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MARTIN MARIETTA

High Temperature Materials  
Laboratory First Annual Report  
(June 1987 Through September 1988)

V. J. Tennery  
F. M. Foust

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HIGH TEMPERATURE MATERIALS LABORATORY  
FIRST ANNUAL REPORT  
(JUNE 1987 THROUGH SEPTEMBER 1988)

V. J. Tennery  
F. M. Foust

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HIGH TEMPERATURE MATERIALS LABORATORY  
FIRST ANNUAL REPORT FOR  
JUNE 1987 THROUGH SEPTEMBER 1988\*

V. J. Tennery and F. M. Foust

**ABSTRACT**

The High Temperature Materials Laboratory, a DOE-designated national user facility at the Oak Ridge National Laboratory, became operational in June 1987. User research in the four User Centers has steadily increased since the first standard user agreement was signed in July 1987. To date, 17 universities and 11 industrial companies have signed agreements to utilize the facility, and 45 research proposals have been approved.

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**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 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 four User Centers in the HTML staffed with highly trained technical personnel who interact with industrial and university researchers. Instruments for these four User Centers represent about \$6.5M or 34% of the total appropriations (\$19.1M) for the facility. Installation of the research instruments for the User Centers was completed in June 1988.

The HTML became an official DOE-designated National User Facility in April 1986. The User Centers are organized to provide materials characterization support to research programs throughout the Oak Ridge complex and to appropriate university and industrial users. Support is defined as including a wide range of involvements with research personnel such as (1) conducting programs relating materials properties to structure, (2) routine single sample characterization, and (3) allowing qualified users access to equipment to perform their own characterization.

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

The first official User Agreement was signed on July 15, 1987. Initial funding for User Center staff was \$2M in FY 1988 from the Office of Transportation Systems.

### PHYSICAL FACILITIES

The four User Centers located in the HTML are: (1) Materials Analysis, (2) Mechanical Properties, (3) Physical Properties, and (4) X-Ray Diffraction. A brief description of each of these Centers and a list of the major equipment in each are given here. The HTML brochure, *The High Temperature Materials Laboratory, A New Research and User Facility at the Oak Ridge National Laboratory*, gives a more detailed description of the capabilities and use of this equipment.

Six offices have been set aside in the HTML for assignment to users during their research time at the HTML. These offices are located within the space assigned to the User Centers.

Figure 1 is a floor plan of the building showing the space assigned to each User Center, the user offices, and the location of each User Center within the building.

#### MATERIALS ANALYSIS USER CENTER (MAUC)

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

The major instruments located in the MAUC include

1. Physical Electronics Industries 660 scanning Auger microprobe (SAM),
2. Hitachi S-800 field emission scanning electron microscope (FE-SEM),
3. JEOL 733 electron microprobe,
4. Vacuum Generators ESCA/SIMSLAB-2 multitechnique surface analyzer,
5. JEOL 2000 FX analytical electron microscope, and
6. JEOL 4000 EX ultrahigh resolution transmission electron microscope.

#### MECHANICAL PROPERTIES USER CENTER (MPUC)

The MPUC is dedicated to the study of high-temperature mechanical performance of structural ceramics and alloys, including silicon carbides, silicon nitrides, aluminum oxide, and transformation toughened zirconia. Facilities are available for the measurement of strength and toughness as a function of time, temperature, and stressing conditions. In addition to standard flexure testing, the MPUC has twelve state-of-the-art tensile systems capable of evaluating the tensile strength to temperatures up to 1600°C.

