

**MULTIYEAR PROGRAM PLAN
FOR THE
HIGH TEMPERATURE MATERIALS
LABORATORY**

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EXECUTIVE SUMMARY

Recently, the U.S. Department of Energy's (DOE) Office of Heavy Vehicle Technologies (OHVT) prepared a Technology Roadmap describing the challenges facing development of higher fuel efficiency, less polluting sport utility vehicles, vans, and commercial trucks. Based on this roadmap, a multiyear program plan (MYPP) was also developed, in which approaches to solving the numerous challenges are enumerated (<http://www.ott.doe.gov/ohvt/>). Additional planning has been performed by DOE and national laboratory staff, on approaches to solving the numerous challenges faced by heavy vehicle system improvements. Workshops and planning documents have been developed concerning advanced aerodynamics, frictional and other parasitic losses, and thermal management. Similarly, the Heavy Vehicle Propulsion Materials Program has developed its own multiyear program plan. The High Temperature Materials Laboratory, a major user facility sponsored by OHVT, has now developed its program plan, described herein. Information was gathered via participation in the development of OHVT's overall Technology Roadmap and MYPP, through personal contacts within the materials-user community, and from attendance at conferences and expositions.

Major materials issues for the heavy vehicle industry currently center on trying to increase efficiency of (diesel) engines while at the same time reducing emissions (particularly NO_x and particulates). These requirements dictate the use of increasingly stronger, higher-temperature capable and more corrosion-resistant materials of construction, as well as advanced catalysts, particulate traps, and other pollution-control devices. Exhaust gas recirculation (EGR) is a technique which will certainly be applied to diesel engines in the near future, and its use represents a formidable challenge, as will be described later. Energy-efficient, low cost materials processing methods and surface treatments to improve wear, fracture, and corrosion resistance are also required.

The HTML focuses on such major national energy-related problems; specifically those where materials technology is a limiting factor. Increasing the efficiency of energy generation and energy utilization processes is one of the highest priority issues to which HTML applies its efforts. For instance, in the area of automotive and heavy vehicle propulsion, HTML works with engine manufacturers such as Caterpillar, Cummins, Detroit Diesel, Ford, and General Motors to advance the state-of-the-art in a wide range of engine materials, from alloys through metal- to ceramic-matrix composites and coatings. Similarly, in the development of advanced turbines for power generation, we are working with AlliedSignal Engines, Siemens-Westinghouse, Allison Engine Company, SOLAR Turbines, GE, and others to develop advanced ceramics, ceramic matrix composites, and ceramic thermal barrier coatings to allow these engines to function at higher temperature, thereby increasing their efficiency.

HTML also focuses its capabilities on the problems of emissions from power generation systems and other fuel burning devices, helping prevent the generation or release of harmful pollutants. We work directly with universities which are advancing the frontiers of catalyst research, as well as with the industrial developers and producers of such materials, like Allied Emission Catalyst, and the end users, such as Cummins Engine Company, Dow Chemical Company, Ford Motor Company, and many others.

Because funding for HTML comes from the Office of Heavy Vehicle Technologies, a part of the Office of Transportation Technologies, which is in turn part of the EERE branch of the Department of Energy, our work is prioritized to first assist the heavy vehicle industry. We have had numerous user projects with the diesel engine manufacturers and their various materials suppliers. HTML's next priority is to support users from the automotive industry, including the engine and vehicle manufacturers and all of their current and potential suppliers. For these transportation

industries, we have worked on catalyst development, wear-resistant or thermally-resistant coatings, stress-induced failures in materials, fuel cells, braking systems, lightweight materials, and a host of other topics with them.

Being a part of the EERE branch of DOE, HTML also supports the Office of Industrial Technologies (OIT), and to a lesser extent, the Office of Buildings Technologies and the Office of Power Generation technologies. The turbine engine work noted earlier, for example, is largely performed for OIT. HTML also supports other branches of DOE, including for example the Fossil Energy (FE) branch. We work with GE, Siemens-Westinghouse, and others to develop improved materials for power generation systems. Similarly, we have collaborated with other DOE national laboratories. Finally, as a national resource, we assist other government agencies, including NASA, NIST, the U.S. military, FHWA, FDA, and others.

In order to support these participants, HTML acts as a User Facility, by supplying access to state-of-the-art equipment, expert dedicated staff, and educational opportunities. HTML maintains facilities (the HTML building and associated laboratory and office spaces elsewhere); supports a staff consisting of permanent ORNL employees, along with post-docs, students, and contractor employees; and houses numerous sophisticated pieces of materials characterization equipment. To be effective, HTML requires an ongoing operating funding stream, as well as funding for updating old equipment and purchasing new equipment.

During the months of information gathering in preparation for this Multiyear Program Plan, it became apparent that HTML's sponsors and customers are asking for all of the current capabilities to be maintained, and in many cases upgraded (higher resolution, higher sensitivity, higher temperature capability, and remote operation). However, they have also indicated that, as a major focus of our efforts, HTML must concentrate on being able to determine material properties and performance characteristics under actual operating conditions, or at least as near to these as possible, via carefully simulated conditions. For example, many engine materials and exhaust aftertreatment devices must be exposed to engine operating conditions to evaluate their performance, but examining them AFTER operation requires formulation of theories as to what exactly is happening inside the engine or exhaust component. Users are asking now for examination DURING operation, so that the information obtained is first-hand and does not require the extra interpretation. One advantage of taking measurements during operation is that we can control the process variables and therefore can formulate models and develop better understanding of the processes involved. Therefore, BOTH testing conditions are complimentary and necessary. HTML has taken steps to develop capabilities that closely simulate many operating environments, and has developed data that is unique throughout the world. For example, we developed an "ex-situ" reactor which allows transmission electron microscopic evaluation of catalyst materials under simulated exhaust gas flow. We have developed mechanical test machines which allow simulated exhaust gas flow during testing, and have developed similar capability for x-ray and neutron measurement of crystal structures and residual stresses. However, these devices have been limited in their range of temperature, pressure, and gaseous environment generation capability, and we must expand these. We also are being requested to develop actual "in-situ" testing capabilities, which will involve collaborating with partners who have access to turbine, diesel, and gasoline engines, or fuel cells, etc. for which we can develop materials characterization equipment.

Within the transportation arena, the utilization of alternative (to petroleum-based) fuels is significant, as the preponderance of these petroleum based fuels are now imported. A major goal of DOE is to reduce this dependence on what are often politically unstable situations. Within OTT are numerous programs focusing on replacement of gasoline and diesel fuels with fuels, such as various alcohols, produced from biomass, and synthetic fuels, such as Fischer-Tropsch process fuels, that could be made essentially impurity (e.g.- sulfur) free. These fuels would burn more cleanly and thus remove major sources of pollution in the U.S., as well as reduce foreign

dependency. Natural gas and hydrogen are also contenders for utilization, as they are much cleaner-burning than petroleum fuels. However, each of these fuels poses potential problems, many of which are materials-related. Some of these fuels cause corrosion problems with current materials, while some produce new emissions, which will require new control systems. HTML is working with, and plans to continue working with, companies and universities in this area.

An example of the way that HTML will work on these problems follows. Natural gas has lower power density than gasoline, so it needs to be compressed and stored on-board to allow useful vehicle range. This poses explosion dangers and fueling problems. Ways are being sought to increase the amount of fuel stored without excessive pressurization. A recent innovation is the use of high porosity, high surface area carbon materials for natural gas storage. HTML proposes to assist the further development of these materials through application of neutron and x-ray scattering techniques, which promise to allow definition of the location, and perhaps, absorption/desorption mechanism of the methane molecule. Currently, we utilize the High Flux Isotope Reactor (HFIR) at ORNL, which provides us with one of the highest flux beamlines in the world. However, this reactor beamline will be superseded by the Spallation Neutron Source (SNS) when it comes on-line in 2006. We will collaborate with the SNS project to enable us to take advantage of its increased power (shorter experiment time, higher resolution capability). This collaboration will require increased funding resources.

Another area in which the HTML will expand capabilities is that of remote instrument operation. We have for three years now been leading the world in development of capability to operate our sophisticated analytical instruments from remote locations, initially as part of the DOE2000 Program sponsored by DOE's Office of Science, MICS branch. This project, called the Materials Characterization Collaboratory (MCC), was one of only two Pilot Projects chosen through a nationwide competitive process. We have developed the necessary interface software, hardware, and operating procedures to allow operations of our scanning and transmission electron microscopes, electron microprobe, mechanical test frames, and neutron diffraction instruments. This operation was initially a demonstration conducted by our staff from remote locations, but it has since blossomed into actual use for User Projects, by universities and industry (Ford Motor Company and Dow Chemical Company). The savings in time and travel costs that it represents will be expanded to ALL of our instruments. This expansion will require a boost in our operating and capital resources.

This document details the HTML's multiyear program plan to address the above issues. The total request, for both operating and capital equipment funding, is provided in Table 1. We anticipate operational support requirements for the six existing User Centers for the years FY2000 through FY2004 to increase from the FY2000 level of 5.0M\$ at 5% per year as depicted in Table 2. There are three step-function increases also requested; one is for \$200k in FY2001 for operation of the NSLS X14A synchrotron beamline shared by DUC and RSUC. The second, for \$250k, occurs in FY2002, to provide funds to accelerate activities into nondestructive examination through specialized imaging techniques. The third, starting in FY2003, adds \$1M as \$750k for capital and \$250k for staff the first year, and \$500k for each of the next years. This funding is to support our major new initiative to take advantage of the Spallation Neutron Source. Justification for these requests are provided in the following text and appendices. Capital equipment funding requirements are also depicted, in Tables 3 - 7, in which it will be noted that the requested amounts vary considerably from year to year. Reasons for this are described in the Appendices.

Table 1. Operating and capital equipment funds requested.

Fiscal Year	Operating Funds (k\$)	Capital* Funds (\$k)	Total Request (\$k)
2000	5000	3315	8315
2001	5450	2080	7530
2002	5973	1915	7888
2003	6521	2310	9581
2004	7085	2125	9710
TOTALS	30029	11745	41774

* Tables 3 – 7 detail the instruments to be funded in each fiscal year.

Table 2. Operating funds requested.

Fiscal Year	Baseline* Funds(\$k)	New** Request(\$k)	New*** Request(\$k)	New**** Request(\$k)	Total Request (\$k)
2000	5000	0	0	0	5000
2001	5250	200	0	0	5450
2002	5513	210	250	0	5973
2003	5788	221	262	250	6521
2004	6078	232	276	500	7085
TOTALS	27628	862	788	750	30029

* Increased at 5% per year to offset effects of inflation and partially accommodate increased demands for user project staff time.

** 200 \$k for sponsorship of NSLS beamline, inflation-adjusted.

*** Costs to develop new Imaging (NDE) User Center.

**** Costs to participate in instrument development for SNS beamline.

Table 3
FY2000 Capitalization Plan

User Center	Equipment Item	\$k	Justification
MAUC	ACEM	3000	This microscope will provide for sub-angstrom resolution capability, which doesn't currently exist in the national laboratory system at accelerating voltages less than about 1 MeV. This capability will allow location of atoms in critical materials like catalysts.
MCAUC	Pancake furnace for Instron 1380 for temperatures to 1800°C	30	Often there is a need to perform testing at 1800°C in air. None of the old furnaces can get close to this temperature.
RSUC	Focusing monochromators for NRSF - single reflection, two xtal systems - XY and rotation table with encoders	85	Study completed and specification being prepared. Gains of 2-6X in figure-of-merit. Critical for aluminum (6x) alloys and other non ferrous alloys. Essential to be in hand by January 2001 to be ready to commission new stress mapping with >10x gains.
TPUC	High Temp (1650 C) DSC	80	Accurate measurement of heat capacity, heat of transformation and melting temperature. Data needed for process modeling and phase equilibria studies. Replace 12 year old out-of-date system that is poorly supported by vendor and has limited temp. range which is inadequate for many steels. Specific heat accuracy is low and limited to 1000 °C and is very time consuming. Critical parts take a year to obtain.
DUC	Capillary furnace for X14A with gas flow and monitoring options	50	X14A has become heavily used for high resolution XRD for lattice and structure analysis. Critical need for high temperature and simulated environment studies. Gas flow and monitoring required.
MIRUC	Portable instrumentation package - development and prototype unit.	70	Develop and construct a prototype of a Labview-based portable instrumentation package for measuring grinding parameters in real time. This package is a key element to our strategic plan involving the leasing of expensive machine tools rather than outright purchase. The portable system would be designed so that it can be easily interfaced with any grinder/controller, and then removed for re-use at the end of the equipment lease.
FY2000 TOTAL=			3315

Table 4. FY2001 Capitalization Plan

Center	Equipment Item	\$k	Justification
MCAUC	Expansion of mechanical/environmental testing capabilities. Steam pressure, combustion environment.	250	This aligns MCAUC with not only the missions of OHVT but also important work in the Microturbine and Reciprocal Engines program of OIT.
MCAUC	Upgrade 4 Flexure test units to Labview software	150	The flexure units are still in high demand from users and other programs. Upgrading the computers and DAC systems is necessary before the old system is non-operational (PC 486-machines).
DUC	Low temperature attachment for XRD systems, closed cycle temperature control	60	User Forum request. Need is to study materials of use in transportation systems at all temps that might be encountered in service. Useful for actuator and other functional ceramics, low CTE materials, hydration studies.
DUC	Sample changer for furnace at X14A with gas flow and monitoring options	50	X14A is heavily used for high resolution XRD for lattice and structure analysis. Critical need for high temperature and simulated environment studies. Sample changer greatly expands utilization and remote collaboration.
DUC	BSKP - ESEM with Noran System	500	An environmental SEM for phase identification, grain by grain. Will be needed by users and DOE programs such as fuel cell membranes, batteries, catalysts, refractories, etc. Requires adequate size specimen chamber and XYZ table. Addresses consignment status of Noran unit. Put on dedicated SEM w/EDAX but moderate resolution.
RSUC	Rotating anode generator, anodes, hutch, optical table; 2-axis horiz. goniometer	300	Replace existing and increasingly expensive to maintain rotating anode generator. Use existing PTS goniometer and add second to gain productivity and flexibility. PTS/Rotating anode is heavily used unit: will be 10 years old by FY01.
RSUC	Bruker 2D detector for PTS/tube	90	For major enhancement in speed and accuracy of stress and texture measurements. Makes unit state-of-the-art. Valuable to User Program, functional materials, biomaterials, batteries, nanomaterials, thin films,
RSUC	Radial collimator attachment	75	Radial collimators enable small sampling volumes while achieving greater distance between slit and sample that is required for larger components and mapping. Well within estimate based on LANL purchase - expandable in future.
TPUC	Bench top mass spectrometer	50	Current 12 year old MS is dead and repairs are not promising. Been dead for 6 months and prospective proposals are being turned away. Essential component for thermal analysis by STA. Portability means we can move to the high mass TG, to the dilatometer, and most importantly to the HTXRD and HTNPD systems. This will open new opportunities and contribute to our uniqueness in HT diffraction and functional materials synthesis.
TPUC	Oxygen partial pressure control system for dilatometer and diffraction system	50	Growing calls for work on functional ceramics such as fuel cell membranes and sensor materials properties. Valuable also for Ti alloy characterization.
TPUC	Modulated DSC (low temp range, to complement HT DSC)	80	Part of planned package to update thermal analysis portion of TPUC. Wide use based on current user activity. Very important for characterization of Al and Mg alloys. Also for studies of amorphous metals and polymers.
TPUC	Large format IR Camera	225	Current camera is over subscribed and aging. New camera technology will expand types of property characterization and non destructive imaging. Supports User Forum requests for enhanced imaging capabilities for both "machine vision" process modeling as well as defect detection in a wide range of materials. Also needed for rolling safety inspection and the aging aircraft and aging infrastructure NDI programs.
MIRUC	Begin equipment leasing program. Program is expected to require up to \$200K annually once the initial leases are in place.	200	The outright purchase of machining and inspection equipment is an enormously expensive undertaking, with the average cost of a new machine being over \$200K. Such equipment would not be instrumented and would necessitate additional purchases of sensors and data collection equipment. A better option would be to establish favorable leasing arrangements with leading machine tool builders whereby state-of-the-art equipment is leased and replaced with new models at the end of the lease. The feasibility of such a program will be investigated in FY 2000. Ideally, the leasing obligation would be no more than \$50K per year for each machine involved. This would allow us to keep our most important pieces of equipment from becoming obsolete.
FY2001 TOTAL=			2080

Table 5.
FY2002 Capitalization Plan

Fiscal Year	User Center	Equipment Item	\$k	Justification
2002	MAUC	CCD Camera for HF-2000 2k X 2K, ultrafast	100	Replacement camera for HF-2000 with fast readout for higher resolution and near real time holography phase images.
	MAUC	Hot Stage for HF-2000	60	Item in support of development of nanostructured materials, for the direct observation of the refinement of structure with temperature and interaction/formation of phases.
	MCAUC	New Nanoindenter Or serious repairs to old one	150	There is a need to plan for upgrade of the Nanoindenter. This equipment is being used for thousands of hours per year, and user demand for percise capabilities remain.
	MCAUC	Continue to upgrade controllers for the Instrons 1380 at 30 k each	180	Assuming our experience remains good with the new controllers we would like to continue to upgrade these
	MCAUC	Continue to retrofit Furnaces for Instrons 1380	90	This is an on-going need - our users expect that the equipment will function reliably at the temperatures we need
	DUC	High flux, high temp x-ray diffraction system	300	Rotating anode theta-theta goniometer with state-of-the-art furnace attachment, detectors, and control system. 6-8x increase speed needed for in-situ, real-time studies. Functional materials, nanomaterials, sensors, catalysts....
	DUC	Ceramic stage environmental furnace for XRD studies with improved gas flow control and monitoring with two+ color pyrometer for accurate temp	95	Ceramic stage and radiant heating can provide highly uniform heating and accurate control of temperature. Supports the most heavily used instrument in DUC. Need CO/CO2 control of oxygen partial pressure for studies of fuel cell and other functional ceramic materials.
	DUC	Upgrade of control systems at X14A to integrate real time in situ accessories and beam line control	50	The change of wavelength is very labor intensive and requires up to 36 hours of meticulous alignments done in a series of small changes in wavelength to avoid losing the beam. Upgrade could reduce time substantially and enable users to remotely request a change at night or weekends when beam likely not manned.
	RSUC-Xray	Detectors for PTS units and for X14A for stress analysis	60	Detectors have a finite life time. Cost of ownership item.
	RSUC-Xray	Load frames for torsional and compressive applied stress	60	x-ray stress attachment, none exist but consistent with the "environmental" thrust of HTML.
	RSUC-NRSF	Load frame for tensile, compression, sheer conditions Furnace attachment for studies to 1000 C Cyclic load capability desired - cost ??? For use at HFIR and SNS - possible cost share with SNS	125	Increasing demand for simulation studies of applied loads. Valuable also for fundamental materials behavior studies needed to develop accurate understanding for deformation modeling arising from anisotropic behavior with crystal plane (hkl). Studies of macro stain at elevated temps and applied load are of wide interest in Recommend for "environmental" thrust of HTML
	RSUC-NRSF	Specimen surface mapping and high precision alignment for NRSF	75	Alignment of samples for NRSF work to be accomplished offline, thereby saving neutron time and improving accuracy of mapping coordinates.
	TPUC	STA with direct line os sight MS (Netzch)	250	Current STA will be 15 years old and not unique nor maintainable. Expand capabilities for gas flow and monitoring. Greater sensitivity, detect condensable evolved gases. Inert gas sheathed gas flow needed for corrosive environment studies. Move to NT based computer for remote collaboration and use.
	TPUC	Upgrade of LFTD system (cost of ownership)	90	Improved accuracy and reliability. Higher throughput. Windows NT computer control provides remote collaboration and use.
	TPUC	Hyperspectral attachment for IR Camera	75	Provides capability to image at selected wavelengths as well as to detect chemical chemical species such as NOx and SOx at 15 ppm levels.
	MIRUC	Non-contact laser surface profilometer	120	The Rodenstock laser surface profilometer no longer represents state-of-the-art technology and needs to be upgraded/replaced with a more reliable unit.
	MIRUC	Upgrade Mahr Formtester Inspection Instrument	35	The computer and related software used for instrument positioning/alignment control and data analysis are based on an obsolete DOS computer. The existing computer/controller needs to be upgraded to a Windows system with additional analysis and control capabilities.
FY2002 TOTAL=			1915	

