing

This study has started with the objective to quantify both the plastic and elastic properties of multilayer thin films. The study will continue into FY 1990 and the results will be used as part of a Master of Science degree thesis.

NORTH CAROLINA STATE UNIVERSITY: 1 Proposal 1 Researcher 8 User Days

Project Title: Microstructural Characterization of SiC Whisker Reinforced SiN Matrix Composites

Status: Continuing

This project involves the microstructural characterization of silicon carbide whisker reinforced silicon nitride matrix composites. These materials will be studied as a function of compressive creep. Particular emphasis is placed on changes of composition and phase of minority constituents (grain boundary phases) as related to stress and thermal history.

NEW MEXICO INSTITUTE OF MINING AND TECHNOLOGY: 1 Proposal 1 Researcher 13 User Days

Project Title: Effect of Tin Dioxide Interphase on the Mechanical Behavior of Alumina/Glass Composites

Status: Continuing

The objective of this project is to investigate the interface characteristics in the alumina/glass system. Specifically, we wish to tailor the interface to obtain an enhanced toughness. The alumina/glass system forms a very strong chemical bond, resulting in a poor toughness. The interface characteristics are changed by incorporating an interphase layer of tin dioxide between alumina and glass. This interphase layer acts as a diffusion barrier between alumina and glass, thereby inhibiting the chemical reaction and providing a weak layer and conditions beneficial for fiber pullout during the failure of the composite.

The HTML research involves Nanoindenter studies on the alumina/glass and alumina/tin dioxide/glass systems, TEM characterization, and high-temperature flexure tests of these composites. Only preliminary studies have been done to date. More extensive experimental work is planned.

PENNSYLVANIA STATE UNIVERSITY: 1 Proposal 1 Researcher 2 User Days

Project Title: Thermal Diffusivity of Specific SiC Materials

Status: Continuing

This project was to determine the temperature dependent thermal diffusivity (and thermal conductivity) of reaction-bonded (Coor's SCRB210) and a sintered-alpha (Sohio SASC) silicon carbide materials. Accurate thermal diffusivity measurements were required for the modeling of transient and steady-state thermal stresses experienced by SCRB210 and SASC radiant tubes subjected to thermal loads and cold-start conditions. This research is part of a Gas Research Institute sponsored project to develop a comprehensive radiant tube testing and analysis methodology.

SOUTHERN ILLINOIS UNIVERSITY: 1 Proposal 2 Researchers 4 User Days

Project Title: Processing of SiC Reinforced Ceramic Matrix Composites

Status: Completed

The HTML Mechanical Properties User Center was utilized to determine the four-point flexure strength and indentation fracture toughness for 16 different compositions of SiCw/ceramic matrix composites. Flexure strength was determined for over 100 specimens and fracture toughness was determined by 4 different methods on the basis of data generated for an additional 100 specimens. These results were the basis for a Masters Degree thesis.

UNIVERSITY OF ALABAMA: 1 Proposal 1 Researcher 3 User Days

Project Title: Composition of Coal Micro Constituents

Status: Pending. Due to funding difficulties and redirection of the program which requested the research, only three days of work were spent on the project before the principal investigator asked that it be suspended until more funding could be obtained at the University.

Three researchers from the university conducted research on characterizing ion implanted surfaces on titanium alloys as part of the Southeastern Universities Research Association summer research program.

UNIVERSITY OF FLORIDA: 1 Proposal 1 Researcher 9 User Days

Project Title: Characterization of Ceramic Oxide Fibers

Status: Continuing

Mullite- and zirconia-based fibers processed by the sol-gel method were studied in the analytical TEM to investigate their morphology, identify major phases present, and detect the presence of any residues from precursors and sintering aids. Mullite fibers derived from one precursor showed no evidence of concentrations at triple grain junctions. However, some intragranular microporosity was found, as well as platelet-shaped grains about 0.05 μm thick and 1.5 μm in diameter. Another

precursor was used to make mullite and mullite-zirconia fibers. In both cases, this precursor resulted in fibers that represent boundary films; in the zirconia-reinforced mullite fibers it showed small regions of amorphous as well as crystalline phase rich in Si and Y. Spherical precipitates of 20 to 60 μm were found in both phases, but mostly in the mullite, EDS (energy dispersive spectroscopy) showed that the precipitates were rich in Zr and Ca in the mullite grains, and rich in Y, Si, and Al in the zirconia grains.

Zirconia-based fibers with 10 to 15 vol % of alumina were also investigated to relate the sintering temperature to the morphology. It was found that elongated alumina grains with a preferred orientation developed at the highest sintering temperature (1500°C) and lower alumina content. At lower sintering temperatures, polygonal, smaller, randomly oriented grains of both phases were observed. Amorphous regions and boundary layers resulted at all sintering temperatures.