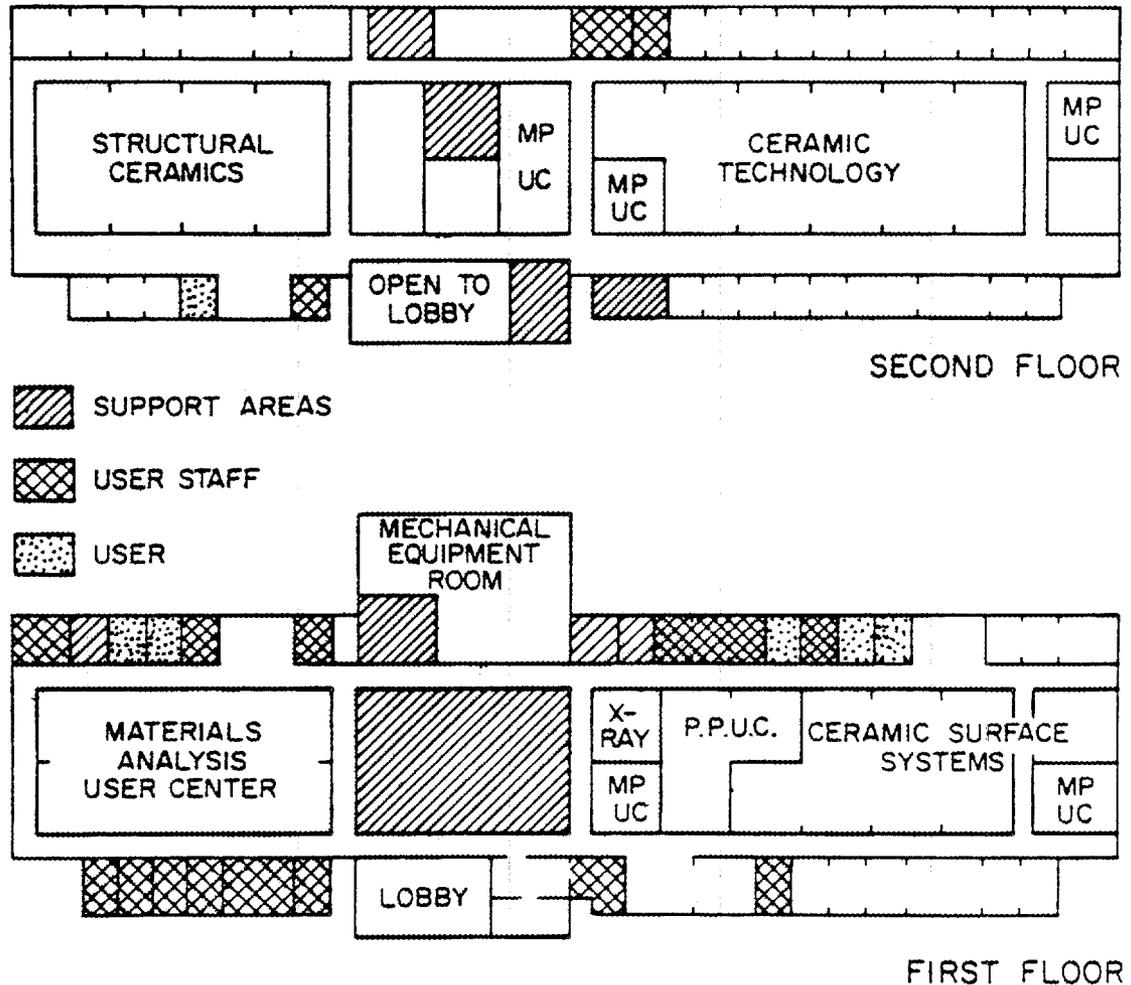


Fig. 1. HTML laboratory assignments. Office space which is not labeled is assigned to the three research groups and the Conservation Program staff.

Major equipment systems in this Center include

1. high-temperature tensile test facility,
2. ceramic flexure test facility,
3. high-temperature load deflectometer,
4. compression/tension uniaxial test system,
5. room temperature hydraulic tensile tester, and
6. mechanical properties microprobe.

#### PHYSICAL PROPERTIES USER CENTER (PPUC)

The PPUC is organized to include instruments for determination of a broad range of the physical properties of solid materials.

The major instruments located in the PPUC include

1. differential scanning calorimeter,
2. simultaneous differential and thermal gravimetric analysis system,
3. dual push rod dilatometer, and
4. thermal diffusivity apparatus.

#### **X-RAY DIFFRACTION USER CENTER (XRUC)**

X-ray diffraction is used to study 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 major instruments located in this Center include

1. Buehler XRD furnace,
2. Scintag theta-theta automated diffractometer,
3. Scintag theta-2theta automated diffractometer, and
4. Data General MV-2000 minicomputer,

#### **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 composition, function, and activities during the past year.

**Advisory Committee.** This committee is charged with the responsibility of advising on policy for operation of the User Centers. It is composed of six members who represent the industrial and academia community and the federal laboratories. The Committee meets at least annually and more frequently when its advice is urgently required on particular matters. A formal report is submitted to the Associate Director of Physical Sciences of ORNL.

**User Advisory Committee.** The major 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 Division staff, and the HTML Director. A more detailed description of the function and responsibilities of this committee is given in the brochure, *User Program for the High Temperature Materials Laboratory*.