Table 6.
FY2003 Capitalization Plan

Fiscal Year	User Center	Equipment Item	\$k	Justification
2003	MAUC	Advanced high-speed computing capabilities	200	Array processors and infrastructure (public-key encryption) in support of live time remote microscopy for non- and proprietary users. Preparation of infrastructure for real time simulations of high-res images from the ACEM
	MAUC	Ex-situ Reactor with Specimen Holder for ACEM	60	Item will give reaction capabilities for ACEM. It will use better technology for temperature control and better materials of construction.
	MAUC	New Imaging X-ray photoelectron spectrometer with enviro/hot chamber	950	This instrument allows the mapping of binding energy states. Thus we could map the storage of oxygen in ceria particles in diesel oxidation and automotive particles in diesel oxidation and automotive catalysts. This is one example of the power of this instrument. Determination of the composition of surface glazes that poison catalysts is another.
	MCAUC	High Temperature Indentation System Vickers and Hetzian Indentation	150	There is a user demand for this capability. More work on high temperature properties of coatings is ongoing, this would be a very valuable tool for this.
	MCAUC	Drop Impact tester	150	Will add an important and unique new capability
	DUC	High throughput RT XRD system	150	Sealed beam tube, graded mirror, sample rotation in sample changer providing 7/24 operation. Intelligent automated data processing and preliminary analysis.
	DUC	Upgrade of detectors and beam optics at X14A to integrate real time in situ accessories and beam line control	50	State of the art detector and diffracted beam optics will further enable real-time, simulated environmental studies.
	RSUC-Xray	HT furnace for use on PTS and X14A - Be dome furnace for 4-axis goniometer use - to 1200 or 1300 C for oxidation stress studies	150	In situ studies growing in demand and technological value. Ideal for oxidation stress, thin films, surface treatments and membrane, ionic conductor, and other functional matl.
	TPUC	Viscosity measurement system for Al and Mg alloys (collaborate with Srinath V. on purchase?)	150	Viscosity is the one property we can't measure that is needed for casting modeling. Many requests by potential users and funded programs.
	TPUC	Containerless melt system	300	Meets demand for viscosity of HT melting alloys for casting modeling. Thermal physical characterization of need but currently not available.
FY2003 TOTAL=			2310	

**Table 7.
FY2004 Capitalization Plan**

Fiscal Year	User Center	Equipment Item	\$k	Justification
2004	MAUC	Dedicated STEM	1400	Needed to provide greater chemical sensitivity to specific imaged atoms. Provide several orders of magnitude greater chemical sensitivity than TEM.
	MCAUC	"Grindosonic" for High Temperature	100	Users have need for knowledge of elastic modulus at elevated temperatures.
	MCAUC	Indentation system for "In-Between" loads	150	A system in an intermediate load range would meet needs in several areas
	DUC	Enhanced in-situ, real-time neutron diffraction furnace (for use at both HFIR and SNS)	100	Quartz lamp furnace for rapid heating and cooling. Wide range of atmosphere to simulate processing and operating environments. Gas flow control and monitoring essential for studies of fuel cell materials, battery materials, H2 storage materials, etc.
	RSUC-xray	Large specimen stress analyzer	275	Large specimen stress measurements are required for many transportation and other industrial system components.
	MIRUC	Continue with leasing program	100	
		FY2004 TOTAL=	2125	

1.0 INTRODUCTION

This document is a detailed description of the multiyear plan for the HTML for the years FY2000 – FY2004. HTML is part of the Metals and Ceramics Division of the Oak Ridge National Laboratory. The MYPP was developed by the Director, with considerable input from the user center leaders and the HTML staff. HTML's mission is in accordance with that of both the Division and the Laboratory. Programmatic guidance was provided by the cognizant program manager from the Department of Energy, Dr. Sidney Diamond.

The document represents a snapshot in time, since the development of strategic plans is an ongoing, continuously evolving process. The multiyear plan will be updated periodically, to represent changes in plan necessitated by the changing needs of our customers. It is expected to be updated approximately biannually.

The Multiyear Program Plan is arranged so as to give the reader a description of the planning process, a summary of the facility and its programs, a brief up-to-date status report, a definition of HTML's customers' needs for materials characterization, and plans and strategies for satisfying these customer needs.

2.0 STRATEGIC PLANNING PROCESS

The approach taken on strategic planning is from a text on Total Quality Management [REF 1], with the conviction that HTML represents a quality organization, providing its customers a more than satisfactory experience. This philosophy is aligned with that of the DOE [REF 2]. Strategic planning is being performed through the following steps:

- (1) The Director and his user center and staff met several times to define and refine the mission and vision statements.
- (2) These same people, taking their direction from these two statements, have developed a set of goals which define the targets that HTML must attain to satisfy the vision.
- (3) User Center Leaders then will meet with their staffs to develop a set of tasks which represent the actions required to attain the goals.
- (4) The Director and his staff will meet to reconcile these objectives and tasks, and initiate the required actions.
- (5) Information gathering from HTML's customers and other stakeholders has been continuously performed, through personal visits with industrial and academic personnel, attendance and participation in conferences and expositions, and especially, through a Workshop held in Knoxville, Tennessee in August of 1999, specifically for the purpose of developing this type of information.

2.1 Mission and Vision Statements

The mission of the HTML is captured succinctly in the following statement:

MISSION STATEMENT OF THE HTML

The mission of the High Temperature Materials Laboratory is to help engineers, scientists, and students in U. S. - based organizations solve materials problems of interest to the U. S. Department of Energy, and advance materials science and technology. We do this by providing users access to state-of-the-art characterization tools, expertise, and educational opportunities.

HTML'S strategic intent is described in the vision statement:

VISION STATEMENT FOR THE HTML

The High Temperature Materials Laboratory will maintain a state-of-the-art facility for characterizing advanced materials of interest to our customers, staffed with the highest caliber engineers, scientists, and support staff, and equipped with the best instruments available.

Everything done by the HTML Director and HTML's staff is, and will continue to be, guided by these two statements. All decisions regarding facility space plans, staffing, selection of users and fellows, purchase of equipment and supplies, and addition of new capability or initiation of new centers, are first tested for suitability by determining whether they comply with the mission and vision.

2.2 Goals, Objectives, and Tasks

Five goals were enumerated as those targets which, when attained, will lead the HTML to the vision of providing the best possible service to our customers. They are:

- (1) Maintain the best facility.
- (2) Maintain the highest caliber staff.
- (3) Equip the facility with the best instrumentation and equipment.
- (4) Obtain secure funding.
- (5) Respond to the customers' needs.

Objectives and tasks for the first four goals are extremely important matters of internal policy, but will not be discussed in this document. The fifth goal involves discerning what HTML's customers' needs are, and will be in the future, and the determination of means of satisfying these needs. Plans for attaining goal five will comprise the bulk of this planning document, and necessarily involve the first four goals.

3.0 CUSTOMER NEEDS ANALYSIS

3.1 National Materials Challenges

The major non-nuclear weapons national challenges that the U. S. Department of Energy is focussing its attention upon consist of energy generation and energy utilization. Elements of these topics include the drive for improved energy efficiency, the use of alternative fuels or power sources (e.g.- solar or biomass-derived power), and reduced emissions. These technologies are most often limited by available materials: that is, advanced materials can be seen as “enabling” progress in energy technologies. DOE has long recognized that, and has made materials R&D a large component of its efforts.

The HTML and its programs are an outcome of that thinking, being designed to assist DOE accomplish its missions by helping U.S.-based industry and academia. Accordingly, HTML sees as its first priority those projects that relate directly to our nation’s energy situation, supporting first those relating to the Energy Efficiency/Renewable Energy (EERE) branch of DOE. We also support the other DOE branches, including especially Fossil Energy (FE). Within EERE, HTML supports first those projects related to transportation, since our support comes directly through the Office of Transportation Technology. We focus on heavy vehicle materials problems to support the Office of Heavy Vehicle Technologies’ mission, as well as the mission of the Office of Advanced Automotive Technologies. However, there are numerous materials needs in other EERE offices, including the Office of Industrial Technology (OIT), which HTML supports. This has become especially true as that office has developed, with industry, the Industries of the Future concept, in which industrial groups have determined their “ideal” factories of the future, and are defining the materials problems hindering them from attaining these ideals. We have studied the “Vision” documents of these industries to glean from them their major needs.

Keeping this mission-support role in mind, information gathering for this document focused on the stakeholders of these DOE offices.

3.2 Summary of Information Gathering, Including Workshops

A significant number of research projects performed in the User Centers has been associated with ceramic material structure, advanced alloy structure, thermophysical properties, and residual stress studies of a wide range of materials. In addition, a broad range of other materials problems has been successfully addressed using the facilities and expertise in the program including: high-temperature alloy thermophysical properties, stability, processing; catalyst formation, stability, and thermophysical properties; superconducting materials processing; whisker and continuous fiber ceramic and metal matrix composites; and carbon based materials and composites.

A small but significant number of projects has involved special bio-materials, including ceramic oxides and carbon based materials, such as pyrocarbons. These extensive working relationships have provided an excellent opportunity to identify additional capabilities which, if available, could substantially help U.S. companies and university researchers address and solve additional important problems which will lead to development and commercialization of additional new materials in the economy. Many strong relationships with U.S. companies have been formed as a result of hundreds of user relationships in the past twelve years. Thus, there is a strong base for recognizing and proposing expansion of facilities and expertise in significant and important new capabilities needed in the next five years.

Below are listed examples of companies who have made major use of the HTML and most have had multiple user projects.

Diesel engine components: Cummins Engine, Detroit Diesel, and Caterpillar

Automotive components: General Motors and Ford

Land based and aerospace turbine systems: AlliedSignal, General Electric Aircraft, Siemens-Westinghouse, and United Technologies Corp.

Ceramic materials manufacturers: Dow Chemical, Norton, Coors, Ceramtec, DuPont/Lanxide, and Ceradyne

Alloy suppliers: Teledyne, Tosoh, United Technology Corp., General Electric Corp.

“Industries of the Future”: the chemical, glass, steel, aluminum, casting, heat-treating, mining, agriculture, forest products industry groups

We are currently capable of meeting these customer needs and are doing so, as described in Section 4.0. We are, however, approaching a level of use in some user centers such that demands on either equipment or staff time are causing the time from approval of a user project to its actual performance to become too long for our customers' desires. Remediating this situation will require either additional equipment or staff. Also, in some cases, our equipment is becoming outdated, and replacement will be required. Finally, some customers are asking for emerging techniques and equipment which we do not currently possess, and we are considering the addition of this equipment or capability, and the need for new user centers. **Especially worthy of note is the fact that many companies now desire evaluation of their materials in environments that are more representative of their actual operating environments, rather than the laboratory air, vacuum, or inert gas that we can typically provide. There is an increased awareness that synergistic effects exist between stress, temperature, and environment that cannot be determined separately. Thus we are endeavoring to provide more realistic environments during materials testing, and have, for instance, added gas control systems where possible to instruments like diffractometers, microscopes, and mechanical test frames. We can simulate some environments this way. However, there is a further need for testing in “actual” operating environments, especially for engines and their aftertreatment components. In this regard, we are planning means of adding this capability to HTML’s repertoire, including partnering with the Propulsion Systems Center (in the Engineering Technology Division of ORNL), with the National Transportation Research Center (NTRC) being built in Oak Ridge, and with engine or component manufacturers. We are also planning the consignment or purchase of engines to provide this environmental capability. To support the Industries of the Future, we are partnering with the Corrosion Science and Technology Group of the Metals and Ceramics Division of ORNL. This group has the expertise and equipment to develop atmospheres that can simulate most industrial processes.**

Experience with the current six user centers has been carefully considered and efforts have been pursued to identify and project the needs of HTML customers during the next five years. Also taken into account are the material and application oriented focus areas of major importance to OTT, EERE, and DOE in general for this same time period. The focus areas are identified and expanded upon in the following section.

The August 1999 Workshop sponsored by HTML helped define the current thoughts on materials characterization needs. The following parts of Section 3 detail our findings, enumerated by technology end-user.

3.2.1. Diesel Engine Components

With the recent past's fuel pricing and availability, heavy vehicle owners have not been pushing strongly on the diesel engine manufacturers to increase efficiency (fuel economy). This is likely to change, with the recent upsurge in fuel prices. However, there is a strong push for lower emissions in large trucks, in particular for lower levels of NO_x and particulates. This comes largely from the U.S. Environmental Protection Agency (EPA), which sets limits for emissions.

There has been recent tremendous growth of the sport utility vehicle (SUV) and pickup truck markets, such that last year there actually were more pickups, vans, and SUVs produced and sold in the U.S. than cars. These vehicles typically exhibit poor fuel mileage, largely because of their increased weight, poorer aerodynamics, and use as towing/hauling vehicles. It is widely recognized that the fuel efficiency of these vehicles could be dramatically increased (by as much as 35-40%) by the introduction of diesel engines, which typically can offer efficiencies of over 40%, whereas the best gasoline engines run at about 25-35%. However, the high levels of NO_x and particulates produced by even the cleanest burning diesels, compared to gasoline engines, is inhibiting their use. Thus, the diesel engine companies have great incentive to produce cleaner engines.

With the push from EPA, and the potential market pull of engine sales into pickups, vans, and SUVs, the diesel engine companies are showing tremendous effort at emission reduction. Work is ongoing in the three major areas of emissions: fuel formulation, combustion, and aftertreatment. Materials research plays a role in all of these areas, albeit a minor role at the moment in fuel formulation. In combustion, materials play a major role in fuel introduction (fuel injection systems), fuel burning (use of thermal barrier coatings (TBCs) to prevent excessive heat damage to pistons, firedecks, valves, etc.), and turbocharging/intercooling applications. Higher in-cylinder pressures are being contemplated, with resultant increased loads and temperatures on all materials of construction, from the block to the heads to the pistons, rods, and crankshafts. In aftertreatment, materials are being scrutinized for use in catalysts, particulate traps, and exhaust gas recycle (EGR) components. EGR is expected to be increasingly utilized, and that will bring with it increased soot levels in the oil (abrasion, erosion), and increased nitrogen- and sulfur- oxide levels (acidic constituents) in the oil, which will cause additional corrosion. EGR will also cause potential corrosion problems in the intake manifold/intercooling system, where condensation may occur. Finally, EGR will also increase exhaust temperature, causing increased corrosion, but especially increased thermal fatigue, in components such as heads and exhaust manifolds.

HTML has seen, from the diesel engine companies, an increase in projects related to fuel injection components, wear and corrosion-resistant coatings and other materials, and stronger materials of construction. We also have experienced greater interest in aftertreatment materials, especially catalysts and particulate traps, and in fact have recently undertaken a characterization of the micro- to nano-structure and chemistry of the particulates themselves. Many projects have been carried out in MAUC, MCAUC, and RSUC in response.

For an excellent summary of the materials needs for diesel-powered vehicles, see the Office of Heavy Vehicle's Technology Roadmap, and their Multiyear Program Plan (available as Adobe Acrobat "pdf" files on the Web from the OHVT at <http://www.ott.doe.gov/ohvt/>).

3.2.2 Automotive Components

The major initiatives being carried out by the automotive manufacturers and their suppliers revolve around the Partnership for a New Generation Vehicle (PNGV), which is a national effort being sponsored largely by the U.S. Departments of Commerce and Energy. These efforts involve

reducing the weight of a full-sized, five-passenger vehicle, increasing its propulsive energy source efficiency, and decreasing its parasitic losses (aerodynamic drag, running resistance, etc.) so that it can attain 80 miles per gallon fuel efficiency without sacrificing customer comfort and safety.

HTML is working with the automotive companies and their suppliers in numerous areas, running from lightweight materials to gas turbine and fuel cell engine concepts to catalysts for emissions control. Lightweight materials include aluminum, magnesium, composites and superstrong structures, with the new challenges posed by strengthening, fabricating, joining, and protecting them. Welding processes and their resultant effects on residual stress and thus performance of the welded components are being heavily evaluated. Some of the engine concepts under consideration include small diesel/battery hybrids, which have the same emission challenges as noted in Section 3.2.1. Transportation fuel cell development poses its own unique problems, especially with regard to making fuel cells lower cost, lighter weight, and higher power density, as well as more accepting of current standard fuels.

Documents pertaining to the materials requirements for PNGV are readily found on the OTT Website, under OAAT (Office of Advanced Automotive Technologies: <http://www.ott.doe.gov/oaat/>).

3.2.3 Land Based and Aerospace Turbine Systems

In order to increase the efficiency of a gas turbine, operation at higher inlet gas stream temperature is a must, and this involves application of either advanced high temperature materials or application of advanced cooling systems. The latter involve system losses to power the necessary pumps, and so are not as effective. Thus, for all sizes of turbine, from the large land based power generation machines of several hundred megawatts, to the local power generation aeroderivative machines of a few megawatts, on down through building-power-sized machines and automotive engines of a few hundred kilowatts, efforts on developing and applying advanced materials continue. Typically, gas inlet temperatures are high enough (looking for up to 1400°C) that only ceramics or ceramic matrix composites are being considered.

Most of the programs are now sponsored by DOE's Office of Industrial Technologies, and details of their programs can be found at <http://www.oit.doe.gov/>, and at <http://www.oit.doe.gov/cogen/>, and at <http://www.oit.doe.gov/cfcc/>. Other turbine materials programs are being sponsored by the DOE Fossil Energy Program, whose projects are described at their website: <http://www.fe.doe.gov/>.

3.2.4 Industries of the Future

The Office of Industrial Technologies recently commissioned a study, performed by the United States Advanced Ceramics Association and ORNL, of the ceramic materials needs of the Industries of the Future Program. The report was published in December 1998 ("Opportunities for Advanced Ceramics to Meet the Needs of the Industries of the Future", D. W. Freitag and D. W. Richerson, DOE/ORO 2076, December, 1998). Ceramics needs for the chemical, forest products, steel, glass, aluminum, and metalcasting industries are addressed. Each industry chapter ends with a table showing the application, industry needs, and opportunities for ceramics.