UNIVERSITY OF ILLINOIS: 5 Proposals 3 Researchers 46 User Days

Research Title: Effects of Fiber Surface Treatments on Adherence of Fibers to Macro Defect Free Matrices

Status: Continuing

The primary objective of this project was understanding the effects of fiber surface treatments on the adherence of fibers to macro defect free (MDF) matrices and correlate the nature of the interface to the mechanical performance of the composites. This has been successfully completed on seven composite systems via indentation experiments on the Nanoindenter to evaluate the interfacial shear strength at the fiber/matrix interface. Results from modified SiC fibers on the scanning Auger microprobe facilitated the correlation of fiber surface chemistries to the mechanical performance of the composites. Further work on the Nanoindenter to evaluate the mechanical properties at the interface is planned.

Project Title: Carbon Interphases

Status: Continuing

This research was to analyze reactions responsible for the formation of a carbon interphase region on the mechanical reliability of high-temperature ceramic matrix composites. The Physical Properties User Center equipment is being used for this study.

Project Title: Interfaces of Nickel/Alumina Composites

Status: Completed

This research involved a study of the interfaces of nickel/alumina composites which are being produced both by conventional and reaction ion

plating. The results obtained in the HTML User Centers will be the basis of a PhD thesis by the principal investigator.

UNIVERSITY OF MICHIGAN: 2 Proposals 2 Researchers 12 User Days

Project Title: A Mechanical Properties Microprobe Study of the Near-Surface Mechanical Behavior of Ion Beam Modified Nickel Subjected to Low Cycle Fatigue

Status: Continuing

The Nanoindenter has been used to quantify changes in near surface hardness of polycrystalline nickel which result from various ion implantation treatments followed by plastic strain-controlled low cycle fatigue. This study has augmented a research effort, sponsored by NSF, which has investigated the influence of ion implantation on fatigue damage accumulation in nickel. Our studies at the HTML have shown that near-surface regions of nickel substrates can be substantially strengthened by self-implantation, aluminum implantation, and ion beam mixing of evaporated Ni-Al multilayers.

Nanoindentation studies have shown that surface hardness alone is insufficient to inhibit fatigue damage accumulation. Self-implanted samples, which were strengthened substantially by implantation-induced damage, softened significantly during cyclic deformation and provided no improvement over unmodified nickel in terms of fatigue damage accumulation. Solid solution strengthening with aluminum and precipitation strengthening by gamma prime was required to suppress fatigue damage. Nanoindentation studies of these materials indicated that surface strengthening was stable with respect to cyclic deformation. Near-surface properties determinations using the Nanoindenter have proved to be a valuable tool in understanding the relationships between surface modification and surface-sensitive bulk properties such as fatigue. Work will continue with the study of coatings produced by ion beam assisted deposition (IBAD) on refractory intermetallic compounds.

UNIVERSITY OF NEW MEXICO: 2 Proposals, 1 Researcher, 4 User Days

Project Title: Fundamental Studies of Ceramic-Ceramic and Ceramic-Metal Interfaces

Status: Continuing

We have studied the nature of interfaces between hexagonal boron nitride and oxide such as alumina, titania, and magnesia. The boron nitride is synthesized by a low temperature polymeric route involving the cross-linking of borazene rings. This yields a preceramic polymer that is suitable for use as a ceramic coating and can also be drawn into BN fibers. Our research has shown that the BN forms tightly adherent coatings on all of these oxides with the basal planes of hexagonal BN always

parallel to the oxide surface. At present, we are performing detailed image calculations to derive information such as interatomic distances from the ceramic-ceramic interfaces in these systems. Another aspect being studied is the stability of these BN coatings at elevated temperatures under air exposure. The work will continue in the next year, and is being funded by the Center for Microengineered Ceramics at the University of New Mexico.

UNIVERSITY OF TENNESSEE: 9 Proposals 5 Researchers 33 User Days

Project Title: Mechanical Properties of Carbon-Depleted Zone in Cr-Mo Weldment

Status: Completed

The purpose of the investigation at the HTML was to study the mechanical properties (hardness, elastic properties, etc.) of the carbon denuded zone in 2.25Cr-1Mo weldment using the Nanoindenter. The carbon depleted zone in the weldment occurred due to diffusion of carbon (brought about by difference in carbon activity due to difference in Cr concentration) from the base metal (2.07% Cr) HAZ adjacent to the fusion line into the weld metal (2.69% Cr) as a result of an annealing and tempering heat treatment. Elastic properties of this zone were found to be similar to the base metal and weld metal although the hardness of this zone was determined to be considerably lower.

Project Title: Inconel 718 Temperature-Transformation Analysis

Status: Continuing

The cracking which generally occurs during any welding procedure on large grain or cast 718 is thought to be liquidation within the grain boundaries. Formation of carbides and intermetallic compounds which have very limited resilience to thermal stresses is also believed to be a reason for cracks in Inconel 718. In order to study the effect of successive laser pulsing (laser pulse tailoring) on the formation and elimination of these cracks, it is essential to identify those unknown phases and to verify the presence of expected compounds. The differential scanning calorimeter (DSC) and simultaneous thermal analyzer (STA) were used to determine temperature zones for different transformations in wrought as well as case Inconel 718. The samples were subjected to heating and cooling cycles with the rates 10 and 40°C/min in the temperature range 40-1350°C. The solidus temperature is about 1200°C and the alloy becomes completely liquid at about 1275°C. At about 1170°C a high-temperature phase formation occurs by solid state transformation. This transformation is not observed during cooling and may be due to redistribution of alloying elements. The two times deviation in the slope of base line in the temperature range 570-870°C indicated the presence of low temperature phases. Existence of these phases is also observed during cooling cycles.