This Committee met quarterly in FY 1988 (December 3, 1987; March 3, 1988; June 15, 1988; and September 14, 1988). Of 46 research proposals reviewed,

the Committee recommended rejection of one and approval of the rest. (Some were required to be revised, but approval was recommended once the revisions were made.)

*HTML User Exchange Group.* This group will be formed during FY 1989. A description of its function is given in the brochure, *User Program for the High Temperature Materials Laboratory.*

#### USER AGREEMENTS

The legal representatives of Oak Ridge Operations/Department of Energy and Martin Marietta Energy Systems, Inc., have approved three types of "Standard User Agreements" that become the binding contract between Energy Systems and the user institution once the appropriate agreement is signed by both parties.

The *Standard Proprietary Agreement* (SPA) is used when an institution wants to perform proprietary research in the HTML and will pay a DOE prescribed fee on a full-cost recovery basis. (To date, three agreements of this type are in effect. These research proposals are reviewed by a special user committee consisting of the HTML Director, a member of the Metals and Ceramics Division staff, and a representative from DOE.)

The *Standard Non-Proprietary Agreement* (SNPA) is used for research which is non-proprietary and the results will be available for public use. This research is generally free of charge to the user. (To date, 21 agreements of this type are in effect, including 9 with industrial firms and 12 with universities.)

The *Standard (State) Non-Proprietary Agreement* (SSNPA) is a modification of the SNPA. It has been amended to make it consistent with liability limitations imposed on most state institutions by their governments. With this new agreement, most schools who want to become a user are signing without going through a cycle of special approvals. (Five agreements of this type have been signed.)

Table 1 is a listing of the user agreements that were fully executed during this reporting period.

#### USERS

Figure 2 is a graph indicating the total number of user days including all industrial and university users per quarter for each of the five quarters covered by this report. The five quarters represented in Fig. 2 start with the third calendar quarter of CY 1987, and end with the third calendar quarter of CY 1988. This figure shows that the industrial component of user days has varied from about 30 to 90% during a particular quarter since the start of the user program.

Table 1. Standard user agreements executed July 15, 1987, through  
September 30, 1988

---

*Nonproprietary agreements - universities*

Alfred University  
 Auburn University  
 Clemson University  
 Dartmouth College  
 New Mexico Tech  
 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 and State Institute

*Proprietary agreements - universities*

Vanderbilt University

*Nonproprietary agreements - industry*

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

*Proprietary agreements - industry*

American Superconductor Corporation  
 Dow Corning (Wright)

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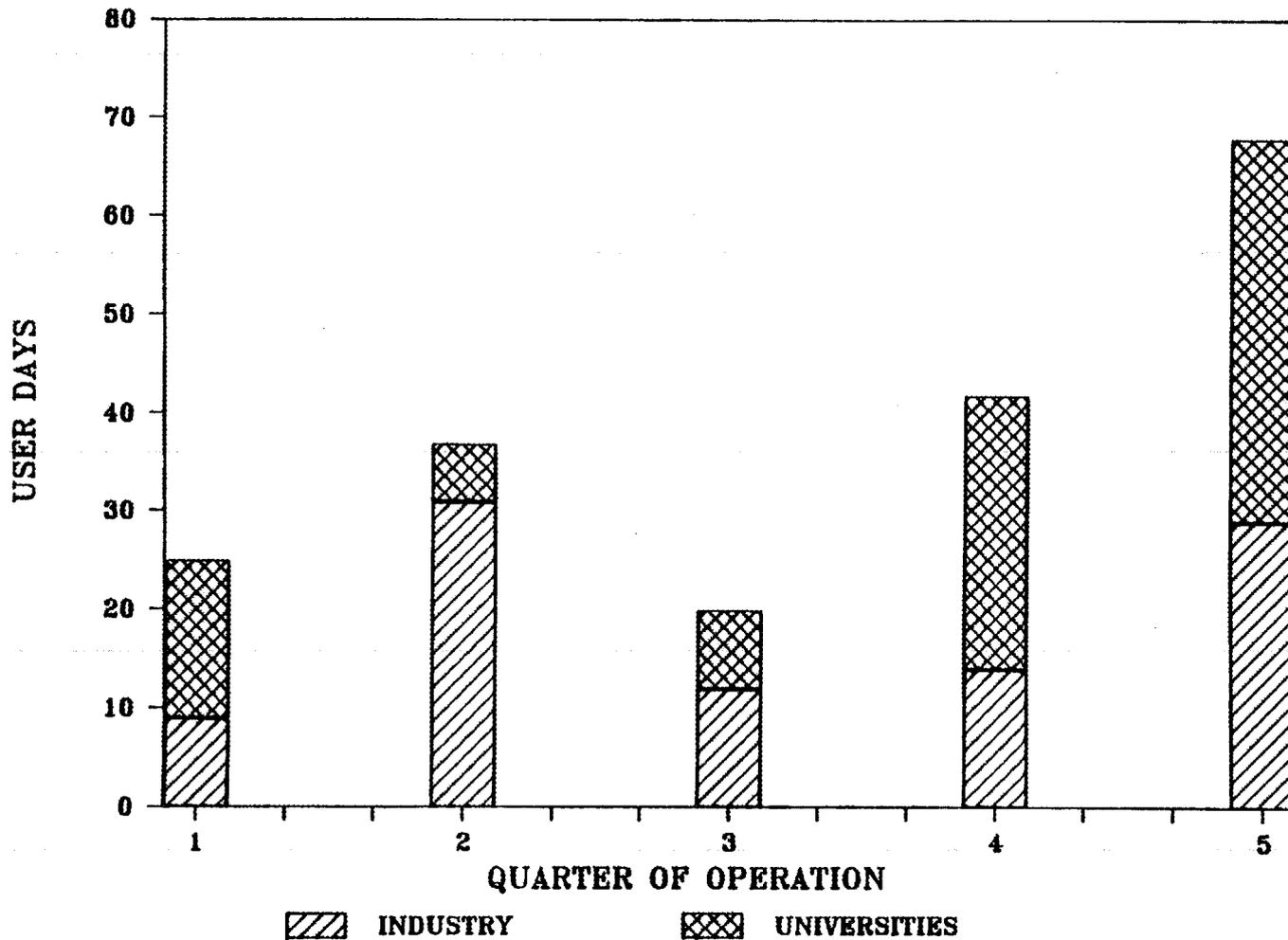