4.0 EMERGING CUSTOMER NEEDS

4.1 Structural ceramics

The drive to reduce the cost of structural ceramic components will increase in intensity, as these products compete to replace metal components in many applications. Many applications have been demonstrated where wide scale application of these materials is severely hindered by current costs. These costs include two major components, the cost of the ceramic powders and the cost of machining the ceramic form to the final required dimensions and tolerances. A major capability in the HTML suite includes the ability to precisely machine ceramic surfaces and then characterize their topography and residual stress and relate these properties to the fracture statistics and wear properties of the surface. This integrated capability is especially attractive to companies trying to either manufacture or utilize these materials. The HTML is unique in the United States in having this ability at the current time. The MIRUC provides unequalled capability in the United States for working with industry in determining the optimum surface grinding conditions for various structural ceramics by coupling the actual grinding research directly with the ability in the MAUC and the RSUC to characterize both the microstructure of the materials and the residual stress generated by the machining process. Current efforts typically focus on attaining the highest possible machining speed, to lower costs, while still retaining the precision required of the finished component.

4.2 High-temperature alloy development

Advanced alloys of many types have been studied as part of user projects in the HTML user program. These alloys have belonged to two main classes, one being gamma-prime strengthened high nickel superalloys for gas turbine applications, while the other includes specialty alloys high in titanium used in bio-implants. The majority of the user projects have involved the high nickel content alloys. One of the needs defined recently has been for higher temperature capability, for cast products (power turbine blades and vanes), rolled alloys (sheet for use in heat exchangers, catalyst supports), and other applications where long lifetime at high temperature is required.

A second major need is for oxidation and/or corrosion resistant alloys. As noted above, the Industries of the Future projects in OIT range from steel to glass to aluminum and others, all of which require use of high temperature processes, commonly including some corrosive chemical or gaseous species. Improved alloys and coatings are a necessity.

4.3 Ceramic & metal matrix composites

This technology area has included a number of user projects, mainly concerned with continuous fiber ceramic matrix composites and ceramic reinforced aluminum and other metal matrix composites. The requests for experimental capabilities associated with ceramic matrix composites were initially associated with room temperature mechanical properties of both the composites and the fiber reinforcements themselves. Relatively rapidly growing interest areas in composite systems include fiber/matrix interface characterization plus general microstructural characterization, and especially measurements at elevated temperatures. The CFCC program has supported, within HTML, the construction of a number of load frames with furnaces which have the ability to expose composites to stress and temperature with a gas flowing.

Considerable growth in terms of User activity, DOE sponsored research [Continuous Fiber Ceramic Composites (CFCC), for example], and CRADAs has occurred. Industry examples include Babcock & Wilcox, Dupont, 3M Company, and AlliedSignal.

4.4 Lightweight materials for transportation and aerospace

Examples of interest in this area include United States Automotive Materials Partnership, CRADAs, User Projects, diesel engine manufacturers, and metals producers, especially the aluminum and magnesium producers.

4.5 Materials for advanced power generation and conversion devices

Materials for special energy intensive applications and energy conversion are studied, with major effort on battery and fuel cell materials. Examples include United States Advanced Battery Consortium, CRADAs, and User Projects

4.6 Thick and thin films

Thermal barrier coatings (TBC) for use in both gas turbine and reciprocating engines (mainly diesels) are clearly going to be a major area of technological effort in the United States in the next five years. These coatings are necessary to provide the proper heat management for the engine. TBC characterization is in a highly empirical state, and must be established on a more quantitative basis in order to allow orderly and economical evolution of these materials for the next generation of applications. The relation of processing to coating stability, thermal transport through the coating, and its microstructure are vital areas in which the HTML is prepared to play a major national role. The mechanical behavior of these coatings is also of major importance, but no method currently exists for quantitatively measuring the coating stability on the metal substrate or its thermal conductance capabilities as a function of thermal cycling history. However, HTML has been working extensively with industrial and academic users/fellows on these aspects of TBCs. Companies participating have included GE Aircraft Engines, Siemens-Westinghouse, and Pratt and Whitney, Cummins Engine, and Caterpillar, while academic participation has been widespread, including the State University of New York at Stonybrook, University of Alabama-Birmingham, and the University of California-Santa Barbara.

There are several other types of coatings which have been the basis of past HTML user projects, and in which there is clearly still major industrial interest and need. Most of these coating interests fall into one of three major areas. The first is coatings applied generally to tungsten carbide based cutting tools or a similar substrate. These coatings greatly reduce interaction of the cutting tool and the metal workpiece during very high energy density cutting or turning machining operations, and are of major technological and economic importance to the machine tool and metal manufacturing industries. The HTML program has made major contributions in this area via user projects. The second area involves so called wear coatings. These materials are typically applied to metal substrates for the purpose of greatly increasing the ability of the underlying metal to resist wear, usually in rubbing or rolling applications, such as in shafts and bearings. Considerable HTML activity has focused on this type of material and the prognosis is that in the next five years this activity will increase substantially if suitable testing systems, which can simulate the wear conditions experienced in engineering systems of interest, are made available to industrial and university users.

Wear resistant coatings (all types of engine and manufacturing operations) are evolving into major industrial activity. An understanding of principles is still very empirical, but we have an excellent opportunity for working with many small to medium sized companies who focus on this very specialized business segment to help these companies produce coatings having improved durability and therefore better economics.

Examples of companies with interest in these areas include General Motors, Ford, Cincinnati-Milacron/Valenite, Detroit Diesel, Cummins Engine, Caterpillar, Eaton, Norton/St. Gobain, AlliedSignal, General Electric Aircraft, Solar Turbines, United Technologies, and Teledyne Allvac.

The third area of thin film and coating research is in “functional” films, e.g.- those that do something more than provide protection. These include thin film catalysts, various type of membranes, micro-electromechanical (MEMs) devices, and electronics. HTML has developed capabilities to measure various properties and characteristics of these films and coatings, including nanoindentation for hardness, modulus, and, recently, toughness. The microstructural and microchemical characterization capabilities of our SEMs, TEMs, scanning Auger microprobe, and electron microprobe have been utilized frequently. Stresses built up in coatings and films often have great influence on properties, function, and lifetime, and these are measured by our grazing incidence x-ray diffraction technique. We have been adding, and will continue to add, capability to more closely represent actual operating conditions of temperature and gas atmosphere. For example, recent catalytic process or exhaust streams, and then these specimens can be inserted directly into the TEM without compromising the results by exposure to lab air.

4.7 Manufacturing and fabrication process modeling for alloys

Goals of major U.S. manufacturers and DOE include more efficient development of engineering properties to meet design objectives; reliable processing with greater yield; and a major reduction of cost and time from drawings to production. The impact will be major in reduced costs, additional new materials in the market place, and therefore, increased U.S. competitiveness.

This is a nationally identified major research area of the next five years. DOE and the National Science Foundation (NSF) are also directing significant efforts into this area at this time. At ORNL several CRADAs (NCMS, GMRC) with USCAR affiliated companies and associations are in place in this area, with a significant amount of the work being done in the HTML by local researchers.

ASM international committees and member industries (Caterpillar, GM) have documented the extensive need for thermophysical, thermodynamic, and thermomechanical property data to accelerate the development and implementation of new materials.

Stakeholders for this area include companies involved with forging, casting, welding, and heat treating of various alloy products. This is especially true for the Metalcasting and Heat-Treating portions of the OIT Industries of the Future Program.

4.8 Environmental and thermal cyclic studies of ceramics and alloys

Large scale use of advanced materials in high temperature engineering systems, such as fuel cells, recuperators, regenerators, engine valves, gas turbine components such as the combustor, stators, and rotors, demand long term data on the material properties in the applicable environment. In its first seven years, the HTML was pivotal in helping U.S. industry establish a national ceramic tensile strength standard, a national standard for tensile creep, and several supplemental studies which were critical to the development of a life prediction model for silicon nitride ceramics. These would not be in place today without this program. The next step in the evolution of these and similar materials was to extend the experimental exposure times from hundreds of hours to thousands of hours. In the current economic environment, this cannot be done by the companies themselves. The DOE has played a key role in the past in assisting these companies in various ways to develop new or improved materials to the point where they can be successfully implemented in real energy generating or conversion systems. The HTML has accommodated these needs, participating, for example, in a long-term study of the stability and strength of silicon nitride structural ceramics for gas turbine use. SOLAR Turbines, the University of Dayton

Research Institute, and ORNL/HTML all purchased identical test apparatus and performed a round-robin evaluation of a standard material to assure interchangeability of data, then together performed a matrix of test exposures for up to 10,000 hours.

The next challenge is to perform more simulative evaluations:

- Oxidation, corrosion, and their effects on key mechanical and physical properties of the materials of choice for major system components is a key area. Exposure systems are needed for proper simulation of long term service applications, up to 5000 h or more. Examples include: (1) silicon nitride creep in selected atmospheres in the temperature range of 1100 to 1350°C and at static and maximum cyclic stresses up to 300 MPa and (2) thermal barrier coating bond strength, thermal transmission, and structure as a function of temperature, thermal cycling, thermal gradient, and time for conditions of, 800 to 1200°C, 10^6 cycles, between 8 and 20×10^6 °C/m, and up to 5000 h, respectively. To simulate the corrosion effects of a gas turbine exhaust, high flow rates of simulated exhaust gases containing significant partial pressures of water vapor are required. Additional complications arise with the need for high pressures in these exhaust streams.
- A high integrity database for key properties is required for component life prediction. In addition, proven micromechanical models are essential for timely and reliable insertion of new, advanced materials into applications such as diesel and automotive engines and advanced turbine systems (land based or aircraft). Currently, the models which can use such data, examples of real engineering comparisons, and materials data required to apply in such models are woefully inadequate. The experience of the past eleven years in the HTML program and in appropriate ASTM Committees indicates that very few or no current U.S. companies are prepared to take the risk and obtain such data for their developing materials without some type of assistance. The HTML user program provides an ideal vehicle for such partnering with industry.
- HTML's role in this case is to provide research instrumentation capabilities and expert staff to facilitate industry testing and evaluating materials prior to simulated and actual engine testing, and then provide characterization resources for comparing the behavior of materials tested in laboratory simulation environments and actual engine environments. The instrumentation and staff can be highly efficient tools for helping industrial researchers both for screening materials and developing improved materials to withstand the anticipated oxidation, corrosion, thermal fatigue, thermal stress, and materials compatibility requirements of new heat engine systems. Such a capability would also serve the university research community as a major educational tool for developing engineers. No similar user facility currently exists in the United States.

4.9 Rapid ceramic microstructure engineering

Due to the interatomic bonding and the crystal structures inherent in most ceramic materials, it is much more difficult to develop and control the microstructure during the manufacturing process than is the case for common metals based upon iron or aluminum. Since the microstructure determines the final physical and mechanical properties of the material, it is necessary to have some major advancements in the processes which industry uses to develop and qualify a material for large scale engineering applications. The HTML can play a major role in such a revolutionary development as a partner with industry. Sophisticated process engineering is essential to develop and quickly learn how to control and consistently manufacture the desired microstructure that is the foundation of advanced ceramic materials properties.

The current development cycle for learning how to first generate and then to control the requisite microstructure in production is typically one or two decades long. This is far too long to be

relevant in the present economic climate. What is needed to remain competitive in the global market is a breakthrough that takes material ideas/requirements and rapidly and cost effectively explores the microstructure-properties-performance envelope which can yield a candidate process ready for pilot plant testing in a few months.

Like the successful specialty U.S. steel industry, specialty ceramics will be the high value ceramics industry in the next decade, and the engine for new job creation based on ceramic materials. Without a specialty ceramic industry, the U.S. capability to maintain a competitive high volume structural ceramics industry will quickly erode, similar to what has already occurred in the electronic ceramic industry. Applications are very broad including wear components, liquid metal filters, electronic ceramics, piezoelectric materials, catalysts, valves, cutting tools, coatings, etc. A thrust in this area would support both small and start up companies in addition to large corporations.

This is an area for a partnership with both universities and industries, requiring a long view of the future. The vision here is very broad and should be approached on the basis of a major long term partnership of major interested government agencies and industry. For a major thrust, a NSF/DOE/University/Industry partnership is proposed. New rapid high-temperature characterization tools which can be applied during key processing or simulated processing steps in the flow sheet using X-ray diffraction and other probes must be central to this endeavor. The development of new instrumentation to meet these demands will be required.

4.10 New Longer Term Needs (> 5 yrs)

One of the major new technologies that will be affecting materials is "nanomaterials: the science of materials at the atomic and molecular scale. The President has just announced, in his FY 2001 budget request, a 227M\$ (84%) increase over this year's funding level for nanotechnology R&D. It is called the "National Nanotechnology Initiative (NNI)", and the President envisions "materials by design" from the bottom up, multi-terabit computer memory the size of a sugar cube, materials ten times the strength of steel at a fraction of the weight, and super-high-speed computers. Nanomaterials challenges for the HTML will involve extremely high resolution imaging (SEMs, TEMs) with high sensitivity/high resolution microchemical analysis (Auger, microprobe, ESCA, etc.) Similarly, for mechanical properties, extremely fine and stable nanoindentors will be required. Investigation into nanostructures will require high intensity, small spot size beams of x-rays, which are generated by synchrotrons. HTML will need to continue its role as a beamline partner in the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory, and continue to work with research centers at other synchrotron sites (APS, for example, where other ORNL staff have beamlines).

Another major emerging technology is biomaterials. In an announcement similar to the President's statement on nanotechnology (above), he requested for FY2001 an **increase** of 1B\$ for biomedical research at NIH. While most of that is for research on specific diseases (diabetes, brain disorders, cancer, etc.) some will involve materials for e.g.- prostheses, drug delivery systems, and materials for medical devices. HTML's biomedical customers will demand additional capability for in-vitro testing, such as friction and wear measurements in bodily fluids, and increased emphasis on surface characterization.

The third major technology that is mushrooming is information technology. The President's FY2001 budget request asks for a more than \$600M increase for this field. Most of the emphasis will be on high speed/high capacity networking, for e.g.- distance learning and telemedicine. However, it will be extremely beneficial to HTML's push into "remote operation". The devices being developed for these computer systems and networks will be smaller and will involve new materials, with challenges to the HTML in specimen preparation, and needs for high resolution and sensitivity.

5.0 CURRENT STATUS OF THE HTML, ITS PROGRAMS AND CENTERS

This section will present the current makeup of the HTML and its User Program, with descriptions of the organization, the user centers, and the modes of operation. A detailed description each User Center's staff and instrument capabilities is given in Appendix A. The reader can keep up-to-date on the HTML's staff, facilities, and capabilities by accessing the World Wide Web homepage at <http://www.ms.ornl.gov/htmlhome>.

The HTML facility is a building of 64,500 square feet capacity, on two floors, with offices arranged on the outside surrounding laboratory spaces clustered in the interior. The user center staff offices and laboratories are co-located, as possible. Staff offices contain sites and furniture for the user and fellowship guests.

The staff are part of the Materials Analysis and Characterization Section, which is itself broken into groups. The groups contain within them the user centers. Descriptions of the user centers are given in the following paragraphs. The Section Manager, who doubles as the HTML Program Manager, is also the Director of the HTML. A senior advisory committee assists in the successful operation of the HTML User Program.

5.1 HTML User Program

The User Program is Directed by Dr. Arvid Pasto, and his Programs and Marketing Assistant is Ms. Billie Russell, who is supported by Ms. Shirley North. A brief description of the operation of this program and its current status follows.

Research projects start as proposals submitted by the potential user, and these, when approved, provide the user access to the requested HTML instruments needed to perform the work. User projects typically include research to relate materials properties to structure or to manufacturing processes, or to train users and provide them access to the equipment necessary to perform their own materials research.

Early in the User Program, user agreements were developed that established the intellectual property and liability rights of the user institution and Martin Marietta Energy Systems, Inc. The first user agreement was signed on July 15, 1987; since that time, several hundred agreements have been executed.

Two types of standard agreements are used: a "Nonproprietary Agreement" and a "Proprietary Agreement." The nonproprietary user agreement requires that users — along with HTML technical staff — jointly publish the results of their research within six months of completing the user project. Proprietary agreements do not require users to publish with HTML staff.

As of the end of 1998, a total of 444 nonproprietary agreements (257 industry, 171 university, and 16 other governmental agency) and numerous proprietary agreements were in effect. These had resulted in 819 distinct user projects. During FY1999, the HTML User Program evaluated 87 nonproprietary proposals (39 from industry, 45 from universities, and 3 from other government facilities) and four proprietary proposals. Complete details of these projects are available annually in the HTML Annual Reports, and thus will not be given here.

The needs for equipment and capability for the User Program are determined through this continuous interaction with users, as well as from periodic workshops and other assemblies at or sponsored by the HTML (For example, an International Workshop on Electron Holography was sponsored in Knoxville, TN. This meeting was a highly successful gathering of nearly 30 invited speakers (20 from Europe and Japan) and approximately 70 other researchers from around the

world. The workshop highlighted materials characterization using electron holography.) The HTML also holds "User Forums" roughly biannually, at which users and staff exchange information on needs and capabilities. One was held in July of 1997, for instance, during the celebration of HTML's 10th Anniversary as a User Facility. A special User Forum was held in Knoxville TN in August of 1999 for the key purpose of eliciting from a broad user base the needs of HTML's customers for materials characterization in the near future. The input derived therefrom is a strong driver of this MYPP. HTML staff also attend numerous conventions and conferences to present the results of their work, and gain valuable insights there. Also, many companies and universities are visited during the course of user projects, and HTML is in turn visited by many current and prospective users. For example, in 1998 approximately 70 briefings and tours of HTML facilities were conducted. Finally, HTML staff spend about half of their effort supporting programs other than the HTML User Program, including the Heavy Vehicle Propulsion Materials Program of OHVT, and the Continuous Fiber Ceramic Composite Program and the Advanced Turbine Systems Program of the Office of Industrial Technologies. Interactions with industrial and other researchers through these programs also yield information on materials characterization needs.

5.2 Current User Centers

This section very briefly describes the thrust areas of each user center. Greater detail is provided on staff and equipment available in each User Center in Appendix A.

5.2.1 Materials Analysis User Center (MAUC)

The Materials Analysis User Center (MAUC) uses electron microscopy and surface chemical analysis techniques to characterize the structure and chemistry of advanced structural materials. The information obtained from these characterizations is used to elucidate the mechanisms that control material performance.

5.2.2 Mechanical Characterization and Analysis User Center (MCAUC)

The Mechanical Characterization and Analysis User Center (MCAUC) specializes in the mechanical characterization of structural materials, including high-temperature materials. MCAUC performs mechanical testing and analysis, develops test methods and supplemental analytical techniques, and conducts finite-element and life-prediction analyses of monolithic ceramics, ceramic composites, and ceramic coatings. Numerous mechanical test frames are available to visiting researchers from industry and academia. Capabilities to conduct tension, compression, flexure, shear, and micromechanical tests in controlled environments and at elevated temperatures on standard and customized specimens. Staff has expertise with a wide range of materials, mechanical test designs, finite-element and analytical modeling, life-prediction analysis, statistical design, and failure analysis.

5.2.3 Thermophysical Properties User Center (TPUC)

The Thermophysical Properties User Center (TPUC) is dedicated to measuring thermophysical properties as a function of temperature and correlating these properties with the processing, microstructure, and performance of materials. Specifically, the TPUC staff works with users to determine thermophysical properties such as thermal diffusivity, thermal conductivity, specific heat, and thermal expansion and to characterize the thermal stability, high-temperature reactions and compatibility, and high-temperature oxidation and corrosion properties of materials. The materials studied include structural ceramics, engineering alloys, ceramic and metal matrix

composites, superconducting materials, ceramic precursors, carbon materials, and carbon fiber composites.