After determining the temperature zones for different phase transformation in Inconel 718, it is essential to identify those unknown phases. In the next stage of research, the X-ray diffractometer with high-temperature furnace will be used to identify the phases at high temperatures.

Project Title: Auger Analysis of the Fracture Surfaces of a High Chromium Steel Embrittled after 30 Years of Service

Status: Continuing

A high-temperature flange bolt which was embrittled after 30 years service in a fossil-fired power plant was studied to determine the cause of the embrittlement. Samples of the as-received, embrittled material and of heat treated material with recovered toughness were impact fractured under ultra-high vacuum in the Auger spectrometer. Fracture facets of these surfaces were then analyzed using Auger electron spectroscopy. In the embrittled condition, the intergranular-appearing fracture areas were richer in P, C, Cr, and Ni than the transgranular areas. In the heat treated condition, all four elements had decreased in concentration. After sputtering, the Cr and C contents increased for only the embrittled condition and only on the intergranular fracture areas.

Project Title: Auger Analysis of Embrittlement in Ordered Ni_4Mo Containing Be

Status: Continuing

The alloy Ni 20 at. % Mo becomes ordered below 868°C, and in this condition it is very brittle, with fracture occurring intergranularly along the prior disordered, high-angle boundaries. There is evidence that Be reduces the brittleness. Thus, we have examined the fracture facets of an ordered Ni_4Mo alloy containing about 1000 ppm Be. The samples were fractured under ultra-high vacuum in the Auger spectrometer, and then fracture facets analyzed using Auger electron spectroscopy.

UNIVERSITY OF UTAH: 3 Proposals 2 Researchers 20 User Days

Project Title: Tetragonal Zirconia Polycrystal Systems

Status: Continuing

This research involved conducting stress/strain experiments and X-ray analysis of tetragonal zirconia polycrystal systems. This project will utilize equipment in all four user centers before it is completed.

Project Title: High Thermal Conductivity Aluminum Nitride Ceramic

Status: Continuing

The energy dispersive X-ray spectroscopy (EDX) capabilities of the analytical transmission electron microscope were used to determine the phase present and examine their morphologies. Changes in second phases as a function of annealing time were determined. Thermal diffusivity will be measured in the Physical Properties User Center and correlated with the phases present in the grain boundaries.

VANDERBILT UNIVERSITY: 5 Proposals 2 Researchers 8 User Days

Research Project: Devitrification of Niobium-Nickel Alloys

Status: Completed

The devitrification of niobium-nickel metallic glasses prepared by containerless melting and splat quenching was studied using the differential scanning calorimeter of the Physical Properties User Center. There is considerable disagreement in the literature about the phases present and the devitrification sequence. Transmission electron microscopy of quenched samples was used to determine the phases present at various annealing temperatures.

Research Project: Microhardness of Ti-4 wt % Ce Alloys with Fine Dispersions of Cerium

Status: Completed

The Vanderbilt Center for the Space Processing of Engineering Materials, in a project with General Electric Corporation R&D Center, has studied the effects of undercooling on microstructural development in titanium-4 wt % cerium alloys. Extremely fine dispersions of cerium particles in the matrix were obtained by undercooling and rapidly quenching these alloys. The Nanoindenter in the Mechanical Properties User Center was used to determine the effects of these small dispersions on the matrix hardness.

PUBLICITY, AWARDS, AND PROMOTIONS OF THE HTML
(Underlines indicate User Center staff)

The exhibit which prominently features the HTML has been displayed at several major conferences in both the United States and other countries. Display locations included Las Vegas, Nevada (November 1989) (3rd International Symposium, Ceramic Materials and Components for Heat Engines); the annual American Ceramic Society Meeting in Indianapolis (April 1989); and the Contractors' Coordination Meeting, Dearborn, Michigan (October 1988). Handouts describing the HTML were distributed at each of these meetings.

The Best Poster Prize presented at the 13th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, January 15-18, 1989, was awarded to K. L. More and R. A. Lowden for their poster, entitled "Electron Microscopy Characterization of Interfaces in CVD-Coated Nicalon Fiber-Reinforced SiC."

The HTML publicity brochure, *User Program for the High Temperature Materials Laboratory*, won a second place Award of Excellence in the Technical Communication Competition sponsored by the East Tennessee Chapter of the Society for Technical Communication, 1988/89. Authors of this publication are V. J. Tennery and F. M. Foust.

The HTML publicity brochure, *The High Temperature Materials Laboratory, a New Research and User Facility at the Oak Ridge National Laboratory*, won a Third Place, Award of Merit, in the Technical Communication Competition sponsored by the East Tennessee Chapter of the Society for Technical Communication, 1988/89. Authors of this publication are F. M. Foust, V. J. Tennery, and J. V. Cathcart.