Fig. 2. HTML external user activities. The bar chart summarizes use of HTML user facilities by external researchers. The level of activity is expressed in user days and is shown on a quarterly basis beginning with the third quarter of CY 1987.

## EXTERNAL USERS

Figure 3 shows the cumulative user days by all industrial and university users in the HTML since the first agreement was signed on July 15, 1987. Appropriately on that day, both an industry (American Matrix Inc.) and a university (Oklahoma State University) agreement were signed. Figure 3 shows that the cumulative number of user days has been steadily increasing since the first quarter of operation. Figure 4 shows the distribution of user days in the Materials Analysis Center, which is representative of the experience to date. For the past two quarters, the number of user days is approximately 30, and it appears that this number will be increasing during the next quarter, based upon inquiries and proposals received. A short summary of the research performed to date by nonproprietary users is given here by user organization.

### Industrial Users

Allied Signal has had one research proposal approved. One user from Allied Signal spent 10 days in the Materials Analysis User Center to study the structure of coatings on SiC whiskers and their interactions with  $\text{Si}_3\text{N}_4$  in composite compositions.

American Matrix, Inc. has had three research proposals approved to date. Two proposals relate to the characterization of SiC whiskers in terms of morphology, microstructure, and surface chemistry. The third proposal involves measurement of thermal diffusivity and thermal expansion coefficients of  $\text{Al}_2\text{O}_3$ -SiC ceramic composites. Three users from American Matrix have spent a total of 57 days using equipment in both the PPUC and MAUC.

One researcher from Dow Corning (Midland) spent 3 days in the MPUC measuring the hardness of thin silica films deposited on a silicon substrate. The films ranged in thickness from about 85.0 to 550.0 nm. These measurements were done in an effort to correlate the hardness of the films with other properties such as density, refractive index, and film thickness, as well as various processing parameters.

Great Lakes Research Corporation has had one approved proposal to evaluate the effectiveness of barrier layers to light element diffusion. One researcher spent 5 days in the MAUC on this project.

Norton Company has had four proposals approved. During this reporting period, research was started on three of these proposals. One proposal included research to characterize the stress rupture behavior of  $\text{Si}_3\text{N}_4$ - $\text{Si}_3\text{N}_4$  joints developed for the DOE Ceramic Technology for Advanced Heat Engines project. One researcher spent 4 days in the MPUC conducting this research. Another researcher spent 5 days in the MPUC to characterize the high-temperature behavior of a hot isostatic pressed silicon nitride containing 4% of yttria as a sintering aid. In research related to a third proposal, one researcher spent 4 days in the XRDUC to study the anisotropic thermal expansions and thermal stability of yttrium disilicate and yttrium nitrogen apatite using high-temperature X-ray diffraction techniques.