5.2.4 Residual Stress User Center (RSUC)

User projects and DOE programs are increasingly concerned with life prediction and failure analysis of engineering structures. In many of these cases, knowledge of residual stress gradients (sign and magnitude), as a function of location at both the surface and throughout the volume of a component is critical information for failure analysis and life prediction models. The Residual Stress User Center (RSUC) was established to meet this need and to provide a facility for research into controlling residual stresses, either through modifying the forming and finishing processes, by changes in the design, or through stress-relief procedures. RSUC activities, now include both the X-ray residual stress facility, the neutron residual stress facility (discussed in detail in Sect. 4.4.3), and the developing use of synchrotron radiation and Raman microprobe. The diffraction facilities can be utilized to measure macro (long-range) and micro (short-range) residual stresses in polycrystalline materials. Raman microprobe is currently focused on alumina.

RSUC also characterizes the non-random grain distribution or texture in materials and relates this to directionally dependent materials properties. Texture is very common in materials subject to deformation and also in thin films, both areas of increasing importance.

5.2.5 Diffraction User Center (DUC)

The Diffraction User Center (DUC) uses room- and high-temperature X-ray and neutron diffraction methods to characterize crystalline phase(s) and stability of advanced structural ceramics, alloys, catalysts, and other industrially relevant materials. The data, obtained individually as a function of temperature and environment and frequently in conjunction with data from thermal analysis or electron microscopy, are used to relate materials processing and performance with phase transformations, reactions (solid-solid, liquid-solid, and gas-solid), lattice expansion, atomic structure, crystallization from the melt, and phase stability.

In addition to supporting users' diffraction needs, the XRD facilities are extensively used by qualified staff in the Metals and Ceramics Division, who are conducting a wide variety of ceramic and alloy R&D efforts. DUC also provides technical expertise in diffraction and materials science in support of a number of DOE-funded projects.

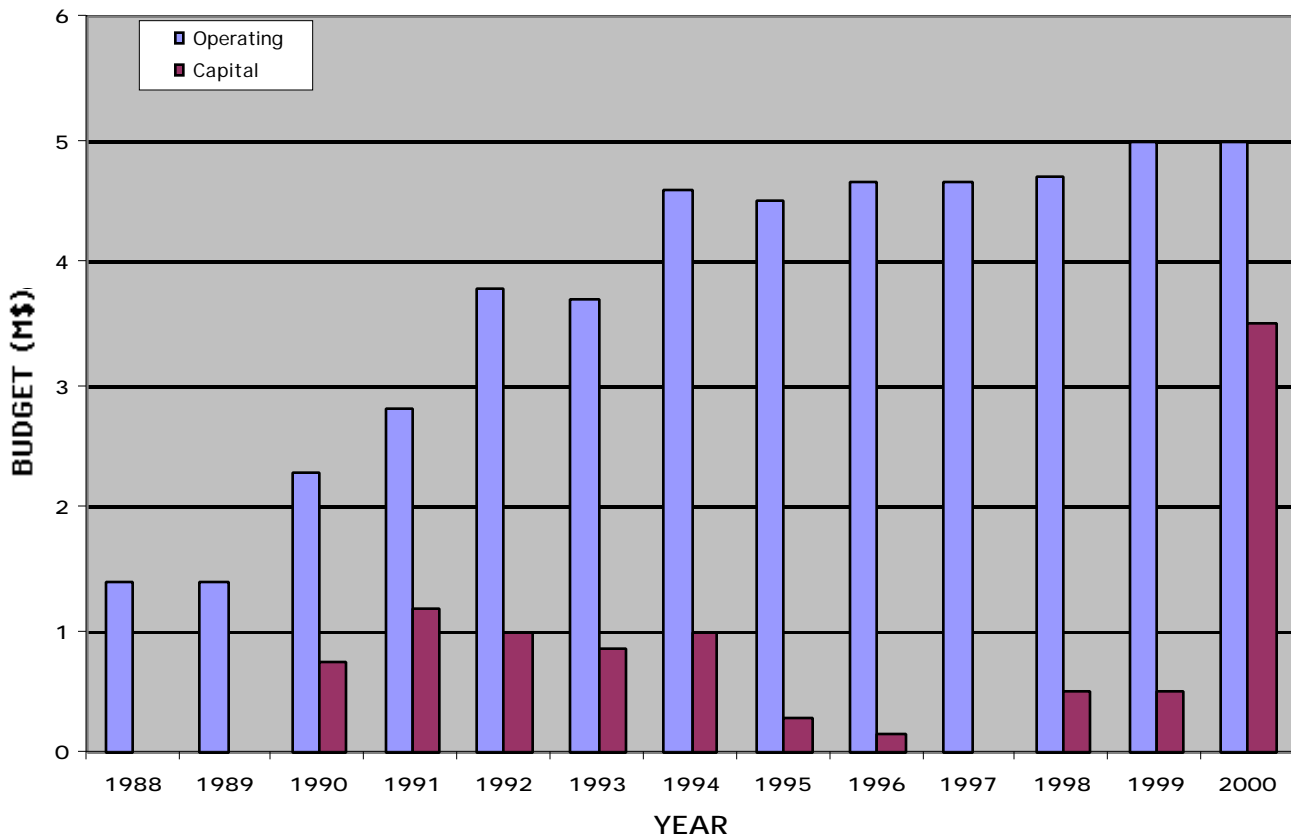
5.2.6 Machining and Inspection Research User Center (MIRUC)

The Machining and Inspection Research User Center (MIRUC) provides basic facilities for investigation of grinding processes for high-performance ceramic materials, design and fabrication of mechanical property test specimens, dimensional characterization of test specimens and other components, and tribology.

5.3 Status of Current User Centers

The six user centers have adapted to the changing and increasing needs of their customers through acquisition of staff skills and equipment. Operating funding increased steadily from the HTML's inception in 1987 to 1994, to about \$4.5-5M/yr (Figure 1), with capital equipment funding usually being made available also. However, from 1993 to 1995, this capital equipment funding

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decreased, until in 1996 it actually went to zero. It has since been restored at the \$500k/yr level, which is sufficient for most smaller equipment purchases. However, few major instruments have been replaced, as the major instruments in many of these centers now cost of the order of \$1M, and therefore, very few replacements of this type can be accomplished while maintaining all of the user centers. Upgrading doesn't work very well for most of the big instruments, such as electron microscopes or Auger spectrometers. Some of the classes of instrument important for state-of-the-art materials characterization change quickly and upgrades for such instruments are not practical. Electron microscopes, atomic force microscopes, and profilometers are good examples of this type of evolution. Under these circumstances, after being in service for about five years, a given major instrument in the user centers is made more or less obsolete by new instrumentation developments and their implementation in the instrument marketplace. We managed to get permission to combine the FY 1997 and FY1998 equipment allocations in order to purchase a badly-needed replacement for the PHI Model 660 Scanning Auger Microprobe (SAM). However, Congress and DOE are not likely to permit this sort of carry-over on a regular basis.

New instruments, including a major transmission electron microscope (TEM), a high resolution scanning electron microscope (SEM), an environmental mechanical properties system, a thermal microprobe, and a high mass, high-temperature microbalance system have been added in the original four user centers. Two new user centers were equipped and became operational in FY 1992. This occurred due to industrial requests and Headquarters support of the requests. The Residual Stress User Center (RSUC) allows measurement of surface strains in solids and the Machining and Inspection Research User Center (MIRUC) provides machine tools and surface

characterization instruments necessary for state-of-the-art machining research on ceramic and hard metallic materials. In addition, metrology instrumentation for dimension and surface characterization is a necessary component of this center. In FY 1993, a high-temperature, large mass Cahn TGA and the scanning thermal conductivity microprobe were added to the TPUC. The DUC and RSUC added two residual stress x-ray spectrometers with position sensitive detectors ("upgrades") to provide faster measurement capability, plus instrumented grinders and surface/dimensional characterization instruments were added in the MIRUC.

Also during FY 1992, the Ceramic Manufacturability Center (CMC) was created in the HTML. This Center resulted from a partnership formed with Defense Programs, Energy Efficiency and Renewable Energy, and Energy Research contributions and it was affiliated with the Machining and Inspection Research User Center. The CMC focused on working with industrial companies on major projects, principally via Cooperative Research and Development Agreements (CRADAs), to achieve cost effective ceramic machining and finishing. In 1997, the funding to start new DP CRADAs vanished, and new projects were not started as the earlier ones were completed. By 1998, all of these efforts had been completed, and new efforts in machining research were all incorporated into the MIRUC.

During the time that the DP CRADAs were operating, HTML received several specialized grinders from Cincinnati Milacron for ORNL use as part of Milacron's "in-kind" contribution to the CRADAs. HTML has subsequently negotiated with Milacron to retain these machines at ORNL and make them available for User Projects.

Two other new user centers were planned for introduction in FY1996: the Friction and Wear User Center and the Environmental and Thermal Fatigue User Center. However, funding was not available to allow their startup. To accommodate the numerous requests for friction and wear research, the existing tribological equipment and capability resident in the Ceramic Surface Systems Group of the Metals and Ceramics Division was added to the MIRUC. This was funded by shifting \$100k/yr from the HTML User Program to the MIRUC, with the funds being made available from savings brought on by Total Quality Initiative-led cost reduction efforts in the User Program.

The customer-identified needs for environmental testing, however, have only been partially met, by incremental addition of capability and equipment to the existing user centers. As will be apparent later on, this effort has not been totally effective.

6.0 STRATEGIC PLANS FOR RESPONSE TO CUSTOMER NEEDS

6.1 Materials Analysis User Center (MAUC)

In the development of a plan for the MAUC, several general issues were identified. During the first five years or so of the user program over half of the industrial users came to the HTML to use the MAUC to conduct research on structural ceramics for intended use at temperatures well over 1200°C. Since that time, there has been a shift in emphasis in the community using this user center to materials for other technologies supporting energy efficiency. The materials systems of importance to these users have shifted and broadened considerably. One example is the rapid increase in studies of structure-performance relationships in catalyst materials. In the past five years MAUC personnel have studied over 50 catalyst materials systems; all but about five of these catalyst systems were being used or developed for gasoline or diesel emissions reduction. The MAUC is well positioned to accommodate this change since the analysis/characterization techniques presently available in MAUC are generally applicable in the study of most of the materials systems that may be encountered. Additionally, a majority of the MAUC staff members have a very broad materials experience and knowledge.

As is discussed in detail later in this document, MAUC is well along in upgrading instrumentation. A new Auger spectrometer, high resolution SEM, and focused ion beam (FIB) micromill have all been added during the past two years. We now have funding for the next-generation TEM, an aberration-corrected electron microscope (ACEM), which will push our resolution capabilities well below 0.1nm (see section 6.1.2). Hitachi is consigning to us free of cost a new STEM worth about \$1.5m, which will push our micro-compositional resolution limit below 0.2nm. We are seeking funding for a new electron microprobe, with hopes to replace our existing microprobe in FY-2001. We continue to lead the nation in the development and use of virtual laboratory concepts. About 15% of user activities are performed using (at least in part) remote microscopy. This shortens research time and saves funds, making the user efforts more efficient.

6.1.1 Research Thrusts

- Continue to improve efficiency via primarily remote instrument operation
- Add environmental reactor capabilities
- Improve capabilities to image polymer and other organic structures
- Increase our fuel cell characterization research to include PEM and oxide-based cells
- Continue catalyst research with emphasis on NO_x reduction/storage
- Develop a major funded effort in the characterization of emission particulates
- Lead the nation in the practical use of aberration-corrected electron microscopy
- Encourage and participate in the design and construction of a new microscopy center

6.1.2 Actions to Meet Thrust Areas and Industrial Pull

Nearly all instruments in the MAUC are remotely operable. Industrial users have enthusiastically embraced the concept of performing research in the HTML from their own offices. Although many industrial researchers have great interest in performing remote research, only a few are actually doing it. This is because the potential industrial user faces a major hurdle presently, the firewall that separates the company intranet from the global internet. This, more than any other obstacle, hinders the growth of remote industrial use. This can be overcome by using Java-based user interfaces that are browser compatible. Based on our industrial user needs, we will be taking the following steps over the next several years. Simple browser interfaces will be developed to allow operation of our major instruments remotely. In addition, more instrument functions will be automated (aperture control, for example) to increase the capabilities of the remote users. Telepresence tools such as video cameras will be upgraded/added to provide a more lifelike interaction environment. Electronic notebooks and whiteboards will continue to be developed

The ex-situ reactor that was constructed a few years ago has been extremely popular with our industrial users. The reactor allows high resolution TEM of areas of a sample before and after high temperature exposure to a variety of gases (such as simulated diesel exhaust) without exposure to room air. This industrial interest in one reactor is an indication of where we as a laboratory need to be placing emphasis for future capabilities: exposure and observation in environments that mimic the real use environment. The MAUC plans to address this need with the following steps. A second ex-situ reactor will be constructed with a design that will accept the ACEM specimen holder, thus providing reaction capabilities with the ultimate spatial resolution. A new reactor for larger samples (SEM, probe, SAM, etc.) will be constructed and integrated into a controlled atmosphere glove box. We will seek funding for an environmental SEM to provide the ability to observe in real time the effects of various atmospheres at elevated temperatures on larger specimens.

Emissions research will be increased in several ways as a result of industrial demand. The diesel and automotive manufacturers are increasingly interested in reducing NO_x by the use regenerable chemical traps. These traps are often made catalytically active (to convert NO to NO_2 for example). Little is known about changes in microstructure with use. This characterization will be a major thrust for MAUC. Particulate emissions from both diesel and gasoline engines is a major issue for both automotive and diesel manufacturers (and DOE). We have begun a study with a number of collaborators to compare the structure, morphology and microchemistry of particulates from these two types of engines. It is our plan to seek funding to make the HTML a national center for such studies.

We have begun a small program characterizing proton exchange membrane (PEM) fuel cells using tools that we have developed for the characterization of catalysts. PEM fuel cells may be one component of the power system for the car of the future and are thus extremely important for the auto makers. PEM fuel cell structure includes a catalyst layer and several other layers/membranes. These are presently poorly characterized. The effects of use on precious metal distribution, particle size and loading need studying in order to produce a better fuel cell. We have the goal of becoming a national center for the microcharacterization of PEM and other fuel cells.

All of our industrial users require (and demand!) that we provide the highest resolution imaging and microanalysis capabilities that are technologically possible. To address this demand, this year we will be ordering a new electron microscope with next-generation technology, designed to our specifications. The instrument will be one of the first instruments in the world to incorporate a corrector for spherical aberration. This aberration-corrected electron microscope (ACEM) will allow sub-angstrom level imaging of atomic structures, along with electron spectroscopy to allow identification of composition down to the single atomic column level. It will also be controlled fully from an external computer, that will permit the instrument to be installed in a room away from the control system to eliminate the influence of human operators on the environment of the instrument. Our laboratory will be a world leader in capabilities for high resolution characterization of specimen structure and chemistry when this instrument achieves beneficial operation in FY2003.

Because the new ACEM requires an environment free from stray electric and magnetic fields, microphonics, and microvibrations, which is not available anywhere in the HTML, it is desired to locate the instrument in a new, specially designed building. MAUC personnel, the HTML Director and others in the M&C Division are working to develop external funding sources for a facility which would house not only the ACEM, but other sensitive microcharacterization instruments in M&C and other divisions at ORNL.

6.2 Mechanical Characterization and Analysis User Center (MCAUC)

The development of advanced materials for heat engine applications has been a major thrust area of research and development programs in the United States, in Japan, and in Germany for over ten years. The two primary drivers for these research activities have been the promise of increased fuel efficiency and lower emissions, if these materials could be made cost effectively. In addition to DOE sponsored programs, the industrial sector (diesel engine, automotive, and gas turbine manufacturers) has committed very substantial resources to the development of new and improved materials for a number heat of engine applications. In the case of high-temperature structural materials, several mechanical performance issues must be resolved before engine components fabricated from these materials are commercialized on a large scale for operating temperatures above 300°C or so. For newly developed materials, short-term mechanical property data (strength, fracture toughness, hardness) are required for manufacturing process optimization and verification. Historically, this has been a very slow and expensive process which has always made implementation of a new material require several years. For relatively mature material systems, an

extensive database describing mechanical performance as a function of time (creep, stress rupture, cyclic fatigue) is necessary for the design of engine components having an acceptable lifetime. Such data are also critical to the development and verification of life prediction models. Finally, the implementation of cost-effective manufacturing technologies for component fabrication generally relies upon mechanical property data to ensure that the mechanical performance of the component is comparable with that established during initial development of the material.

A major barrier to the measurement of the mechanical performance data discussed above has been the limited availability of state-of-the-art facilities. This has been particularly true for advanced ceramics, ceramic matrix composites, and metal matrix composites, which require specialized hardware for high-temperature property measurements. To address this problem, the strategy of the MPUC has been to make such instrumentation available to U.S. companies and universities through the HTML User Program. Current capabilities in this center include four facilities each with a special capability and mission. The four include a flexure/compression test facility (FCTF), the world's largest ceramic tensile test facility (TTF), an environmental test facility (ETF), and a micromechanical test facility (MMTF). The FCTF has been used primarily for the evaluation of the mechanical properties of newly emerging materials. Data generated from this facility have been utilized by manufacturing engineers to optimize and verify the mechanical performance of their materials. Time-dependent mechanical behavior (stress/creep rupture, cyclic fatigue) of relatively mature materials has been evaluated using the TTF. To date, extensive databases for advanced ceramics, high-temperature alloys, and composites have been generated using this facility. Issues pertaining to the effects of environment upon mechanical performance have been addressed with the ETF. Finally, the MMTF has been instrumental in the study of the mechanical behavior of thin films, thermal barrier coatings, polymers, and fiber/matrix interfaces in composites.

6.2.1 Research Thrusts

- Materials development and optimization studies
- Micro-mechanical behavior of materials including thin films, coatings, and interfaces
- Environmental effects on mechanical properties
- Long-term mechanical reliability modeling and verification
- Component failure analysis
- Development of test standards and design codes

6.2.2 Identification of Industrial Pull Areas

Discussions with key personnel in the industrial sector, particularly in the area of advanced materials for heat engine applications, have led to the identification of several new potential thrust areas. The first involves the development and implementation of techniques for the accelerated measurement of time-dependent mechanical behavior of advanced ceramics, high-temperature alloys, and composites. To this end, the MPUC is currently examining the application of stress relaxation tests for the measurement of creep rate versus stress data. Initial results have been encouraging.

The increased emphasis upon the measurement of creep and fatigue behavior of high-temperature alloys, as reflected by a number of recent user proposals, constitutes the second industry pull area. In response to this demand, several existing TTF test stands have now been modified to better accommodate the required test specimens. No increase in the support level will be required to address this area. Capital equipment resources were allocated in FY 1994.

The third industrial pull area is concerned with the application of instrumented machining to the fabrication of components with predictable mechanical performance. Because the facilities in the MPUC are well-suited to addressing this need, no increase in the support level or capital equipment

will be required to address this area.

A fourth industrial pull area is that of environmental effects on mechanical properties. Application of advanced materials involves exposure to a variety of corrosive environments including coal combustion by-products, contaminants present in natural gas used to fire gas turbines, and reactive gas constituents typically present in industrial incinerators. Currently the environmental exposure facility consists of two test chambers with inert gas capability and various test chambers for the conduction of tests in high temperature steam. However, the ability of life prediction models to account for corrosion-induced failure will require the extensive evaluation of mechanical performance of structural materials in representative industrial environments, including tests under actual service conditions.