The 1st Prize in the Ceramographic Exhibit at the American Ceramic Society Meeting, Indianapolis, 1989, was awarded to L. F. Allard, T. A. Nolan, and M. H. Rawlins, for their Poster, entitled "Structure of a Grain Boundary Phase in a Monolithic Silicon Carbide Ceramic." AMERICAN MATRIX, INC.

A presentation promoting the HTML, "The High Temperature Materials Laboratory at ORNL - A National Resource," was made at the following locations.

Southeastern Section of the American Physical Society, Raleigh, November 10-12, 1988, by V. J. Tennery

College of Ceramics, Alfred University, Alfred, New York, December 8, 1988, by C. R. Hubbard

Physics Department Colloquia, University of Delaware, Newark, March 16, 1989, by C. R. Hubbard

Federal Laboratory Consortium for Technology Transfer, Chicago, May 2-5, 1989, by V. J. Tennery

Deutsche Forschunge - und Versuchsanstalt für Luft - und Raumfahrt e.V. (DFVLR), Cologne, Federal Republic of Germany, June 16, 1989, by V. J. Tennery

ACKNOWLEDGMENTS

The authors wish to express appreciation to the HTML User Center staff members and to our users for making the User Program a success.

APPENDIX A

HTML PHYSICAL FACILITIES AND PERSONNEL

The Director of the High Temperature Materials Laboratory (HTML) User Program is Dr. V. J. Tennery, who is also a section head in the Metals and Ceramics Division, the parent organization of the HTML. Dr. Tennery is Chairman of the User Advisory Committee which is responsible for review of the research proposals from university and industrial users. Ms. Carole Yount is Dr. Tennery's secretary.

The administrative support staff and their functions are

Ms. Felicia Foust, User Coordinator
Ms. Donna Conger, secretarial responsibilities, User Program
Ms. Pam Rice, secretarial responsibilities, User Staff
Ms. Robin Martin, secretarial assistance

A description of the function of each of the four user centers in the HTML is given here. The User Centers' staff are listed also.

MATERIALS ANALYSIS USER CENTER (MAUC)

The materials characterization performed in the MAUC refers primarily to microstructure determinations and to the chemical and morphological characterization of surfaces utilizing sophisticated state-of-the-art instruments, generally not available to the user at the home institution.

The Group Leader of the MAUC is Mr. T. A. Nolan. Other staff and their equipment expertise are

Dr. L. F. (Larry) Allard, 4000EX transmission electron microscope and 2000FX analytical electron microscope
Dr. A. (Ashok) Choudhury, ESCA/SIMSLAB2 multitechnique surface analyzer and scanning Auger microprobe
Ms. D. W. (Dorothy) Coffey, field emission scanning electron microscope and sample preparation
Mr. L. A. (Larry) Harris, ESCA/SIMSLAB2 multitechnique surface analyzer
Mr. T. J. (Tommy) Henson, electron microprobe
Ms. K. L. (Karren) More, 2000FX analytical electron microscope and 4000EX transmission electron microscope
Mr. T. A. (Tad) Dodson, computer services support

MECHANICAL PROPERTIES USER CENTER (MPUC)

The MPUC is dedicated to the study of high-temperature mechanical performance of structural ceramics and alloys, including silicon carbide

silicon nitride, aluminum oxide, and transformation toughened zirconia. Facilities are available for the measurement of strength and toughness as a function of time, temperature, and stress conditions. In addition to standard flexure testing, the MPUC has 12 state-of-the-art tensile systems capable of evaluating tensile strength to temperatures up to 1600°C.

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

Dr. M. G. (Michael) Jenkins, tensile and flexure test facilities
Mr. R. L. (Ralph) Martin, Universal test machines and sample preparation
Ms. D. C. (Denise) Sammons, Nanoindenter

PHYSICAL PROPERTIES (PPUC) AND X-RAY DIFFRACTION USER CENTERS (XRDUC)

The PPUC is organized to include instruments for determination of a broad range of the thermal physical properties of solid materials. Facilities are operational for the measurement of thermal diffusivity, specific heat, thermal expansion, DTA/TGA/mass spectroscopy and DSC up to 1500°C. Efforts have begun to expand the modeling and measurement of thermal transport in materials.

X-ray diffraction is used to study anisotropic thermal expansion, phase equilibria, and microstructural defects of materials at either elevated temperatures or room temperature. The high-temperature X-ray diffraction furnace is capable of operation to 1600°C in air or other environments and to 2700°C in vacuum. Extensive data processing facilities and gas environment monitoring and control capabilities are being developed.

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

Mr. O. B. (Burl) Cavin, all X-ray equipment
Dr. R. B. (Ralph) Dinwiddie, laser flash diffusivity

APPENDIX B

SUMMARY OF FIRST ANNUAL USER EXCHANGE GROUP MEETING

The first User Exchange Group meeting was held at the HTML on August 18, 1989. In addition to in-house users and staff, 27 users and potential users from 5 universities and 17 industries attended.