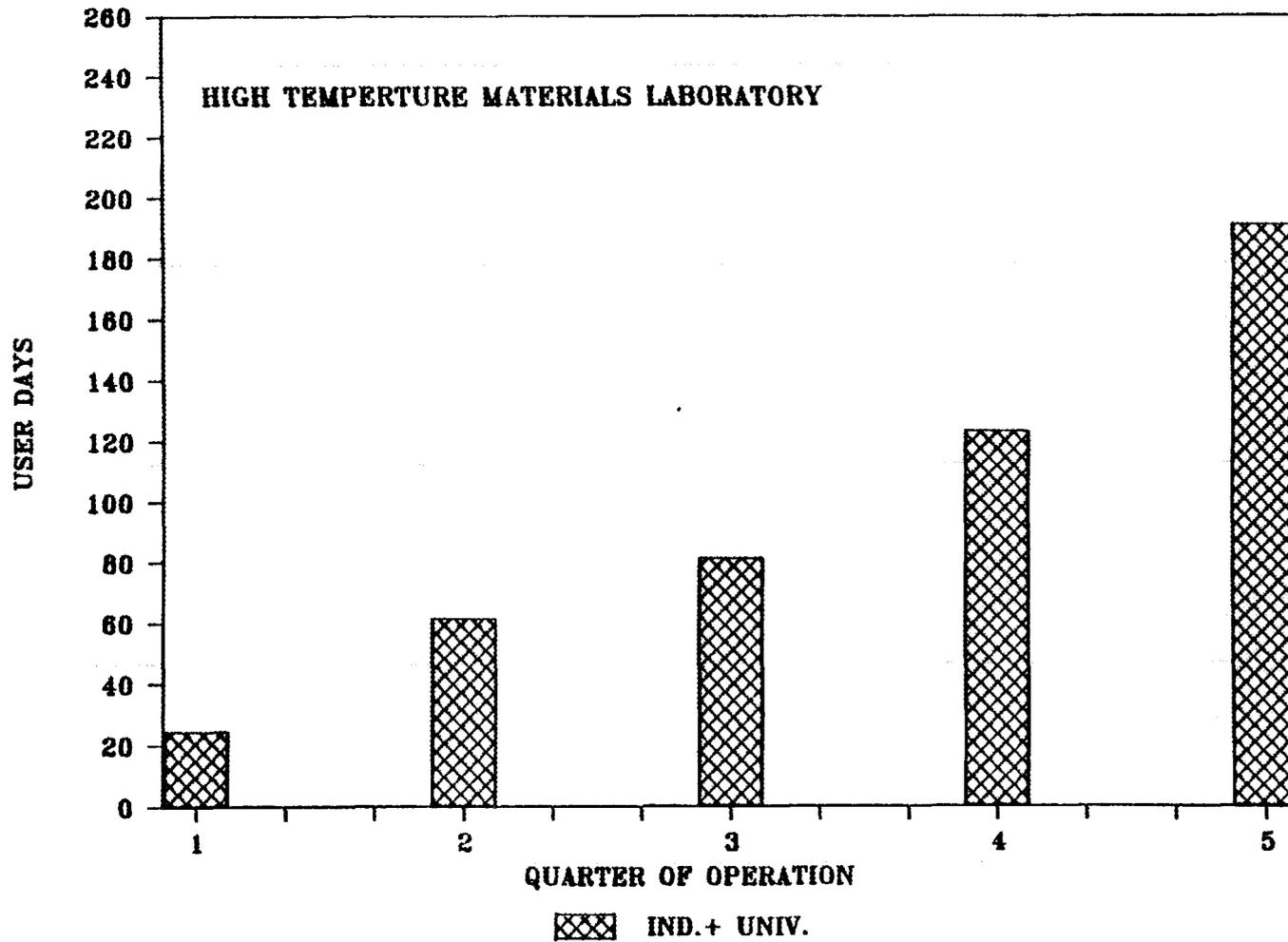


Fig. 3. Cumulative user days. The bar chart shows the cumulative increase of industrial and university user activities in the HTML by quarters beginning with the third quarter of CY 1987.