The final industrial pull area is coatings, which are playing an increasing thermal and structural role in diesel engines and gas turbines. Issues related to the dominant failure modes in these coatings and prediction of lifetime are still largely unresolved. Fundamental mechanical property data are lacking due to the uncertainty in suitable test specimen geometries as well as the inability to simulate the high-temperature gradients expected during in-service operation. In order to establish a dedicated mechanical test facility for coatings these two issues must be resolved. The most reasonable approach to this problem is through the direct involvement of both the engine manufacturers and mechanical test equipment companies. Facilities including a dedicated high-thermal gradient test system should be established.

6.3 Thermophysical Properties User Center (TPUC)

The TPUC determines thermophysical properties such as thermal diffusivity, thermal conductivity, specific heat, and thermal expansion and also characterizes thermal stability, high-temperature reactions, compatibility, and oxidation and corrosion properties. TPUC also uses its focal plane array IR camera for high speed imaging of temperature distributions and for nondestructive testing. The materials studied include structural ceramics, superalloys, ceramic and metal matrix composites, low conductivity foams, die casting coatings and sands, superconducting materials, ceramic precursors, carbon materials, and carbon fiber composites. The major facilities are divided into three groups: thermal analysis, thermal transport, and IR sensing.

In the thermal analysis laboratory the current instruments include a simultaneous thermal analyzer (STA = DTA + TGA + MS) and a differential scanning calorimeter (DSC), both capable of operation to 1500°C in inert or oxidizing atmospheres. Both these are 12 years old; fortunately a new state of the art DSC (to 1650 °C) is on order. The thermal analysis laboratory also contains a dual push rod dilatometer for bulk thermal expansion measurements to 1500°C and a large mass, high-temperature, high sensitivity Cahn TGA with DTA attachment currently provides measurement capability to 1700°C for samples weighing up to 100 g. The Cahn TG unit permits measurements in vacuum, inert, oxidizing, and corrosive atmospheres and thus provides a considerable extension to the capabilities in thermal analysis. Complementary capabilities for thermal analysis by in situ diffraction methods exist in the DUC (see next section).

Facilities in the thermal transport laboratory include the laser flash and xenon flash units for determination of thermal diffusivity. Measurements can be made as a function of temperature from -100 °C to 2000°C with the laser flash system. This system allows for the measurement of thermal diffusivity of molten metals, as well as 2- and 3-layer samples. The xenon flash system, optimized for room temperature thermal diffusivity measurements, also enables measurement by the “step heat flux” method, which measures thermal diffusivity of heterogeneous materials including laminar composites. The step heat modification permits us to measure low thermal conductivity

materials. In addition, the xenon flash software has been modified to allow the determination of thermal diffusivity of coatings on substrates of known thermal conductivity. GE Aircraft Engines, who contributed significantly to this development, has heavily utilized the system to study the effect of thermal aging on thermal barrier coatings for turbine engine applications.

A Scanning Thermal Conductivity Microscope (STCM) provides for mapping thermal conductivity on a micrometer scale. This system is the first instrument in the world capable of direct measurement of thermal conductivity of such materials as intergranular phases, single grains and powder particles, fibers in situ, fiber-matrix interfaces, and environmental damage zones.

Recently added capabilities include the 3-Omega instrument, built by our staff, for measurement of thermal conductivity of coatings as well as bulk non conducting materials from RT to ~500 °C. It has been used for a variety of user and DOE projects in the first couple years. Another new instrument, purchased by OIT programs, is the Hot Disk Thermal Constants System which is capable of measuring thermal conductivity and specific heat from bulk and heterogeneous materials such as casting sands, foams, and refractory bricks. This instrument has proven to be useful to HTML users as well as OIT projects and provides a capability previously lacking in TPUC. The third recent addition is the focal plane array high speed infra red camera. This camera is capable of speeds up to 6000 Hz and temperature sensitivity of 0.015 °C. For full 256x256 data collection the speed is limited to 120 Hz. This major acquisition was funded by the Advanced Turbine Systems Program of OIT, but it is now extensively used by HTML users such as Ford, GM, and Bosch for brake system thermoelastic instability studies and by a host of other users from both industry and university. Coupled with a controlled spot size laser, the camera can be used for determining the in-plane thermal diffusivity of thin coatings or the thermal diffusivity of a 2-D specimen simultaneously. The camera has also proved valuable to users in a number of nondestructive testing applications and for unique temperature mapping in process environments (e.g. casting).

In addition to instrument capabilities, the TPUC also provides access to computer programs designed to model the temperature dependence of thermal conductivity and to provide information on the microstructure and purity of the specimens. The TPUC also maintains a computer database of literature published in the field of thermal conductivity over the past 14 years. This database contains over 5500 records and is updated every month. Users of the TPUC also have access to a thermophysical properties reference library and a large collection of journal articles filed by material and measurement technique.

6.3.1 Thrust areas requiring thermophysical property data

Thrust areas of OTT and DOE customers have been identified via user feedback, the user forum, and from stated needs of OTT and DOE. These include:

- Environmental stability testing
- Thermophysical properties for computational modeling
- Coatings for thermal barriers, oxidation resistance, and wear resistance
- Composites - ceramic fibers or whiskers in ceramic or metal matrices
- Ceramic microstructure engineering
- Non destructive testing using IR sensing

6.3.2 TPUC actions and plans to meet industrial pull in the new thrust areas

Property data for new materials are widely recognized as essential for insertion of these materials into engineering applications. The TPUC provides expertise for measurement of thermophysical properties of new materials developed by industry and/or DOE such as intermetallic alloys, low thermal expansion ceramics, aerogels, carbon-carbon composites, fiber reinforced ceramic matrix

composites (CFCCs), silicon nitrides, aluminum nitrides, etc. The TPUC facilities also contribute to user studies of melting temperatures, high-temperature oxidation, and reaction to corrosive atmospheres of these new materials.

As advanced materials such as structural ceramics and lightweight alloys are considered for use in reactive or corrosive atmospheres, the need for fundamental and in-situ testing will increase. TPUC, with the new DSC and the recently purchased Cahn TG unit, can serve users in this thrust area. In addition, we should add capability to deliver controlled atmospheres to such instruments and to monitor the test gas environment throughout exposure. Growth in environmental testing is anticipated to be increasingly important and requiring long term exposure testing.

Process modeling is a rapidly growing industrial thrust and is being supported by new programs in DOE and other government agencies. These data are critical to the modeling effort as lucidly stated by William W. Scott Jr., executive editor of *Advanced Materials and Processes*: *"Data are required that no one ever needed before. Detailed information is critical to develop the precise models and computer simulations that are the tools of today's researchers ... Unfortunately, the data are often missing for essential physical, thermodynamic, and thermomechanical properties. At three recent ASM international committee meetings, the lack of critical data was noted in regard to forging, casting, heat treating, and diffusion. In these as well as other areas, I have no doubt that the major obstacle to future development will be a dearth of reliable data with which to model both materials behavior and resultant properties."* TPUC is ideally suited to fill this national need of OTT and DOE customers and to support the growingly important computational modeling thrust of industry.

As stated in the thrust areas above, a major new need for thermophysical properties is developing in the manufacturing sector, particularly the foundry (casting), rolling, and welding areas related to transportation materials and other sectors. Broad need has developed for thermophysical property data of molten and semi-solid metals. While many institutions and industrial companies are now developing advanced capabilities for modeling forming processes, only one or two U.S. institutions can measure the data needed by these models. TPUC staff has responded to inquiries from industry (e.g., Ford, and General Motors), universities (University of Pittsburgh) and internal ORNL programs (CRADAs in casting, and consortia in welding), concerning existing capabilities to make measurements in the semimolten and molten range. The new laser flash system allows for the determination of thermal diffusivity of molten metals. The new DSC will accurately determine specific heat capacities to higher temperatures, including those needed for molten metals. Further, we have demonstrated that, with modification, the existing dual push rod dilatometer can measure the thermal expansion and density of molten metals. In the future a capability to measure the viscosity of molten metals as a function of temperature will be required, as viscosity is an essential property for modeling molten metal flow (e.g. in a casting).

The TPUC has met a number of requests (UTRC, Pratt & Whitney, GE Aircraft Engines, Westinghouse, Arizona State University) for measurements of thermal conductivity of thin coatings (50 μm - 200 μm). While these coatings are too thin for measurement by the flash diffusivity technique, the TPUC staff developed two techniques for the measurement of thin films – the 3-Omega and the IR camera.

Composite manufacturers (FMI, Hercules, Textron) require the high temperature thermal diffusivity/conductivity of fibers to predict and model the thermal conductivity of composite materials. New fixturing techniques will allow fibers to be measured using the high temperature laser flash system.

By FY 2001, the STA/MS will be over fourteen years old and will need to be replaced with a new, more sensitive full-featured unit. Such a replacement is critical to HTML addressing the thrust area of real time, in situ measurements related to materials processing and stability. In addition, before conducting extensive in situ property testing (e.g. tensile strength), one must know the material's

response to the test environment. Along with replacing the STA/MS we will also need to develop extensive gas flow metering and mixing capabilities.

The acquisition of the high speed, high resolution infra red camera by OIT has opened the door to a totally new field for HTML – thermal sensing and imaging. Besides its use to determine thermophysical properties, the IR camera also serves as a nondestructive testing and temperature imaging tool. The thermal signature of a material is quite sensitive to hidden voids, inclusions and/or corrosion. We have made the camera portable and by doing so have vastly expanded industry's use of TPUC. Notable projects include studies with Ford, GM and Bosch on thermoelastic instabilities in disk brake rotors. Users, via proposals and input at the user forum, have given strong recommendation to expanding the use of thermal sensing and imaging into a new user center activity. This would include addition of new cameras and, continued development of single point detector tools.

6.4 Diffraction User Center (DUC)

The DUC examines, frequently in situ and in near real time, the phase composition, atomic or crystal structure, phase transformations, reaction of solids on heating (processing), phase equilibria, lattice thermal expansion, and high-temperature stability of ceramics and metals. The major facilities include: (1) two high resolution, room temperature X-ray powder diffractometers (RTXRD); (2) the high-temperature furnace attachment and diffractometer system (HTXRD); (3) high temperature furnaces for the high resolution neutron powder diffractometer; (4) x-ray synchrotron beam line X14A with diffraction, reflectivity and EXAFS capabilities; and (5) comprehensive computing facilities for both automation and data processing. The X-ray furnace has been used for studies to 1500°C in oxidizing and reducing (including H₂), to 2000°C in nitrogen and inert atmospheres, and to 2500°C in vacuum.

6.4.1 Thrust Areas

The trends in user requests and the recent user forum input for the Diffraction User Center was dominated by increasing emphasis on in-situ, real time measurements, building on the existing strengths of this user center. Materials evaluations in the coming years include:

- Oxides for gas separation and fuel cells
- Catalyst materials characterization, stability, and behavior
- Functional ceramics (e.g. piezoelectric, electrostrictive and electronic)
- Environmental testing of ceramics, composites, and alloys
- Ceramics and alloys for high-temperature structural applications
- Ceramic processing (e.g. crystal chemical engineering and process optimizations)
- Process modeling data (crystallization kinetics, phase equilibria)

6.4.2 DUC Actions and Plans to Meet Industrial Pull in Thrust Areas

DUC staff have developed, and users have benefited from, capabilities for HTXRD analysis in 1 atm hydrogen up to 1500°C and in 1 atm nitrogen up to 2000°C. These are unique capabilities within the U.S. and have been of considerable value to industry (e.g. Dow, Tosoh, Tennessee Eastman, NASA, and Detroit Diesel). However, recent inquiries call for a broader array of in situ capabilities, e.g. steam, sulfidizing, combustion, controlled partial pressures, and other gas environments with substantially enhance computer control of gas mixing and monitoring. Also recommended to DUC were enhanced real time capabilities (expanded capabilities at X14A and new high flux HTXRD systems at HTML are required to meet this expectation). For

measurements in corrosive and/or sulfidizing atmospheres, additional furnace chambers and fixtures need to be obtained so that cross contamination between projects can be avoided. In addition, an exhaust gas analyzer needs to be added as well as a gas flow control manifold for accurate mixing of gases to better simulate processing conditions or environmental conditions.

Position sensitive detector (PSD) systems can greatly speed data collection and permits capturing rapidly occurring reactions and phase transitions as well as determinations of reaction kinetics. Software and instrumentation efforts using existing PSD detectors are being pursued.

Recent emphasis on process modeling and process development has led to new user proposals for crystallite size, grain size, and strain distribution analysis. The RTXRD goniometer is ideally suited for these studies due to high resolution and the solid state detector. However, the addition of multilayer mirror optics is now essential if HTML is to meet the demanding expectations presented by user projects such as characterization of nanocrystalline materials.

The considerable increase in user proposals to determine atomic structure at RT and HT is certain to continue and grow, with particular emphasis on computation facilities and tools. Computing facilities for the DUC are based on Windows NT workstations and PowerMac computers. Substantial upgrades have been completed in the last three years, but this is a fast changing field and continued investment is needed and expected by the user community. Having strong expertise in computational tools complements the strength in instrumentation and are both required.

To complement the HTXRD and RTXRD facilities, HTML and the Solid State Division (SSD) established in 1995 a working agreement to provide HTML users access to neutron facilities at HFIR. Access to neutron powder diffraction facilities was included in the EE-ER agreement for HTML development of a residual stress facility at HFIR (see RSUC Section). This joint program provides limited access to the neutron powder diffraction facilities for HTML users. We have contributed to furnaces for the neutron powder diffractometer to further optimize that instrument for our users. Such capability significantly strengthens DUC, since we can now choose the appropriate scattering probe for the users' problem. In fact, this agreement makes DUC a single point of contact for access to laboratory x-ray, synchrotron (see below), and neutron diffraction facilities – making DUC a unique user facility worldwide.

Synchrotron facilities are an important and essential component of DUC and RSUC. With over 1000-fold higher intensity, wavelength tunability for unique resonance and EXAFS studies, and highly parallel beams, the beam line opens new opportunities to HTML users. In FY98 and FY99 we provided limited support for a portion of the beam time. We found that there is a breadth and depth of problems in the user community that are best, or can only be, addressed by synchrotron radiation. Following major upgrades (6x in flux) in FY99 and installation of remote collaborative tools, in FY00 HTML was offered the opportunity to assume management of the ER created beam line, providing expanded access to HTML users. With some instrument development, X14A can provide for exceptional time-resolved high temperature, in-situ X-ray diffraction for residual stress measurement from areas as small as 0.25 mm and at high temperature, and for resolution of fine structural changes in materials that impact properties but are currently not detectable. As this beam line would revert to NSLS and be lost to materials science if we did not act, we have put in place an interim plan to operate the beam line while requesting adequate operating funds in FY01 to maintain and improve this beam line.

Spallation Neutron Source

The Spallation Neutron Source (SNS), 12x higher in neutron flux compared to the best pulsed facility in the world today (ISIS in England), is being built at ORNL and scheduled to be operational in year 2006. With the increased flux, along with improvements in neutron optics,

instrumentation and detectors, SNS will open unparalleled new research and characterization opportunities of importance to EE/RE programs and customers. For example, the proposed Engineering Materials Science instrument will be able to examine equipment in operation (e.g. lubricated gears under loads or operating fuel cells), manufacturing processes (e.g. casting, extrusion and welding), materials failure mechanisms (e.g. low and high cycle fatigue), and life prediction (e.g. stress evolution through life).

At SNS a suite of 10 instruments (averaging \$8.5 million each) are to be built by the SNS program to meet a wide range of materials research and characterization needs. Coupled with the High Flux Isotope Reactor the SNS will be able to assist customers in research on engineering materials deformation, residual stress mapping, materials stability and transformation studies, and materials processing characterization for verification and validation of models. The capabilities for real time, in situ measurements will be a major thrust of many SNS instruments. An additional 8-12 instruments likely will be built by future Instrument Development Teams (IDT) for specific research of the IDT members, and construction of a second experimental hall is being considered by NSF.

In order to have timely access and appropriate instruments at SNS to meet the needs of EE/RE customers the HTML User Program plans for a significant presence at SNS on several instruments as well as maintaining its existing presence at the High Flux Isotope Reactor. Without such access to these major DOE facilities, US industry will fall further behind their overseas competition in use of the unique properties of neutrons for understanding materials and materials processing. This is particularly important for OTT customers whose industrial problems often require high resolution, high sensitivity and fast measurement, can require quick access, and may need to be proprietary.

6.5 Residual Stress User Center (RSUC)

The RSUC provides a world class facility for measurement of macro strains and texture in ceramics, metals, and composites by laboratory X-ray, synchrotron X-ray and neutron diffraction methods. To our knowledge, it is the only program in the world with all capabilities managed by one Group. The X-ray facilities provide near surface residual strain measurement on conventional flat as well as highly curved surfaces, a unique capability. The neutron diffraction facilities provide non-destructive through thickness mapping of strain gradients.

The major X-ray diffraction facility is the Scintag powder-texture-stress (PTS) four-axis goniometer and an 18 kW rotating anode generator. FY 1994 procurement of a position sensitive detector extended the system and provided for more rapid data collection, necessary to complete many of the user projects in a reasonable length of time. A second Scintag PTS goniometer was installed in FY 1994 to meet the strong demand for laboratory x-ray stress and texture measurements by industrial and university users. With RSUC development of grazing X-ray incidence stress measurement, depth profiling of stresses for depths of a few hundred Angstroms to one micrometer is another unique capability. This grazing incidence capability is also very important in characterizing grinding damage in ceramics.

The user requests for characterization of stresses in thin films at room temperature and in situ and the near surface stresses in ground ceramics has grown such that access to a high flux, highly parallel synchrotron beam line was essential to meet user needs. In 1997 HTML's RSUC and DUC became partners with BES and others in the X14A synchrotron beam line built by the BES program at ORNL. Due to the growth in demand by October 1999 RSUC&DUC have been granted leadership of the beam line. Due to upgrades in 1998-1999 today X14A is the highest flux bending magnet beam line at the National Synchrotron Light Source (NSLS) and is ideally suited for a wide range of materials studies. The investment in this beam line has been over \$2M in

equipment alone.

In another innovative method to expand our facilities, a large specimen TEC goniometer was obtained from our sister laboratory Y12 in late 1998 and made operational in 1999. This provides the user center with the capabilities to meet user requests for surface stress measurements. This system is already being used to characterize stresses in such components as aluminum casting dies (Ford) and boiler tubes (OIT – Forest Product Industries).

Neutron diffraction facilities located at ORNL's High Flux Isotope Reactor were added to the RSUC in FY 1994 under a joint agreement between ER-Division of Materials Science and EE-Office of Transportation Materials. Neutron diffraction uniquely enables nondestructive mapping of strain fields throughout the thickness of a component and can be used to obtain the strain and stress tensor. This subsurface strain measurement capability is unique for materials like aluminum and steel. ORNL's HFIR is the highest flux facility in the United States. The RSUC facilities for macrostress mapping currently consist of an XYZ mapping attachment, an array of seven position sensitive detectors (FY98), and LabView based computer control. The XYZ table for mapping is placed on an existing triple axis spectrometer at the HFIR made available on a part time basis (~2/3 of the time) by the ER program. For micro residual stress characterization the High Resolution Neutron Powder Diffractometer at HFIR is made available to the RSUC program up to one quarter of the available time.

The EE/ER agreement called for construction of a dedicated macro residual stress mapping facility, which is currently underway. New goniometers have been purchased, new beam tube and shutters designed (by Solid State Division), and studies to optimize the monochromator have been completed. The goal is to provide an effective improvement in residual stress measurement by over a **factor of ten** via a combination of increased beam tube dimensions, improved monochromators, and complete utilization of the 7-detector array. The monochromator package will optimize strain measurement for a wide range of materials. For example, strain mapping in aluminum alloys will be improved over twenty fold! These facilities will be installed during the upgrade of HFIR's beryllium reflector and beam tubes in FY2001.