Mr. A. A. Chesnes, Director of the Heat Engine Propulsion Division of the Office of Transportation, Conservation and Renewable Energy Office, DOE, attended and addressed the Group in the afternoon. Ms. Martha Rohr of the Oak Ridge Operations Office gave a brief welcome and introduced the speakers in the morning session. An abstract of each user presentation is included in this Appendix.

Representatives from the Oak Ridge Associated Universities were available to discuss potential financial support for university students and faculty in support of their research projects in the HTML User Program.

After a buffet luncheon in the building, the group reconvened for presentations from Mr. Chesnes; Dr. V. J. Tennery, Director of the HTML; and Dr. Barry Burks, Director of the ORNL Office of Guest and User Interaction.

Mr. Chesnes discussed the growth of the user center activity and how the program is funded. Dr. Tennery discussed the items needed to upgrade our existing equipment and the possibility of adding two new user centers to the existing four.

Dr. Burks gave a short presentation on the function of the new Office of Guest and User Interaction to facilitate users' entries into the Laboratory.

General Comments

The requirements of badging a user into ORNL was discussed briefly. Some of the users had experienced a delay in being admitted due to requirements for completing forms in the Personnel Office and going through the medical procedure. Although this is time consuming, most comments were positive in that the users were appreciative of the treatment they received in Medical.

Dr. J. Danko of the University of Tennessee commented that the university people were very impressed with their ability to access the equipment in the HTML to further their research projects. He complimented the User Center staff who provide expert training to the users; he also mentioned that access to office space for users on site was very appealing to the users.

Dr. R. Young of Energy Conversion Devices brought up the point of having to spend so much time on specimen preparation which cut down on actual instrument time for the characterization. Dr. J. Wolf of Clemson commented that this was an area in which the recently organized Southeastern University Research Association (SURA) program might do something. Dr. V. J. Tennery commented that this was a problem and that it would be great for the User Program if the SURA specimen preparation area did come to pass.

Mr. J. Stevenson of Ionic Atlanta had two concerns. The first was the time it might take to get approval to do research after the initial visit. Dr. Tennery answered that unless there was a drastic change in the direction of the research, the Committee did not have to meet to approve other visits. Mr. Stevenson also questioned what was considered high-temperature research. Dr. Tennery answered that that depended on the materials being studied and the instruments being used.

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 nine quarters of operation to date. Approximately 70% of the users have been local researchers, while about 21% have been from industry and 9% have been from universities.

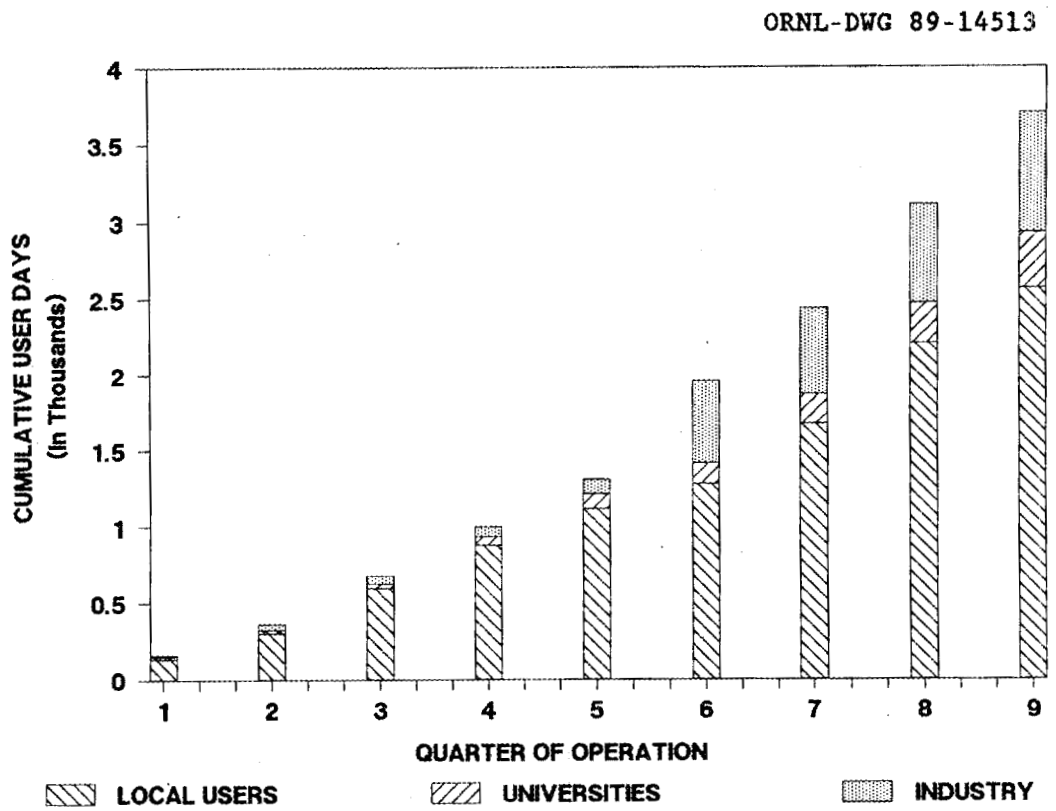

Fig. C.1. HTML cumulative user days from start of User Program.