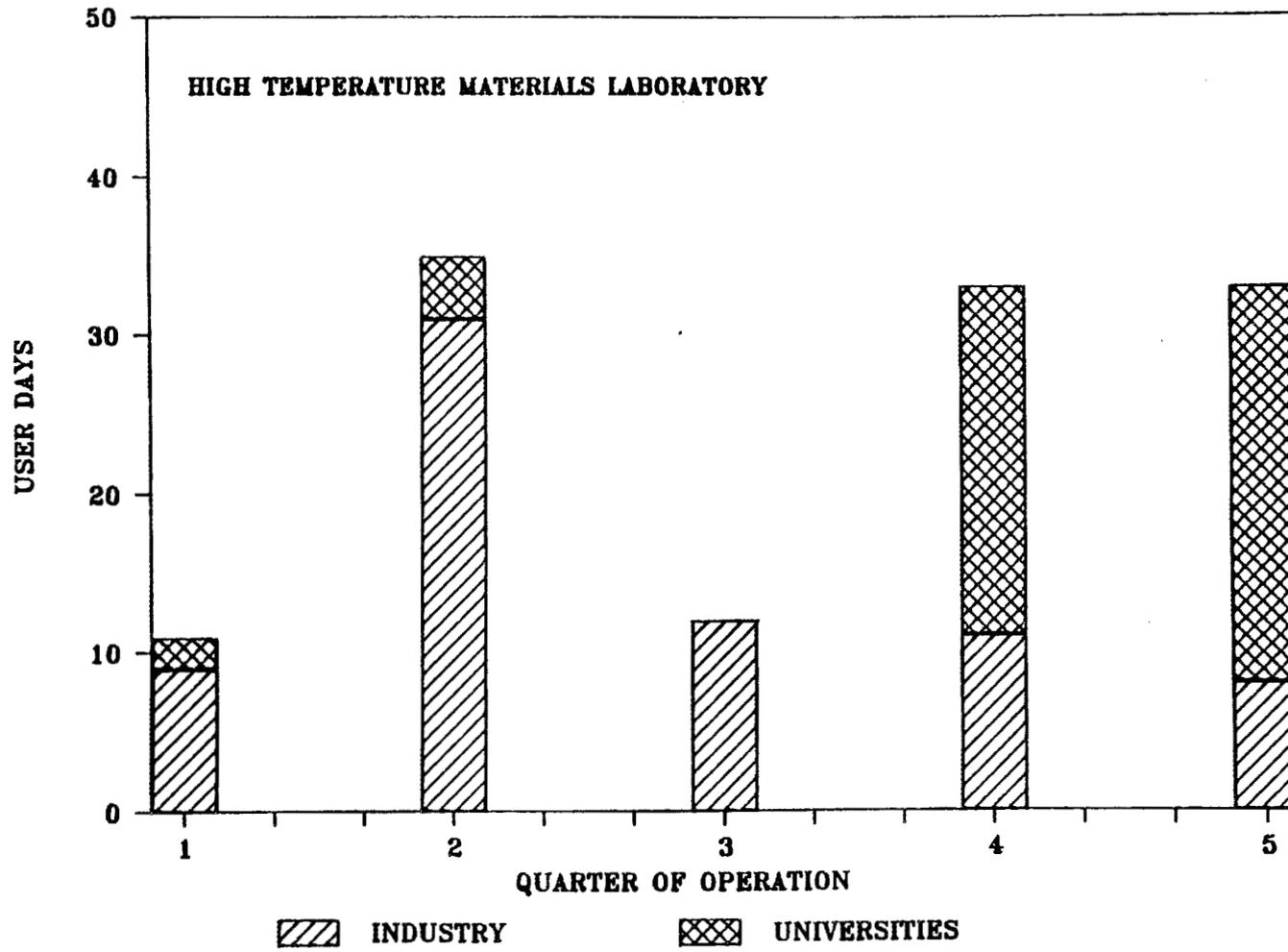


Fig. 4. Summary of industrial and university user activities in the Materials Analysis User Center of the HTML. Data were accumulated on a quarterly basis beginning with the third quarter of CY 1987.

Selee Corporation has had one proposal approved. One researcher spent 2 days in the XRUC to analyze phase changes which occur during firing of a commercial ceramic foam filter. The filters are used for filtering molten alloys during fabrication. They were failing due to precipitation of a second phase, which was identified during this research.