6.5.1 Thrust Areas:

There are numerous thrusts in national programs and customer technology driving the development and focus of the RSUC. These include

- Metal and alloy advanced processing, joining, and surface conditioning

The transportation industry continues to make advances in metal and alloy processing, joining and surface conditioning aimed at improving efficiency, reducing emissions, and lowering costs. Since residual stresses add to the applied loads they must be known to predict materials life and failure probability. Diffraction stress measurement is often the best method to obtain this data both at the surface and through thickness. As international standards for neutron strain mapping are developed and we put into service the new facility next FY we expect this demand for the RSUC instrument is likely to grow. Studies such as with Cummins Engine Co. on nitrided steels, friction welding of pistons, and induction hardening are typical of the problems we anticipate users to bring to RSUC.

- Ceramics for high-temperature structural applications

Characterization methods for processing induced stresses, such as from grinding and finishing, are of major importance as increases in engine efficiency or lowering the cost of manufacturing are pursued. Work in this area will often be in coordination with the Materials Characterization and Analysis and the Machining and Inspection Research User Centers. Examples of industrial request

include Norton and AlliedSignal's visits to our facilities in an attempt to find a nondestructive means to measure subsurface damage due to grinding. The thrust is to be able to distinguish between various grinding-finishing schedules in silicon nitride ceramics that lead to the same part dimensions but have different subsurface residual stresses and cracks. Based on the observations on the AlliedSignal specimens, the RSUC developed grazing incidence diffraction methods and has shown that this approach is a powerful tool for characterizing the subsurface region. Recent reports from Germany indicate that this approach is being extensively developed there for such characterization. As we recently demonstrated for a Mo film, this technique can successfully be employed in a laboratory with a rotating anode source, but is better and would be much faster using synchrotron radiation due to the vastly superior intensity (about 1000 fold). Grazing incidence methods are also proven valuable for characterizing stresses in thin films and heat-treated layers.

- Coatings and thin films - Thermal barrier, wear, protective, photovoltaic, microelectronic, etc.

Residual stresses in coatings arise from several sources. First, a thermal expansion mismatch between coating and substrate leads to residual stresses. Second, in TBCs for example, an interface oxide can grow over time between the bond coat and the zirconia top coat. The oxide itself will introduce a residual stress state that can lead to spallation. To meet the industrial needs, we anticipate growing use of X-ray and neutron methods as well as optical and/or Raman methods for stress characterization. The diffraction method has been shown to be applicable for growth stress measurement of oxide coatings at 1100°C, a measurement not able to be performed any other way. When the interface stresses are of importance in thick coatings then non-destructive neutron methods are appropriate. We have proposals from companies interested in the residual stresses in plasma spray metal wear resistant coatings within aluminum engine cylinders, for example. Again, neutron methods are used to gain access near to the interface region. Characterization of thin coatings for other applications continues to grow, including diamond coatings and the effect of processing. Residual stresses in thin films for use in photovoltaic and microelectronic devices also are of growing interest and several proposals for measurement have already been received. The x-ray synchrotron facilities are the key to fully meeting the user requests for stress measurements in thin films.

- Smart materials, gas separation membranes, fuel cell electrodes, batteries

The use of functional ceramics in energy production is a rapidly growing area with expectation that they will contribute to reduced emissions, reduced green house gas generation, and improved performance. Stresses are one of the important design issues that effect long term stability and life. First ever proposals for use of the x-ray and neutron diffraction strain mapping facilities for in situ studies of response of piezoelectric and electrostrictive and shape memory alloy materials to changes in applied voltage or temperature were supported in 1999. Based on the success of these projects and growing national programs we anticipate growth in this and related areas. In addition, the use of neutron diffraction techniques to characterize residual stresses at buried interfaces is now established and has begun to be used by users, e.g. ceramic to metal joints. We expect in the future to use RSUC facilities to characterize stress and phase composition gradients both at room temperature and in situ during operation in operating fuel cells and gas separation membranes.

- Composites

Micro residual stress determination at room and elevated temperatures are of growing interest in ceramic and metal matrix composites. We have also received inquiries concerning stresses in single crystal fiber composites. A neutron diffraction furnace is available (ER and EE) for these requests for temperature dependent micro residual stress studies. These studies will be greatly improved once the Spallation Neutron Source is available (see below).

- Computational Process Model Development and Validation

Process modeling by finite element modeling methods is being extensively pursued by every major industry we talk with. Described in some detail was the need for material specific, accurate temperature dependent thermophysical properties for input to the models. The next most frequently asked question is how to validate the model predictions. Macro residual stress mapping by X-ray and neutron diffraction methods was selected by industry as one of the most revealing tests of the model predictions. Accurate, reliable stress results are essential for verification of process models and to improve process modeling methods, as well as to solve failure problems. Fundamental data on stresses in laser and friction stir welds and ceramic joints are also of major current and future interest.

The residual stress characterization is felt to be one of the most important calibrations that relate directly with the model's processing and/or life prediction results. For example, we participated in the NCMS/GM/Ford/Chrysler/Torrington/Gear Research Institute/DOE CRADA on Heat Treat Distortion to demonstrate our capability for these characterizations to major corporations in the transportation sector. Results from the first neutron characterization were very well received and based on this several user proposals and other projects have developed. Near surface depth profiling using X-ray methods are also being used to aid these companies understand the strengths and applicability of the two methods.

Neutron diffraction techniques offer a new and exciting potential for studies of the response of grains to materials deformation. Based on the work reported in the last couple of years the results of such studies will be critical in developing verified and validated materials deformation computational models. To accomplish this, a load frame for applying tensile and compressive loads at elevated temperatures is attached to the neutron spectrometer and the phase and grain orientation dependence of strain versus applied load is determined. This new area of characterization supports improved processing models and is best accomplished at a pulsed neutron source such as the under construction Spallation Neutron Source (SNS). We look forward to establishing a formal arrangement with SNS for HTML to participate in the design, construction and ultimately user access to instruments to be built for SNS.

- Exploiting Anisotropic Materials Properties via Texture

Many materials have anisotropic properties and most economical processing methods yield textured or non random orientation of crystallites in the microstructure. This is particularly important in aluminum alloys being adopted for weight savings in vehicles. Advances in processes are often through enhanced control of the microstructure and texture. This trend to improve performance of existing materials by means of improved control of texture will likely grow. We have received many proposals for texture analysis and have developed a foundation in this area. The two PTS goniometers make the necessary measurements and computer programs have been established for conversion of the measured pole figures into orientation distribution functions. Several user proposals have been supported, and we anticipate this capability to grow in the future due to its importance in materials properties.

6.5.2 RSUC Actions and Plans to Meet Industrial Pull in the Thrust Areas

Growth in the next few years in user activity and DOE funded research will be substantial in both the X-ray area, and even more so, in the synchrotron and neutron diffraction facilities provided that the foundations continues to be adequately funded. The importance to industry is well documented. The major thrust in 2000 and 2001 will be to complete the construction of the new neutron residual stress facility and to provide full operational support for the synchrotron beam line. The next key item is replacement of the aging, unreliable rotating anode x-ray generator with a modern system of

higher reliability and performance. Following that the next critical item would be replacement of the TEC large specimen goniometer with a more flexible, robotic based residual stress instrument.

Accessories for RSUC X-ray diffraction facilities can have a major impact on productivity and research capabilities. These include a 2-D detector, load frames, and furnace stages for in situ stress measurements (composites, films and coatings require this in particular. The 2-D detector is a revolutionary means to collect strain data providing more than an order of magnitude gain in speed plus major enhancements in the information obtained during the diffraction experiments.

Raman and optical fluorescence methods are making major advances toward stress mapping in ceramics and microelectronic materials. The Raman technique is used most extensively to characterize diamond, silicon, quartz, and alumina materials. Raman is also valuable for characterizing silicon nitride and silicon carbide structural ceramics. With micro Raman capabilities the spot size is extremely small and even depth profiling is possible.

6.6 Machining and Inspection Research User Center (MIRUC)

The MIRUC provides basic facilities for (1) investigation of material-removal processes associated with machining of high-performance ceramics; (2) design and fabrication of new specimen test geometries required to evaluate the mechanical performance of structural ceramics; (3) dimensional inspection and characterization of machined specimens, including measurement of surface roughness, form, orientation and subsurface structure; and (4) tribological (friction and wear) testing.

6.6.1 Thrust Areas

The investigation of material-removal processes is an extremely complex issue because of the large number of variables involved. Consideration must be given to the mechanical stiffness and other performance-related properties of the grinding equipment, the composition and performance of the grinding wheel, the composition and performance of the coolant, establishment of an “envelope” of acceptable grinding conditions, and evaluation of the effect of grinding on the mechanical properties of the material being ground. Recent user projects have focused on the testing of coolants and grinding wheels in an attempt to develop products that are well-suited to ceramic machining. Current and future thrust areas will include:

- Understanding the effect of machining processes on materials properties
- Rapid detection of processing flaws or machining damage
- Development of a comprehensive database for machining processes
- Production of cost effective ceramic components for heat engine application
- Development of improved grinding wheels and coolants
- Development of intelligent machine control systems that optimize the grinding process and minimize machining damage
- Development of improved dimensional measuring techniques for ceramic components, including the measurement of surface texture, size, and form.
- Development of innovative tribological instruments that accurately duplicate the operating environment of the materials being tested.

During the next five years, we will actively pursue user projects that:

- utilize the MIRUC to reduce manufacturing costs for ceramic and other exotic materials
- provide a better understanding of machining processes
- provide a better understanding of the effects of machining on material properties.

Our target customers will include heavy-duty diesel engine companies, automotive engine manufacturers, ceramic machining and manufacturing facilities, machine tool builders, and grinding wheel and coolant manufacturers.

6.6.2 Facility Plans

Our existing laboratories are very crowded, with nine different grinding machines and other machining equipment currently located in four labs. The inspection facility has been reduced from two laboratories to a single laboratory to reduce the cost of operation of the user center, and to accommodate the expanding needs of other user centers. However, there are no plans at this time to expand the amount of laboratory space available for the MIRUC. Older, less-utilized equipment will be surplussed or donated to university-operated machining research labs to make room for new equipment.

6.6.3 Equipment Plans

Grinding equipment is relatively expensive, costing anywhere from \$100K to \$1M for a typical computer controlled grinder with instrumentation. Rather than requesting capital funds to buy new equipment, we are investigating the possibility of using operating leases to acquire new equipment. A typical lease would be negotiated with a machine tool builder for a three or four year period. At the end of the lease, the equipment would either be purchased for its fair-market value or exchanged for new, state-of-the-art equipment. In order to instrument the equipment without making permanent modifications that would affect resale value, we are developing a portable instrumentation system that can be easily moved from one piece of equipment to another. Once this is available, our most pressing need is for a high-quality surface grinder that is capable of both conventional and creep-feed grinding. Negotiations are under way to determine the cost of this equipment.

7.0 PROPOSED PLAN FOR HTML RESPONSE TO CUSTOMER NEEDS

7.1 Facilities

The current HTML building is in excellent shape, with some minor problems of roof leaks, plumbing blockages, etc. Repairs of these problems are being funded by ORNL General Plant and Equipment funds. The office and laboratory space taken by the current HTML staff is fully and efficiently utilized. The need described in previous sections for space to expand to accommodate new equipment and user centers, and especially to accommodate the special requirements of the new ACEM, can only be satisfied by an addition to the current structure and/or allocation of space elsewhere in the Division. This has already begun, with some instruments from RSUC and MAUC being located in Buildings 4508 and 4500S, across the street from HTML.

Building for Electron Optical Instruments

Frequently, as a result of interactions with HTML users, and particularly industrial users, needs for additional facilities are identified. Due to the system of funding, it is necessary to identify and

justify any such new capabilities a minimum of two and preferably three years in advance. A period of four years is easier to accommodate, due to the time required to obtain the necessary industrial concurrence and support and to develop the plans to submit to DOE Headquarters staff identifying and justifying such requests. For the past several years, we have been receiving indications of a need for additional capabilities.

There is a special laboratory space need within the HTML Programs. Our current suite of scanning and transmission electron microscopes is limited in resolution capability not by the instruments themselves, but by interferences induced within the HTML building. These interferences include physical vibrations of the structure, temperature gradients induced via air flow spatial and temporal variations, and electrical and magnetic interferences induced from the electrical circuitry in and near the building. We have been able to partially compensate for these. However, the new Aberration Corrected Electron Microscope (ACEM), for which we received \$3M in FY2000, will ABSOLUTELY require a special facility in order to produce its best imaging quality. Thus, we are proposing that this instrument (and a staff person) be located in a special-built building, for which we are proposing to obtain ORNL GPP funding. The building would be large enough to contain ACEM, as well as a TEM from the BES-funded microscopy effort in M&C, and possibly an aberration-corrected STEM being built for the Solid State Division of ORNL.

The building would be specially designed and constructed to be as free as possible of the types of interference limiting the current, and especially the new generations, of electron instruments. It would also be constructed with a Class 1000 clean room atmosphere capability for the instrument labs, to minimize contamination of the instrument interiors when e.g.- changing electron sources out. The physical layout of the building would reflect the additional near-term future needs for dealing with remote microscopy, with a "studio control room" in the center surrounded by the individual instrument laboratory spaces.

7.2 Staff

New staff are continuously being hired, typically but not exclusively, through hiring of post-doctoral staff. These people bring the latest technological backgrounds, and a special enthusiasm, to the job. After having proven themselves to be suitable for HTML's needs, they are converted to regular staff. For some special staffing needs, however, experienced personnel are hired in order to either gain access rapidly to a new technology, or to fill in for a specialist who has left.

7.3 Equipment

Based on the customer requirements specified in previous sections, the capital equipment needed is shown in Tables 2 - 5. It is broken down by User Center and the year in which it is needed, with a short reminder of its utility and an estimated cost.

7.4 Operational Funding

The funding profile required to accommodate the staff, user center additions, and equipment is depicted in Table 6. We are assuming essentially no major growth in staff, but an increase in support is needed to keep up with rising costs (inflation). We anticipate operational support requirements for the six existing User Centers for the years FY2000 through FY2004 to increase from the FY2000 level of 4.7M\$ at 5% per year as depicted in the Table. There are three step-function increases also requested; one is for \$200k in FY2001 for operation of the NSLS X14A synchrotron beamline for DUC and RSUC. The second, for \$250k, occurs in FY2002, to provide funds to accelerate activities into nondestructive examination through specialized imaging techniques. The third is a major increase required to handle the SNS requirements, initiating in FY2003.

A sharing of costs of SNS instrument construction and operation are essential as time assigned to a partner will be primarily based on commensurate contribution to the construction and operation. The HTML User Program staff is highly experienced in neutron scattering via building and operating instruments at HFIR and can form the basis for an EE/RE presence at SNS. To impact SNS instrument development we are currently participating in two SNS Instrument Advisory Teams. By FY2002 OHVT should execute a formal agreement on presence and beginning in FY2003 contribute from \$1.0M dollars/year toward instrument and ancillary equipment. This would be split into \$750k for capital and \$250k for staff costs. By FY06 this funding level should transition from capital to dominantly operational expenditure (2 SY/yr.). Such investment would provide instruments that can address OTT materials problems with the staff expertise and ready access needed to optimally meet the needs of OTT customers.

7.5 Need for New User Centers

Imaging User Center

A capability frequently requested by HTML's customers is that of general non-destructive testing or evaluation (NDT/NDE). M&C was well known and respected in the areas of ultrasonic measurement, eddy-current testing, and x-ray projection macro- and micro-radiography. In the 1980's, a microfocus x-ray CAT-scan machine was added, as well as an ultrasonic microscope. However, DOE programmatic support for NDE at ORNL has vanished, and the capability is greatly diminished, as staff have moved on to other efforts. The eddy-current and ultrasonic capabilities have largely disappeared, and there is no one to run the CAT-scan system.

We propose to re-energize NDE/NDT activities to support customer requests for this type of analysis. The infra-red camera has outstanding capabilities in assessing, for instance, hidden damage due to corrosion, poor bonding of joined materials, and the presence of defects in materials. Several user projects have taken advantage of the camera, including Motorola, looking for poor bonding between battery interconnects, and a ceramic powder supplier looking for impurity agglomerates in their powders. The camera is now so busy, in fact, that another is needed, and we are proposing adding another, larger-format, camera in FY2001. These two cameras would form the basis of a new NDE user center, to be called the Imaging User Center. This center would not, however, be limited to the IR cameras.

Work initiated on a Laboratory Directed Research and Development (LDRD) Project by staff from the RSUC/DUC included neutron radiography and tomography, which are also imaging techniques. These efforts utilized the neutron beams of the HFIR and ORELA, and showed great promise. Follow-on funding is needed to bring these efforts to fruition. Similarly, x-radiography is an imaging technique that HTML should develop, utilizing the NSLS synchrotron beamline as well as internal ORNL x-ray capability. We would include in this center the x-ray CAT-scan instrument already in the Division.

The High Flux Isotope Reactor has available one beam tube (EF-1) and instrumentation area that could be dedicated to neutron tomographic efforts. The unique advantage of neutrons is the great penetration length, the high sensitivity to hydrogen containing materials (lubricants, water, hydrides) and distinctly different attenuation for elements with adjacent Z (e.g. Ni -Fe). This yields a much different contrast compared to x-ray tomography. The neutron instrumentation LDRD project proved the feasibility and outlined the unique advantages of developing a tomography effort at the world's highest flux reactor (HFIR) and identified the key aspects of development that would require future investment. The best opportunity to configure the EF-1 beam tube for this effort is in FY2002 during a scheduled upgrade period. To be ready for this window, engineering design and materials funding of \$150K (operating) would be needed in

FY2001. In FY2002 the beam tube would be installed, shielding constructed, specimen rotation and translation stages and detector system procured (\$300K operating and \$300K capital). Design of the instrument and detector would be modular so that if a Spallation Neutron Source option later proves advantageous the facility could be moved to that facility.

Synchrotron based x-ray tomography facilities have been developed for biomedical and microelectronic characterization. However, there is no high-energy synchrotron based x-ray tomographic facility for materials studies. Due to the large advantage for very high energy x-rays this would be best developed at the APS although initial work could be performed at NSLS. A collaborative effort with Argonne National Laboratory would be pursued, as the cost of independently developing beam lines is typically \$5M or more. This effort would require a budget of \$250k/year and initial capital equipment funding of \$400k.

To complement the neutron and synchrotron facilities the existing x-ray CAT-scan instrument must be upgraded, requiring new detection systems, new computer systems, and upgrades in software for both data collection and more importantly for image reconstruction and user interpretation. An investment in FY 2002 and 2003 of \$250K operating (~1SY) and \$100k per year capital would be required.

Because of the inexact nature of these cost estimates, they are not fully included in the Cost Tables in this MYPP. We show only \$250k/yr operating costs, inflation-adjusted through FY2004. We will generate better cost data when this document is updated next year.