APPENDIX D

PUBLICATIONS AND PRESENTATIONS

User Center Staff are indicated by an underline. The user's home institution is listed at the end of the citation.

INDUSTRY USERS PUBLICATIONS

M. H. Rawlins, T. A. Nolan, V. J. Tennery, and L. F. Allard, "Dynamic and Static Fatigue Behavior of Sintered Silicon Nitrides: II, Microstructure and Failure Analysis," *J. Am. Ceram. Soc.* 72(8), 1338-1342 (1989). AMERICAN MATRIX, INC.

R. T. Young, G. A. Van der Leeden, B. Chao, D. Pawlik, S. R. Ovshinsky, L. F. Allard, and K. L. More, "Oriented Superconducting YBaCuO(F) Film on Sapphire," submitted, *Applied Physics Letters*. ENERGY CONVERSION DEVICES

K. R. Selkregg, K. L. More, G. Seshadri, and C. H. McMurtry, "Microstructural Characterization of Silicon Nitride Ceramics Processed by Pressureless Sintering, Overpressure Sintering, and Sinter/HIP Cycles," to be published in the conference proceedings, 14th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 14-17, 1990. CARBORUNDUM

Camden Hubbard, Ran-Rong Lee, Wen-Cheng Wei, "XRD Investigation of SiC-AlN Solid Solution," to be submitted to the International Center of Diffraction Data. CERAMICS PROCESS SYSTEMS

INDUSTRY USERS PRESENTATIONS

T. A. Nolan, M. H. Rawlins, R. H. Krabill, and L. F. Allard, "The Influence of Whisker Surface Modifications on the Microstructure and Properties of SiC Whisker-Reinforced Alumina," annual meeting, American Ceramic Society, Indianapolis (1989). AMERICAN MATRIX, INC.

R. T. Young, G. A. Van der Leeden, B. Chao, D. Pawlik, S. R. Ovshinsky, L. F. Allard, and K. L. More, "Oriented Superconducting YBaCuO(F) Film on Sapphire," HTML User's Meeting, Oak Ridge, August 18, 1989. ENERGY CONVERSION DEVICES

L. F. Allard, T. A. Nolan, M. H. Rawlins, and J. Chang, "The Effects of Anomalous Macrograins on the Failure of a Monolithic SiC Heat Engine Ceramic," poster session at annual meeting, American Ceramic Society, Indianapolis (1989). AMERICAN MATRIX, INC.

L. F. Allard, T. A. Nolan, T. N. Tiegs, S. A. Bradley, and K. R. Karasek, "The Ultrastructure of Silicon Carbide Whiskers: Structure and Chemistry of Surfaces and Inclusions," TMS Fall Meeting, Indianapolis (1989) (invited presentation). ALLIED SIGNAL

K. R. Selkregg, K. L. More, G. Seshadri, and C. H. McMurtry, "Microstructural Characterization of Silicon Nitride Ceramics Processed by Pressureless Sintering, Overpressure Sintering, and Sinter/HIP Cycles," 14th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 14-17, 1990. CARBORUNDUM

G. J. Sundberg and M. K. Ferber, "Joining of Silicon Nitride for Heat Engine Applications," 13th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 1989; will be published in conference proceedings. NORTON COMPANY

Camden Hubbard, Ran-Rong Lee, Wen-Cheng Wei, "XRD Investigation of SiC-AlN Solid Solution," Advances in X-Ray Analysis, Denver, August 2-4, 1989. CERAMICS PROCESS SYSTEMS

C. R. Hubbard, N. Corbin, O. B. Cavin, and D. Devlin, "Anisotropic Thermal Expansion of Delta-Yttrium Disilicate and Yttrium Nitrogen Apatite by High Temperature X-Ray Diffraction," Advances in X-Ray Analysis, Denver, July 31-August 4, 1989. NORTON COMPANY

G. B. Engle, J. A. Tallon, and R. S. Graves, "High Conductivity, High Strength Carbon-Carbon Composites," joint NASA/Air Force sponsored meeting to be held in Cocoa Beach, Florida, January 1990. NUCLEAR AND AEROSPACE MATERIALS CORPORATION

UNIVERSITY USERS PUBLICATIONS

A. K. Datye, R. T. Paine, C. K. Narula, and L. F. Allard, "Novel Approach for High Resolution Imaging of Ceramic-Ceramic Interfaces," in *Interfaces Between Polymers, Metals and Ceramics*, B. M. Dekoven et al., ed., *MRS Proceedings*, Vol. 153 (1989), in press. UNIVERSITY OF NEW MEXICO

D. A. Koester, R. F. Davis, and K. L. More, "Kinetics and Mechanisms of High Temperature Creep of SiC Whisker Reinforced Si_3N_4," to be submitted to the *Journal of the American Ceramic Society*. NORTH CAROLINA STATE UNIVERSITY