#### University Users

Alfred University has had one proposal approved. One researcher spent 4 days in the PPUC and 5 days in the XRUC. This research involved studying  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ , 1:3:2 -- a perovskite phase very similar to the 1:2:3 yttrium barium high-temperature superconductor -- in  $\text{H}_2$ ,  $\text{O}_2$ , and air at temperatures up to  $1050^\circ\text{C}$  in the X-ray diffraction furnace. A study of  $\text{LiFe}_5\text{O}_8$  in  $\text{O}_2$  at temperatures up to  $1000^\circ\text{C}$  was also initiated. Resolution of the high-temperature stability of  $\text{YBa}_2\text{Cu}_3\text{O}_{6.5}$  was investigated using equipment in the PPUC. Coupling data from both User Centers provided a complete understanding of 1:3:2 oxidation and high-temperature phase diagram. A summary of the work performed in the HTML was submitted to the HTML Director in early August; a publication which referenced the work at the HTML was received in September.

Dartmouth College has had one proposal approved. One user spent a total of 8 days in the MAUC studying grain boundary chemistry in FeAl and NiAl, which are advanced high-temperature alloys.

Oklahoma State University had one proposal approved. One user was here for 2 days in the MAUC studying etch channel defects in quartz crystals.

The University of Alabama has had one proposal approved. One user spent 3 days in the MAUC to characterize oil shale with framboidal pyrites and various coal samples.

The University of Illinois has four approved proposals. One researcher spent 5 days in the MPUC studying the effect of processing variables on the interfacial properties of SiC whiskers in a Li-Al silicate glass matrix. The Nano Indentor was used to evaluate the shear strength of the interface.

The University of Michigan has two approved proposals. One researcher spent 8 days in the MPUC to study the mechanical properties of ion-beam modified surfaces, using the mechanical properties microprobe.

The University of New Mexico has two approved proposals. One user spent 6 days in the MAUC to study small ceramic particles deposited on a carbon film. An abstract based on the research performed in the HTML has been submitted to the Materials Research Society for publication. A coauthor on this paper is an HTML staff member, L. F. Allard.

The University of Tennessee has four approved proposals. Research has been started on two of these during this report period. Three users have spent a total of 25 days in the MAUC performing research on these two proposals. One proposal was for determination of the microstructure on composition

samples from three tectonic units in the southwestern North Carolina Blue Ridge in order to establish the metamorphic P-T paths for each unit. Another proposal was to study ceramic-metal bonding.

Virginia Polytechnic Institute and State University submitted five proposals to date. One was rejected. One user spent 19 days using instruments in the XRDUC to perform characterization on ceramic materials with low thermal expansion. On a second proposal, three people spent a total of 8 days in the MPUC. They fractured five sets of alumina bars at room temperature using the electromechanical tester and analyzed the data.

#### INTERNAL USERS

Figure 5 indicates the number of user days of internal research in the four User Centers for each quarter. These users came from several divisions within ORNL as well as from three other Martin Marietta Energy Systems locations (Y-12 Plant, Oak Ridge Gaseous Diffusion Plant, and the Paducah Gaseous Diffusion Plant). Programs supporting this research included Conservation, Fossil, and Basic Energy Sciences.

#### PUBLICITY, AWARDS, AND PROMOTIONS OF THE HTML

The HTML was awarded high honors in the *R&D Magazine* Lab of the Year Contest. This event was recognized with a luncheon and presentation of plaques to the personnel involved. The ceremony was held at the HTML on May 6, 1988. Special guests included Mr. A. A. Chesnes, Director of the Heat Engine Propulsion Division of the Office of Transportation Systems; Mr. Clyde Hopkins, President of Martin Marietta Energy Systems, Inc.; and Mr. Joseph A. Lenhard, Assistant Manager, Energy Research and Development of Oak Ridge Operations/Department of Energy.

An exhibit which prominently features the HTML has been displayed at several major conferences in both the United States and other countries. Display locations included Travemunde, Germany, 1986 (2nd International Symposium, Ceramic Materials and Components for Heat Engines); the annual American Ceramic Society meetings in 1986, 1987, and 1988; and the Contractors' Coordination Meeting, Dearborn, Michigan, in 1986 and 1987. Handouts describing the HTML were distributed at each of these meetings.

#### SUMMARY

Construction of the HTML was completed in February 1987. Procurement and installation of all equipment for the four User Centers was completed in June 1988. The support staff for the User Centers were chosen and the anticipated level of staff is almost complete.