APPENDIX A

STAFF AND INSTRUMENTS IN THE HTML USER PROGRAM

(For up-to-date information, see <http://www.ms.ornl.gov/htmlhome>)

A.1 Materials Analysis User Center (MAUC)

Staff and their areas of expertise are as follows:

T. A. (Ted) Nolan is group leader of MAUC. Additional MAUC staff members are listed below, along with the instruments/technologies for which they have primary responsibility:

Dr. L. F. (Larry) Allard- Hitachi HF-2000 field emission gun transmission electron microscope (FEG-TEM);

D. W. (Dorothy) Coffey- Hitachi S-800 FEG scanning electron microscope (SEM) and Hitachi Focused Ion Beam (FIB) micromill;

Dr. E. (Edgar) Völkl- Electron holography, digital and remote microscopy;

L. R. (Larry) Walker- JEOL 733 electron microprobe and S-4700 FEG-SEM;

Dr. D. (Doug) Blom- (postdoctoral research associate) Development of methods for the analysis of nanostructures such as fuel cells;

Dr. B. (Bernhard) Frost (University of Tennessee research staff)- Nanotip electron emitter development;

Professor D. C. (David) Joy (Joint ORNL and University of Tennessee Distinguished Scientist)- SEM and electron solid interaction theory.

Equipment Status Prior to 1998:

We were fortunate to obtain funding for a major new high resolution analytical electron microscope (AEM) in FY 1991 (an investment of about \$1M). One of a new class of TEMs, this instrument, the Hitachi HF-2000 200-kV field emission gun-transmission electron microscope (FEG-TEM), added three major new capabilities to the User Program. (At the time of purchase, this was one of only three in the United States.) 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 grain boundary compositions. A second 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 third capability introduced on the HF-2000 (and also being added to the JEOL 4000EX HRTEM) is digital imaging. This means that all images will be obtained digitally which results in an additional benefit of the reduction of photographic wastes and conventional packaging of data for users. For example, a user's images can now be transmitted to the home site via Internet.

The Vacuum Generators (VG) electron spectroscopy for chemical analysis/secondary ion mass spectrometry (ESCA/SIMS), one of the original instruments in the MAUC, had many capabilities

but was difficult to operate and was simply not suitable for a user environment. Therefore, the purchase of a new large entry port high resolution SEM was completed with the manufacturer accepting the ESCA/SIMS for a major portion (approximately \$250K) of the purchase price. This transaction was done utilizing FY 1993 funds.

Electron Microprobe Upgraded in 1998

The JEOL Electron Microprobe had been in used for approximately 13 years without significant upgrade. The old computer system (PDP-1173) and software were lagging well behind the technological advances that had been realized with other instrumentation within the group and industry. All data and some images required hard copy or manual input into the plant data system. Additionally, new correction algorithms and other software advances were simply not available for this system. For these reasons a new automation system from Geller Microanalytical was purchased. This system utilizes a PC running Windows 95 and includes software that incorporates many of the advances in technology that were developed over the last several years.

Several of the more recently developed correction algorithms as well as the classic algorithms are available for converting intensity ratios to compositional values. Large area imaging, combining stage and digital beam scanning in one collection routine, can be used to show macro effects such as corrosion attack or an entire weld. Most importantly, the data is compatible with other PC and Mac based software so that a multitude of programs can now be used for processing, transmitting to the customer, and storing to central servers.

To a lesser extent than some of the other instruments, the microprobe can now be operated remotely. Functions controlled through the computer are now accessible remotely. Included are stage, wavelength spectrometers, energy dispersive spectrometer, image collection including X-ray maps, qualitative and quantitative analyses. The complexity of the probe is such that it may never be operated routinely from remote locations by users. However, Larry Walker, our probe operator, is now making excellent use of the remote capabilities. Maps showing the locations of elements in metal alloy samples, for example, may take many hours to acquire. Larry waits to start these maps in the evening. He takes a few minutes once or twice in the evening to sign on from home on his PC (password protected access), initiates new scans, and evaluates results. Thus the probe has considerably more productive time at essentially no additional cost to our programs.

Hitachi S-4700 SEM Acquisition

The Hitachi S-4500 field emission SEM has been extremely valuable in the study of ceramics with its excellent resolution at low accelerating voltage. Low voltage imaging minimizes electrical charging on insulating materials such as ceramics and ceramic composites. A new SEM, the S-4700, has been introduced by Hitachi. This instrument has significant improvements over the S-4500 and any other SEMs presently on the commercial market. The S-4700 has the very best low voltage resolution available. It employs a virtual aperture and alignment system that allows changing the accelerating voltage without having to realign the microscope. This is a major advantage for the user program, since expert operator intervention is minimized during user sessions. Finally, the S-4700 is completely controlled by and operated from two PCs. Thus it is fully remote operable. We negotiated with Hitachi to trade our S-4500 and our JEOL 2000FX TEM for the S-4700 (worth about \$500k) for a differential cost of \$125k. The installation date was December 1998.

In early 1999 Noran Corporation consigned at no cost an energy dispersive x-ray analysis and phase ID system that was connected to the S-4700. This system has a value of about \$250k and will remain with us for at least one year. The phase ID portion was developed at Sandia (Albuquerque) and commercialized by Noran. The hardware portion produces a high resolution

backscattered Kikuchi pattern and the software analyzes the pattern automatically, and combines the EDS elemental data to produce an identification of the phase present in crystalline materials with grain sizes as small as 0.1 μ m. If projects develop that use the phase ID system, funds will be sought to purchase it or a newer version.

PHI 680 Scanning Auger Microprobe Acquisition

The state-of-the-art PHI model 680 Field Emission Scanning Auger Nanoprobe has been installed in the HTML and is operational. All performance tests were met or exceeded. A minimum electron beam size of 11 nm and a SEM resolution of 6.0 nm were measured; both were better than the guaranteed values of 13.0 nm and 10.0 nm, respectively. The observed signal/noise ratio of 725:1 also exceeded the specified ratio of 500:1. These specifications are an order of magnitude better than our previous SAM. A special analysis software package called "Multipak" has been installed to enable a wide range of spectra manipulations and processing. The instrument computer workstation has been connected to the local ethernet which permits data transfer and partial remote control to any remote PC or Mac.

Jane Howe, a graduate student at the NYS College of Ceramics at Alfred University, was the first user on the SAM; she performed Auger surface analysis on CVD diamond films. The study dealt with the effect of various heat treatments and environments on the surface composition of the CVD diamond. The work resulted in the publication of a winning poster presented at a Carbon Conference in July, 1999.

FIB Consignment

The Materials Analysis User Center was chosen by NSA-Hitachi Scientific Instruments company for a no-cost consignment of the latest generation Focused Ion Beam instrument (FIB), valued at ~\$700K. The period of the consignment for the FD-2000 FIB was a minimum of 6 months, and will probably extend to at least a year. The delivery date was April 1999

The FIB is an instrument that uses a beam of gallium ions to precisely mill a bulk (~50 micron thick) plate from a direction normal to an edge of the plate. This leaves a very thin "shelf" of material up to 20 microns wide by 20 microns deep by about 30 nm thick, supported by the remainder of the plate. The shelf is essentially parallel sided and nearly uniform in thickness over the whole area, and ideal for electron transmission. This is the newest method of preparation of specimens for electron microscopy, and is especially useful for preparation of cross-sections of layered materials and coated/treated surfaces. It is a rapid process, and allows virtually 100% success rates for sample preparation to be achieved, from just about any type of solid material, in 2-3 hours. The instrument has greatly facilitated our HTML user and other programs studying cutting tools, hardened or coated metal parts, composites, and electronic devices. It can be used to produce specimens for SEM and Auger analysis, and to manufacture micromechanical devices. The FIB is also available for BES and other research programs.

It is planned that, at the end of the consignment, we will lease the FIB. Since programs having several DOE funding sources are already using the FIB, leasing is seen as the favorable method of financing, with various programs providing for one or more month's payments. If we choose to continue to lease for five years the FIB would then belong completely to the DOE.

STEM Consignment

The Materials Analysis User Center was chosen by NSA-Hitachi Scientific Instruments company for a no-cost consignment of the HD-2000 dedicated scanning transmission electron microscope (STEM) valued at ~\$1500K. The period of the consignment for the HD-2000 is expected to be for at least one year and it is to be delivered in February 2000.

This instrument is the first commercial STEM since Vacuum Generators quit making their instrument several years ago. The HD-2000 is designed to be user-friendly (unlike the VG instruments, which had about one switch or knob for every dollar of cost). The HD-2000 is PC controlled with a generalized user interface quite similar to the S-4700 described above. Thus an operator of the S-4700 can quickly become an operator of the STEM. It is remote-operation ready and is ideal for a user environment. With 20X the sensitivity of any commercial TEM for elemental analysis and nearly as good spatial resolution as our HF-2000, it will be an invaluable tool for the analysis of catalysts, fuel cell materials, and other nano-structured materials.

The placement of the FIB and STEM in the Materials Analysis User Center is the result of Hitachi's recognition of the international reputation of the HTML in electron microscopy. They know that these instruments will receive a great deal of exposure from the very many users and visitors that come to the HTML.

In summary, major instruments in the MAUC now include the PHI-680 Scanning Auger Microprobe (SAM), the Hitachi S-800 FE-SEM, the JEOL 4000EX TEM, the Hitachi HF-2000 FEG-AEM, the Hitachi S-4700 FEG-SEM, the FB-2000 Focused Ion Beam micromill, and, soon, the HD-2000 dedicated STEM.

A.2 Mechanical Characterization and Analysis User Center (MCAUC)

Staff and their areas of expertise are as follows:

Dr. Edgar Lara-Curzio, Group Leader

Characterization of mechanical behavior of composites and their constituents (fibers, fiber coatings, interfaces), environmental effects, reliability and durability, modeling of mechanical behavior and constitutive relations, standard test methods and special test configurations.

Dr. Mattison K. Ferber, Senior Research Staff Member

Characterization of creep, stress-rupture, and fatigue behavior of structural ceramics in tension, flexure, and special test configurations; data acquisition software and hardware.

Mr. Timothy P. Kirkland, Principal Technologist

Measurement of mechanical properties of structural materials.

Ms. Laura Riester, Development Associate

Micro-mechanical testing, atomic force microscopy, microscopy, imaging, web-page development and maintenance.

Dr. Andrew A. Wereszczak, Development Staff Member

Creep, fatigue and corrosion behavior, life-prediction, and reliability analysis; finite-element analysis, fracture mechanics, and testing methodologies

Ms. Jessie B. Whittenbarger, Group administrative support

Ms. Donna L. Haynes, HTML Graduate Fellow

Metal matrix composites.

Mr. James G. Hemrick, Graduate student, (University of Missouri-Rolla)

Mechanical characterization of advanced refractories.

Facilities and equipment include:

High-Temperature Tensile Test Facility

- Eleven 35 kN capacity electromechanical test machines equipped with self-aligning grips for accommodating a wide array of specimen geometries, including button-head tensile, and flat specimens.
- Two servohydraulic test machines capable of monotonic, static, dynamic, and cyclic testing of a wide variety of specimen geometries.
- All test machines equipped with integral, electronic controllers and function generators, and compact two-zone resistance-heated furnaces (maximum temperature, 1700°C; sustained testing in ambient air, 1500°C).
- All machines equipped with low-contact force, capacitance extensometers (accuracy, ~0.1 μm at room temperature and ~0.5 μm at 1500°C).

Servohydraulic axial-torsion test machine capable of loads to 100kN.

Tensile Creep/Stress Rupture and Stress Relaxation Facility

• Creep and Stress Rupture.

- Nine electromechanical tensile machines designated for long-duration creep-stress rupture, and low frequency tension-tension fatigue testing.
- Computerized control and data acquisition using in-house developed software application.
- Compact two-zone furnaces (maximum temperature, 1700°C).
- Low-contact force capacitance extensometers.

• Stress Relaxation

- Two stress-relaxation test frames devoted to metals and superalloy testing equipped with averaging extensometers and furnaces capable of 1100°C
- Two stress-relaxation test frames devoted to ceramics testing equipped with averaging extensometers and furnaces capable of 1500°C

Ceramic Flexure Test Facility

- Six test frames with pneumatic actuators, each with loading capacity 2.2 kN to test simultaneously three flexure samples, C-rings, or compression specimens.
- Standard mechanical property tests (room temperature to 1500°C):
 - Static fatigue (time to failure measured as a function of static stress).
 - Dynamic fatigue (fracture stress measured as a function of loading rate).
 - Low cycle flexural fatigue.

Electromechanical Test Facility

- 200 kN universal (electromechanical) test machine with high-temperature (1800°C) furnace.
- Small universal test machine dedicated to room-temperature testing of flexure specimens and C-rings, as well as fracture toughness testing.
- Standard mechanical property tests (using standard as well as specially designed test fixtures):
 - Static fatigue (time to failure measured as a function of static stress).
 - Dynamic fatigue (fracture stress measured as a function of loading rate).
 - Cyclic fatigue (cycles to failure measured as a function of cyclic stress).
 - Fast-fracture tensile and compressive strength.
 - Shear strength.
 - Fracture toughness.
 - Capability for compression creep tests with straight cylindrical specimens and contact extensometer (accuracy, ~0.5 μm) and compressive creep rates (room temperature to 1800°C; stresses, as low as 0.05 MPa).
- Rotary bend fatigue machine equipped with small furnace for testing small (~ 75 mm long and ~ 10 mm in diameter) cylindrical specimens in fully

reversed cyclic loading.

Electromechanical Test Facilities with Environmental Testing

- High-temperature clamshell furnace and ceramic retort, for both compression and flexure tests in air, inert gas, or vacuum at temperatures up to 1500°C.
- Environmental tensile test facility: environmental chamber mounted on test machine (screw-driven), mechanical pump, diffusion pump, regenerative gettering furnace, and temperature controller. High-temperature static, tension-tension cyclic, or dynamic loading in vacuum or inert environments (Room temperature to 1600°C using button-head tensile specimen geometry).

High Temperature Compression Test Facility

Designated high temperature compressive creep test facility with clamshell furnace and contacting extensometers (SiC and sapphire extensometer rods).

Test Facility for Composites and their Constituents

- Six in-house developed electromechanical, universal test machines.
 - 10 kN load capacity, 150 mm stroke, and 8 nm displacement resolution.
 - Computerized data acquisition and control.
 - Environmental chambers to conduct tests in controlled atmospheres (e.g., inert gases with less than 10 ppm O₂, oxidizing and reducing environments, water vapor).
 - Non-contact laser extensometry.
 - Various specimen geometries, gripping arrangements, and fixtures (e.g., shoulder-loaded tensile specimens, minicomposites, single small diameter fibers and filaments, double-notched shear specimens, Iosipescu shear specimen).
- Micromechanical universal testing machine.
 - 1000 N load capacity, 25 mm stroke and 100 nm displacement resolution.
 - Equipped with miniature load cells, encoders and capacitance gauges.
 - Specially suited for composite interfacial testing by means of single-fiber push-in and push-out tests.
 - Array of indenter geometries (e.g., Vickers, Knoop, Cube Corner, Diamond and WC flat punch) to conduct indentation tests.

Mechanical Properties and Scanning Force Microprobes

- Special microhardness tester capable of operating at four load ranges (0-4, 0-20, 0-100, 0-650 mN).
- Sensitive capacitance gage for constant monitoring of position of indenter relative to surface of specimen.
- Constant monitoring of load as a function of displacement.
- Plastic and elastic components of displacement are separated either by continuous sensing of sample stiffness while force is being applied, or by calculating the hardness based on the recorded measurements of load vs. displacement.
- The scanning force microprobe gives information on the response of materials to indentation by compiling a three-dimensional image from an array of single line profile scans.

Life-Prediction Analysis (FEA + ERICA / CERAMIC + CARES)

- Work station with two life-prediction algorithms and integrated finite element programs for performing finite-element analysis (FEA) and reliability modeling work to predict probability of component survivability in known service environment and stress level:
 - Mechanical test data from standard specimens.
 - FEA data for stress in actual components.
- FEA support provided for other mechanical testing of monolithic ceramics and composites.

Resonant Ultrasound Spectroscopy (RUS) Facility

- Characterization and inspection of mechanical integrity of ceramic specimens and components.
- Resonance spectrum generated by sweeping frequency of ultrasound signal applied to component and by detecting resonance frequencies of component.
- Applicable to determining with high accuracy the full elastic constants of isotropic and anisotropic materials (e.g., single crystals, textured polycrystals, and composites).

Additional Capabilities

- Infrared Imaging used in-situ with mechanical testing.
- Acoustic emission capabilities.
- Strain gaging facilities.
- Testing of ceramic-ceramic joints.
- Testing of intermetallics.
- Thermal shock testing.
- Failure analysis
- Tube testing and test of tubular filters

A.3 Thermophysical Properties User Center (TPUC)

Members of the group with prime responsibilities for thermophysical property activities include the following:

Dr. Ralph B. Dinwiddie —thermal transport and thermal imaging;

Mr. Wallace (Wally) D. Porter —thermal analysis;

Dr. Hsin Wang —thermal transport and thermal imaging;

Ms. Joy L. Kilroy —group administrative support; and

Dr. Camden (Cam) R. Hubbard —group leader

Facilities and equipment include:

In FY 1993, funds were assigned for a new high-temperature micro-balance system. The TG system has a sensitivity to 1 ppm, permits corrosive gas environments, and can operate to 1700°C - features identified by past and prospective users as needed in the HTML. In cooperation with Topometrics, Inc., an AFM was modified to allow the measurement of heat flow in material surfaces on a microscopic scale. To our knowledge, this is the first such instrument for performing such measurements and gives the HTML a very unique instrument for a period of time. This type of measurement is of major interest to designers, fabricators, and users of continuous fiber reinforced composites, and other materials in which multiple phases are present. The measurements can be made on a scale of the order of a few micrometers. No commercial system is available for these measurements. A room temperature xenon flash diffusivity system was also developed and made operational with a portion of the funding coming from the Space Power Programs (which obtained a duplicate instrument). This instrument has been of considerable interest to industrial users who are developing advanced new materials, such as structural ceramics and composites, who have a need for knowing the thermal transport characteristics of their materials below about 100°C.

Major instruments in the PPUC now include the Xenon Flash Thermal Diffusivity, Laser Flash

Thermal Diffusivity, Longitudinal Bar System, Scanning Thermal Conductivity Microscope (STCM), Dual Push Rod Dilatometer, Differential Scanning Calorimeter (DSC), Simultaneous Thermal Analysis (STA), Mass Spectrometer (MS), High Mass Thermogravimetric/Differential Scanning Calorimeter, and a Pycnometer.

The major facilities currently supported by TPUC are divided into three areas: thermal analysis, thermal transport, and the new focus of thermal imaging.

Thermal Analysis

Stanton Redcroft STA1500 simultaneous thermal analyzer (STA)

–differential thermal analysis (DTA)

–thermogravimetry (TG)

–evolved gas analysis (EGA) by mass spectrometry

Stanton Redcroft DSC1500 differential scanning calorimeter

Theta dual push rod dilatometer

Cahn thermogravimetric system

The simultaneous thermal analyzer (STA = DTA + TG + EGA) and the differential scanning calorimeter both are capable of operation to 1500°C in 1-atm inert or oxidizing atmospheres. The thermal analysis laboratory also contains a dual push rod dilatometer for bulk thermal expansion measurements to 1500°C. The Cahn large-mass, high-temperature, high-sensitivity TG, with concurrent DTA attachment, provides measurement capability to 1700°C for samples having masses up to 100 g. This instrument supports measurements in vacuum, inert, oxidizing, reducing, and corrosive atmospheres, and thus considerably extends TPUC's capabilities in thermal analysis.