J. Hanigofsky, K. L. More, and J. Lackey, "Characterization of the AlN + BN Single Phase and Co-Deposition System," to be submitted to *Journal of the American Ceramic Society*. GEORGIA RESEARCH INSTITUTE OF TECHNOLOGY

J. S. Wolf, O. B. Cavin, and J. H. DeVan, "The Oxidation of Type 310 Stainless Steel in Mixed Gases at Elevated Temperatures," to be published as an ORNL/TM report (1989). CLEMSON UNIVERSITY

I. Baker, E. M. Schulson, J. R. Michael, and R. A. Padgett, "Grain Boundary Chemistry in Ni_3Al and Ni_3Si," submitted to *J. de Physique*. DARTMOUTH COLLEGE

I. Baker, R. A. Padgett, and E. M. Schulson, "Auger Electron Spectroscopy Study of Ni_3Si," to be published in *Scripta Metallurgica* (November 1989). DARTMOUTH COLLEGE

UNIVERSITY USERS PRESENTATIONS

A. K. Datye, R. T. Paine, C. K. Narula, and L. F. Allard, "Novel Approach for High Resolution TEM Studies of Ceramic-Ceramic Interfaces," Materials Research Society, Spring Meeting, San Diego (April 1989). UNIVERSITY OF NEW MEXICO

L. F. Allard, A. K. Datye, and R. T. Paine, "Atomic Structures of Crystalline Boron Nitride Interfaces with Ceramic Substrates," Fall Meeting of the Materials Research Society, Boston (December 1989). UNIVERSITY OF NEW MEXICO

K. L. More, D. A. Koester, and R. F. Davis, "Microstructural Characterization of SiC Whisker Reinforced Si_3N_4 Composites Before and After Creep Deformation," 91st Annual Meeting of the American Ceramic Society, Indianapolis, April 23-27, 1989. NORTH CAROLINA STATE UNIVERSITY

D. A. Koester, R. F. Davis, and K. L. More, "Kinetics and Mechanisms of High Temperature Creep of SiC Whisker Reinforced Si_3N_4," 91st Annual Meeting of the American Ceramic Society, Indianapolis, April 23-27, 1989. NORTH CAROLINA STATE UNIVERSITY

A. Choudhury, C. R. Brooks, and R. A. Padgett, "Auger Analysis of the Fracture Surfaces of a High Chromium Embrittled After 30 Years of Service," Spring AIME Meeting. UNIVERSITY OF TENNESSEE

A. Choudhury, C. R. Brooks, and R. A. Padgett, "Auger Analysis of Embrittlement in Ordered Ni_4Mo Containing Be," Spring AIME Meeting. UNIVERSITY OF TENNESSEE

O. B. Cavin and J. S. Wolf, "High Temperature Oxidation of Stainless Steel," 38th Annual Denver X-Ray Conference, Denver (July 31 through August 4, 1989). CLEMSON UNIVERSITY

P. H. McCluskey, R. K. Williams, R. S. Graves, and T. N. Tiegs, "Thermal Diffusivity/Conductivity of Alumina-Silicon Carbide Composites," 13th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, Florida, January 15-18, 1989. ALFRED UNIVERSITY

LOCAL USERS PUBLICATIONS

L. F. Allard, D. W. Coffey, E. K. Ohriner, and V. K. Sikka, "Interface Reactions in a High Temperature Metal Matrix Composite," *Proceedings EMSA 1989*, G. W. Bailey, ed., Claitors Publishing Division. ORNL

H. C. Foley, J. S. Brinen, A. J. Garratt-Reed, and L. F. Allard, "Bimetallic Catalysts Comprised of Dissimilar Metals for the Reduction of Carbon Monoxide with Hydrogen," submitted to *Applied Catalysis*. ORNL

C. S. Yust and L. F. Allard, "Wear Characteristics of an Alumina-Silicon Carbide Whisker Composite at Temperatures to 800°C in Air," in *Tribology Transactions* 32, 331-338, proceedings of conference held at Baltimore, October 16-19, 1988, Society of Tribologists and Lubrication Engineers (1989). ORNL

R. E. Clausing, L. Heatherly, K. L. More, and G. M. Begun, "Electron Microscopy of Growth Features on Filament Assisted CVD Diamond Films," to be published in *Surface and Coatings Technology*. ORNL

K. L. More and R. A. Lowden, "Characterization of Interfaces in CVD-Coated Nicalon Fiber-Reinforced SiC," published in the Conference Proceedings of the Electron Microscopy Society Meeting in San Antonio, August 7-11, 1989 (pp. 556-557). ORNL

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R. E. Clausing, L. Heatherly, K. L. More, and G. M. Begun, "Structures of Filament Assisted CVD Diamond Films and Crystals," to be submitted to *Journal of Vacuum Science and Technology*. ORNL

S. P. Withrow, K. L. More, R. A. Zhur, and T. E. Haynes, "Ion Beam Deposition of Beta-SiC Layers onto Alpha-Sic Substrates," to be published in *Vacuum*. ORNL