The first user agreements for both a university and industry were signed on July 15, 1987, and by September 30, 1988, there were a total of 29 fully

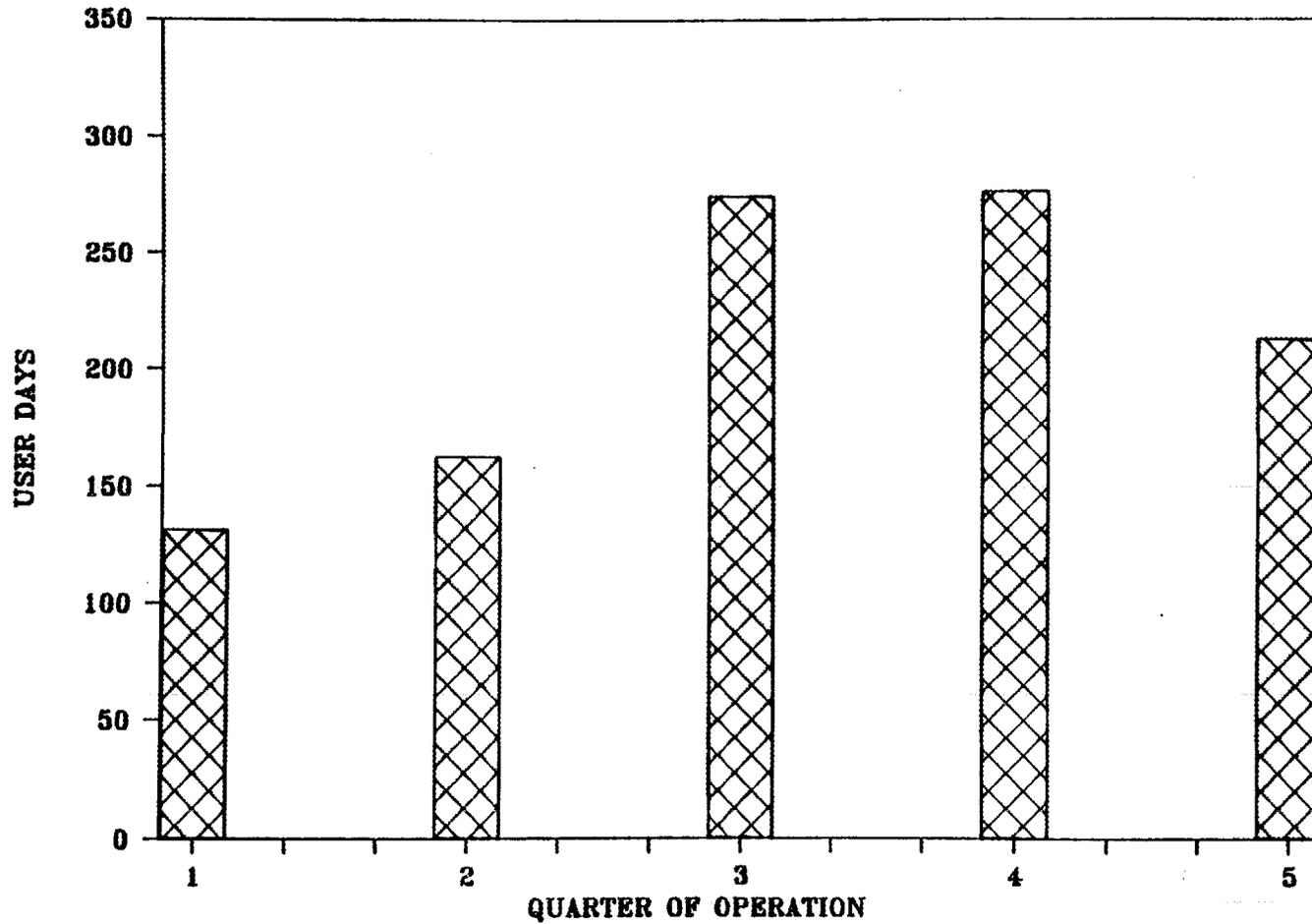


Fig. 5. HTML internal user activities. The bar chart summarizes use of HTML user facilities by internal researchers. The level of activity is expressed in user days and is shown on a quarterly basis beginning with the third quarter of CY 1987.

executed user agreements in effect (nonproprietary, 21; state nonproprietary, 5; and proprietary, 3). The User Advisory Committee met and reviewed 46 nonproprietary proposals and recommended approval of 45. There have been a total of 25 different individual users from industry and universities who have spent a total of 183 days (90 industry, 93 university) in the four user centers of the HTML. In addition, user days from within Martin Marietta Energy Systems have totaled 1088 (825 from Metals and Ceramics, 208 from other ORNL divisions, 55 from K-25, Y-12, and Paducah).

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