K. L. More, S. P. Withrow, T. E. Haynes, and R. A. Zuhr, "Growth of Epitaxial SiC Layers Onto On- and Off-Axis 6H-SiC Substrates by Ion Beam Deposition," submitted for publication to the Materials Research Society (1989). ORNL

T. B. Lindemer, J. F. Hunley, J. E. Gates, A. L. Sutton, Jr., J. Brynestad, C. R. Hubbard, and P. K. Gallagher, *Experimental and Thermodynamic Study of Nonstoichiometry in* $\langle YBa_2Cu_3O_{7-x}\rangle$, ORNL/TM-10899 (May 1989). To be published in *J. Am. Ceram. Soc.* (Oct. 1989) also. ORNL

K. L. More, R. A. Lowden, T. M. Besmann, and R. D. James, "Microstructural Characterization of Multiphase Coatings Produced by CVD," submitted for publication to the Materials Research Society (1989). ORNL

M. K. Miller, P. Angelini, A. Cerezo, and K. L. More, "Pulsed Laser Atom Probe Characterization of SiC," to be published in *Journal de Physique*. ORNL

K. L. More and M. K. Miller, "Composition and Microstructure of a Nickel-Base Superalloy, Udimet 720," to be published in the conference proceedings, Annual Meeting of the Electron Microscopy Society of America, San Antonio, August 7-11, 1989 (pp. 280-281). ORNL

C. R. Brinkman, B. E. Foster, K. C. Liu, G. M. Begun, R. L. Graves, W. A. Simpson, O. B. Cavin, and W. K. Kahl, *Influence of Diesel Engine Combustion on the Rupture Strength of Partially Stabilized Zirconia*, ORNL-6513 (December 1988). ORNL

E. P. George, C. T. Liu, and R. A. Padgett, "Comparison of Grain Boundary Compositions in B-Doped and B-Free Ni_3Al," *Scr. Metall.* 23, 979-82 (1989). ORNL

H. M. Henson, M. J. Bridges, R. M. Sadler, and T. J. Henson, *Microstructural Evaluation of Refractory Coatings on Turbine Engine Components*, K/QT-183, (November 1988). OAK RIDGE GASEOUS DIFFUSION PLANT,

C. E. Bamberger, C. S. MacDougall, and O. B. Cavin, "Formation of Molybdenum Carbides by Reaction of Molybdenum Compounds with Molten Sodium Cyanide," *React. Solids* 6, 369-72 (1989). ORNL

K. L. More, H. S. Kong, J. T. Glass, and R. F. Davis, "Electron Microscopy of Defects in Epitaxial Beta-SiC Thin Films Grown on Si and C (0001) Faces of Alpha-SiC Substrates," submitted to *Journal of the American Ceramic Society*. ORNL

Y. Zhang, J. M. Stewart, B. Morosin, and C. R. Hubbard, "The Importance of Consistent 1/d Scans in Determining Size and Strain by Powder Diffraction Profile Analysis." to be published in *Advances in X-Ray Analysis* (1990). ORNL

LOCAL USERS PRESENTATIONS

P. Angelini, T. A. Nolan, and C. E. Bamberger, "Analytical Electron Microscopy of TiN and CrN Whiskers," Symposium on Advanced Characterization Techniques for Ceramics, San Francisco, October 23-26, 1988. ORNL

K. L. More and R. A. Lowden, "Electron Microscopy Characterization of Interfaces in CVD-Coated Nicalon Fiber-Reinforced SiC," poster session at 13th Annual Conference on Composites and Advanced Ceramics, Cocoa Beach, January 15-18, 1989; 91st Annual Meeting of the American Ceramic Society, Indianapolis, April 23-37, 1989. ORNL

K. L. More and R. A. Lowden, "Characterization of Interfaces in CVD-Coated Nicalon Fiber-Reinforced SiC," Electron Microscopy Society Meeting in San Antonio, August 7-11, 1989. ORNL

D. P. Stinton, D. M. Hembree, K. L. More, M. Headinger, and R. F. Davis, "Matrix Characterization of Fiber-Reinforced SiC-Matrix Composites Fabricated by CVI," 176th Meeting of the Electrochemical Society, Hollywood, Florida, October 15-20 (1989); paper in progress. ORNL

D. P. Stinton, D. M. Hembree, K. L. More, and T. M. Besmann, "Characterization of Ceramic Matrix Composites Fabricated by CVI," Materials Research Society Annual Meeting, Boston, Nov. 27-Dec. 2, 1989. ORNL

R. E. Clausing, L. Heatherly, K. L. More, and G. M. Begun, "Electron Microscopy of Growth Features on Filament Assisted CVD Diamond Films," International Conference on Metallurgical Coatings, San Diego, April 17-21, 1989.

R. E. Clausing, L. Heatherly, K. L. More, and G. M. Begun, "Growth and Characterization of Diamond Films," 9th Symposium of the Tennessee Valley Chapter of AVS, May 15-17, 1989, Oak Ridge. ORNL

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