Thermal Transport

Anter laser flash thermal diffusivity system

Xenon flash thermal diffusivity system

Topometrix scanning thermal conductivity microscope (STCM)

3-omega system thin film thermal conductivity of non electrically conductive materials

Hot disk thermal constants analyzer

The laser flash and xenon flash systems measure thermal diffusivity of materials under a variety of conditions. With the laser flash system (discussed in more detail in Sect. 4.3.3), researchers can make measurements as a function of temperature to 2500°C, measurement of thermal diffusivity of molten metals, and analyses of two- and three-layer samples. The xenon flash system is optimized for room-temperature thermal diffusivity measurements.

The Topometrix STCM maps thermal conductivity variations in materials such as composites on a submicrometer scale, providing microstructural information not available by other means.

The 3-omega system was recently assembled to provide a means to measure thermal conductivity as a function of temperature directly. The greatest strength of this technique is the ability to measure the thermal conductivity of electrically insulating thin films and bulk specimens.

The Hot Disk system is based on the Transient Plane Source (TPS) method, which is one of the most precise and convenient techniques for studying thermal transport properties. This technique is capable of measuring thermal conductivity, thermal diffusivity and volume specific heat simultaneously. The Hot Disk system can be used to measure thermal properties of a variety of materials, especially low conductivity ceramics, plastics, powders and granular materials such as sands. Many of these materials are difficult to measure using the flash diffusivity technique due

to transparency in the infrared spectrum and high porosity. Some typical materials that have been measured are: high T_c superconductors, glasses, refractories, casting sand, metal alloys, composites, polystyrene, bricks, liquids and biomaterials.

Thermal Imaging

Amber infrared (IR) camera
Selected Ge lenses for both microscopic and broad field imaging
Image processing software
Portable system including computer and triggering circuitry

A high-speed IR focal plane array camera for 2-D thermal diffusivity and nondestructive thermography is the newest instrument in TPUC. The camera was purchased by the DOE-OIT Advanced Turbine Systems program to study thermal transport characteristics of TBCs on turbine blades and to assess its applicability to detection of defects.

Supporting Facilities

Five-cell helium pycnometer for density
Sample preparation facilities, including the following:
–diamond core drill
–low-speed diamond cutoff saw
–diamond band saw
–vacuum deposition chamber
–polisher

DEVELOPMENTS AND NEW CAPABILITIES

Thermal Conductivity of Coatings

Coatings are being widely used to increase the performance life of gas turbine engine hot-section components, with a future goal of increasing operating temperatures and, hence, fuel efficiency. Thin films are also widely used as protective oxide coatings, wear-resistant coatings, and corrosion-resistant coatings, and as active elements in electronic components. The 3-omega technique is used to determine thermal conductivity of electrically nonconductive coatings. A photolithographic method is used to deposit a small heater on the surface of the specimen. Using a variable-frequency ac power supply, a lock-in amplifier, and a furnace enables measurement of thermal conductivity as a function of both depth and to 500°C. The new 3-omega capability enhances HTML's ability to study the effects of processing conditions, thermal history, and microstructure on the thermal conductivity of high-performance coatings.

Laser Flash Thermal Diffusivity System

TPUC purchased a state of the art laser flash thermal diffusivity (LFTD) system in FY96 that significantly expanded TPUC's thermal-transport measurement capabilities. This highly flexible system can operate from RT up to 2500°C in vacuum or inert gas. Two furnaces are used to achieve this: one for the range from RT to 500°C and a carbon furnace for the 500 to 2500°C ranges. In addition, measurements of thermal diffusivity may be made in oxidizing or reducing atmospheres from 25 to 1700°C using a third alumina tube furnace. Six-sample carousels on the two vacuum/inert-atmosphere furnaces greatly increase the number of tests that can be made in a single day. In addition to standard disc-shaped samples, the new system will also measure square plates, powders, molten metals, and coatings. Completion of installation of accessory attachments

and debugging of the measurement software was followed by significant growth in productive user activity.

Thermophysical Properties of Molten and Semisolid Metals

In FY96 TPUC carefully assessed industry's current and future needs for knowledge of thermophysical properties. Based on that study, TPUC expanded its focus to include measuring the properties of molten and semisolid metals. These data have recently become very valuable in the national efforts to model materials processes such as welding, casting, quenching, and hot forging or rolling. TPUC has contributed to several user and DOE projects needing properties of molten metals. The new laser flash thermal diffusivity system has demonstrated the capability to determine the thermal diffusivity of difficult-to-measure molten alloys. TPUC also developed and applied techniques for determining the density of molten metal utilizing our existing dilatometer. These data are often coupled with studies on casting modeling.

Infrared Camera for Two-Dimensional Thermal Diffusivity Mapping and Thermography

TPUC has developed a system capable of producing quantitative thermal diffusivity maps of test coupons, plates, and tubes, as well as thermal effusivity maps of components with complex shapes, such as turbine blades and vanes. This system is ideal for the study of composites and coatings. At the heart of the system is a high-speed, high-sensitivity IR camera with a 256×256 -pixel focal plane array operating in snapshot mode. Each one of the 65,536 detectors that make up the focal plane array is exposed at the same time and for the same length of time. This allows for a straightforward pixel-to-pixel comparison in each image. With Ge lenses the spatial resolution can be adjusted down to $7.5 \mu\text{m}$, and the temperature resolution can be as sensitive as 0.015°C . The camera can operate at speeds up to 130 full frames per second, or up to 1480 images per second at a resolution of 64×64 pixels. The exposure time for each image can be adjusted down to $2 \mu\text{s}$, allowing the study of very hot and/or fast-moving targets, such as cutting tools, boilers, and brake rotors, during operation. The camera can be calibrated for absolute temperature measurement.

For 2-D thermal diffusivity mapping, 1000-W quartz lamps, a 1000-W xenon illuminator, or 4800-W xenon flash lamp heats the sample or part. The camera is used to record the temperature response of the heated surface with time. In some cases the thermal response of the backside of the sample is recorded. Custom software is then used to calculate the thermal diffusivity or thermal effusivity for each point in the image. The result is a thermal diffusivity or effusivity map of the object under test. This data can be viewed in a variety of ways, including a spreadsheet format, a 2-D false-color image, or a 3-D surface plot.

In addition to the measurement of temperature and thermal properties, the camera can also be used for thermography applications for nondestructive evaluation of components. The use of IR transmitting fibers coupled to the camera was demonstrated and has great potential for temperature measurement within systems such as dies during liquid metal insertion.

TPUC developed a portable camera control capability to allow off-site fieldwork. The first demonstration of this portability was in a reverse HTML Fellowship with the Ford Scientific Research Laboratory. The purpose of this investigation is to simultaneously image hot spots on the inboard and outboard sides of a brake rotor during braking on a dynamometer. These hot spots result in localized thermal expansion of the rotor that cause the torque variation known as brake roughness or brake judder. During the three visits to Ford, the TPUC staff was able to record thousands of images of this hot-spotting phenomenon as a function of initial rotor temperature and speed, brake pad manufacturer and design, and braking behavior (i.e., dragging or stopping). In addition to the qualitative images, which show the spatial and time evolution of the hot spots, a calibration was performed that allowed the quantitative measurement of temperature as a function

of time and location.

These results have both short- and long-term benefits. In the short term, brake system components that are less likely to cause hot spotting can be selected for cars about to be introduced into the market. In the long term, the thermoelastic models that describe hot spotting may be refined and used to evaluate future designs before they go into production. Visits to GM Delphi Chassis Systems and the University of Southern Illinois are currently being planned for additional work on brake systems and materials.

A.4 Diffraction User Center (DUC)

Members who had prime responsibilities for activities in this User Center include the following:

Dr. E. A. (Andrew) Payzant—RT & HTXRD, neutron and synchrotron diffraction methods

Dr. C. J. (Claudia) Rawn—X-ray, & neutron diffraction, crystal chemistry

Mr. O. B. (Burl) Cavin—RT & HTXRD

Dr. R (Robbie) Peascoe—RTXRD and crystal chemistry

Dr. Jianming Bai—synchrotron diffraction

J. L. (Joy) Kilroy—group administrative support

Dr. C. R. (Cam) Hubbard—group leader, X-ray and neutron diffraction

Mr. Cavin, who had been with DUC since the beginning of the HTML, retired September 1993. Fortunately, he has continued to participate in the operation of the X-ray facilities by supporting users and assisting ORNL staff members. Dr. Payzant, formerly an ORISE postdoc in RSUC, became the lead staff member with prime responsibility for the DUC. Dr. Rawn joined the group as a postdoc and became an ORNL staff member in October 1998. Her expertise is in use of XRD for phase equilibria and solid state chemistry. Dr. Jianming Bai joined the Group as a postdoc and works at the X14A synchrotron beam line. Dr. Hubbard, whose primary research focus is X-ray and neutron diffraction method, fills out the Diffraction program's team.

The instrumentation in this Center has remained generally unchanged, except for major computer and software upgrades in FY 1990 and FY1998-FY1999. The spectrometers are now controlled by a Windows NT workstation system, and this advanced computer hardware has greatly improved the efficiency with which users can obtain and analyze their data either from the room temperature or the high-temperature spectrometers. In addition, we have known for some time that position sensitive detectors for the spectrometers would greatly accelerate the acquisition of diffraction data. An industrial user brought a detector of this type to the HTML for use in conducting measurements. These detectors were shown to be very powerful in detecting and measuring the characteristics of fast phase reactions at high temperatures. Fast reactions in aluminum nitride compositions during initial sintering have been studied at temperatures of the order of 1900°C in one atmosphere of nitrogen. To our knowledge, this is the first time such measurements have been made anywhere under these conditions. One of these detectors was purchased from a combination of FY 1992 and FY 1993 funds for the high-temperature spectrometer.

The major instruments in the DUC are:

Scintag Theta-2-Theta PAD V goniometer with Peltier cooled Si(Li) detector;
Scintag Theta-2-Theta XDS 2000 goniometer with graphite monochromator and scintillation detector;
Scintag Theta-Theta PAD X goniometer with Buehler high-temperature furnace system and choice of either a position-sensitive detector or a Peltier cooled Si(Li) detector;
High-resolution neutron powder diffractometer (HB-4) at HFIR with furnace and cryostats;
X14A beam line at the NSLS with Buehler high-temperature furnace system.

The neutron powder diffractometer facilities are part of the Neutron Scattering Research Facilities (NSRF) at HFIR sponsored by ER. HTML user projects that also require neutron powder diffraction are accommodated under a collaborative arrangement.

DUC is a member of the Participating Research Team (PRT) for the X14A beam line at NSLS. This beam line was constructed with funding from the DOE-ER, via ORNL, and industrial and academic members of the PRT. As a member of the PRT, HTML is conducting high-temperature diffraction and stress analysis as well as adding reflectometry and extended X-ray absorption fine structure (EXAFS) capabilities for research use by HTML users. The extremely high flux, 3 to 6 orders of magnitude greater than our laboratory-based systems, will enable users to study smaller specimens, to study rapid reactions and kinetics, and to resolve subtle phase transformations that cannot be determined with the laboratory-based systems. The X-ray optics of X14A will be upgraded in FY99 providing a 6-fold increase in flux and replacing some troublesome components.

A.5 Residual Stress User Center (RSUC)

The neutron residual stress facility (NRSF) is operated in a team mode, with members of the Metals and Ceramics, Solid State (SSD), and Instrumentation and Controls (I&CD) Divisions participating. Unless otherwise noted, the following staff members are from the Diffraction and Thermophysical Properties Group, Metals and Ceramics Division.

Dr. E. Andrew Payzant—neutron residual stress mapping, instrument specification;

Dr. Roberta (Robbie) Peascoe—texture;

Dr. Steve Spooner—residual stress mapping by neutron diffraction (SSD);

Dr. D. Q. (David) Wang—residual stress mapping and neutron transport modeling

Dr. Xun-Li Wang —macro residual stress by x-ray and neutron diffraction;

Dr. Thomas Ely —X-ray residual stress and grazing incidence diffraction;

Dr. Michael Wright—instrumentation design for neutron strain mapping (I&C Division);

Ms. Joy L. Kilroy—group administrative support; and

Dr. Camden R. Hubbard—group leader and NRSF project leader.

This User Center was created at the request of industrial users and provides an intense rotating anode X-ray source and a highly accurate, sophisticated X-ray spectrometer designed to measure the diffraction intensities necessary to compute the stress field in a solid surface. This instrument became operational on time in FY 1992, and for its first year, had a backlog of about six months of

users waiting to conduct their research. Due to this demand, an additional spectrometer with a sealed beam X-ray tube was purchased from FY 1993 funds in an attempt to alleviate the long delays being encountered by users to perform this particular research. A position sensitive detector and laser device for determining sample position were obtained in FY 1994 to further improve throughput of the rotating anode system.

During early operation of the X-ray instruments of RSUC, it became clear that many industrial researchers were very interested in being able to characterize the stress field inside complex components. This could only be done by means of neutron diffraction. This type of work had been done at the Chalk River facility in Canada where use is expensive, and opportunity is limited. The Canadian reactor flux is one fourth that of ORNL's High Flux Isotope Reactor (HFIR). Therefore, the effective resolution with which the details of the stress field could be determined was rather limited, except for very large objects. In FY 1991, an ORNL Laboratory Director's Research Development (LDRD) Fund project was funded to demonstrate that stress fields in solid objects could be more effectively measured using one or more of the neutron beam lines at the HFIR whose operation is funded by ER through ORNL's Solid State Division (SSD). Staff in the SSD teamed with HTML User Program staff to conduct this work. A special neutron spectrometer was constructed and installed on a beam line at the HFIR, and staff from the HTML RSUC and the SSD operated the facility and worked with users to conduct user research projects. The research team demonstrated the ability to measure the stress field inside metal weldments and inside ceramic to metal joints, for example, with a gage volume down to 1 mm³. In addition, the stress field inside whisker reinforced ceramic matrix composites was measured as a function of various microstructural variables. After successful completion of the project, a formal proposal to create a special Neutron Residual Stress Facility, which would be part of the RSUC, was approved and funding received in April 1994.

Today, major instruments in the RSUC include:

- Scintag PTS four-axis goniometer with 18-kW rotating-anode generator;
- Scintag PTS four-axis goniometer with 2-kW sealed-tube generator;
- neutron spectrometer with XYZ mapping system for macroresidual stress mapping;
- neutron powder diffraction spectrometer and furnace for microresidual stress analysis; and
- X14A beam line at the National Synchrotron Light Source (NSLS) [RSUC is a participating research team (PRT) member on this ORNL beam line].

Both of the neutron spectrometers are owned and managed by the Neutron Scattering Section of the Solid State Division, ORNL. RSUC is provided a portion of the time on these instruments to establish and operate the neutron residual stress effort based on an agreement between Department of Energy's (DOE's) Basic Energy Sciences (BES) and Energy Efficiency (OTT) programs and ORNL's Solid State and Metals and Ceramics Divisions. The long-term goal is to establish a dedicated stress mapping instrument at the High Flux Isotope Reactor (HFIR).

Accessory Instrumentation

Position-sensitive and Peltier Si(Li) X-ray detectors;
Laser position sensor for X-ray polycrystalline-texture-stress (PTS) units;
Grazing incidence optics;
Strain gage capabilities;
Tensile load frame for use on the neutron diffraction, residual stress mapping facility; and
Huber full-circle Eulerian cradle for use on the neutron diffraction, residual stress mapping facility
High-temperature furnace

Grazing Incidence X-Ray Diffraction (GXR)

Following an earlier demonstration of the application of GXR methods for depth-profiling residual stresses over a very shallow, near-surface region of specimens, the user interest in this technique has exceeded the capacity at which we can make measurements on our laboratory systems. Numerous proposals and inquiries related to grinding-induced subsurface stresses and to stresses in thin coatings have been received. GXR employs low angles of X-ray incidence to control the depth of X-ray penetration. By approaching the angle of total external reflection, the depth of sampling can range from tens of angstroms to a few micrometers. With this technique, diffraction information about phase content, texture, and residual stress can be obtained as a function of sampling depth.

Of particular importance is the nondestructive measurement of strains induced by grinding and the range of depth profiling. RSUC continues to pursue projects to provide a scientific basis for aspects of this technique and to support further development of this technique and instrumentation. The particular limiting aspect is slow data collection, which limits the extent of user proposals we can address and further limits the number of specimens that can be studied. To address this shortfall we have assessed several options to increase the flux, instrument time, or develop alternate instrument access. These include multilayer X-ray mirrors, focusing capillary optics, and synchrotron radiation. We demonstrated that a 30-fold enhancement could be achieved with use of a multilayer X-ray mirror. A capillary optics device did not look promising for this application. Tests using the ORNL X14A beam line at NSLS have been encouraging, and additional testing and optimization of the facilities there are under way. An order was placed for X-ray mirrors in FY99, and they should be operational in FY2000.

Residual Stress Facility

A cooperative joint effort—signed in April 1994 between EE, OTT, and ER/BES, Division of Material Sciences—established NRSF. As part of the HTML's RSUC, NRSF is a collaborative effort to meet expressed industrial and academic needs for through-thickness strain mapping. This joint program is a direct result of a multiyear LDRD project proposed and led by HTML staff at HFIR. NRSF is operated by a team consisting of staff from the HTML in the Metals and Ceramics Division, the Neutron Scattering Group of the Solid State Division, and the Instrumentation and Controls Division.

The goal of the joint program is to establish and operate user facilities for macro- and microresidual stress analysis in conjunction with the existing RSUC X-ray facilities in HTML and the neutron scattering facilities program at HFIR. We currently are meeting this goal by using a portion of the beam time on two Solid State Division spectrometers (HB-2 and HB-4). Another goal is to develop a dedicated instrument at HFIR optimized for macroresidual stress analysis, and thus to greatly expand both the capabilities and speed of measurement.

A.6 Machining and Inspection Research User Center (RSUC)

Members who have prime responsibilities for activities in this User Center include the following:

S. B. (Sam) McSpadden, leader of the Machining and Inspection Research Group, has overall responsibility for MIRUC. Other group members and the equipment and technologies for which they have primary responsibility are as follows:

Peter Blau – a nationally recognized expert in the field of Tribology (friction, wear, and lubrication). Dr. Blau is responsible for all tribological research performed in MIRUC. Numerous

custom-designed friction and wear testing instruments, scratch testers, and repetitive-impact testers are now available for use by guest researchers.

Tyler Jenkins – technical support technician responsible for the operation of the Harig surface grinders; coolant management; health, safety, and environmental issues.

Tom Morris – a technical specialist in grinding and ceramics machining. Mr. Morris is the principal investigator on a CRADA with Cummins Engine Company's Fuel Systems Division, which involves cost-effective machining of ceramics. Mr. Morris is an employee of the Oak Ridge Y-12 Plant, on loan to the HTML.

Lawrence O'Rourke – technical support technician responsible for operation of the Weldon cylindrical grinder, Nicco creep-feed cylindrical grinder, grinding wheel management, and procurement. Mr. O'Rourke is an employee of the Oak Ridge Y-12 Plant, on loan to the HTML.

Ron Ott – recently received his PhD from The University of Alabama at Birmingham. He has a broad technical knowledge of ceramic grinding processes, especially creep-feed and surface grinding. His efforts are currently divided between tribological research and grinding activities. He is also responsible for the grinding elements of a cooperative research and development agreement (CRADA) with Caterpillar, Inc.

Randy Parten – technical support technician responsible for dimensional metrology, with emphasis on programming and operation of the coordinate measuring machine (CMM), Mahr form tester, Taylor-Hobson Talysurf surface profile measuring instrument, and the Rodenstock non-contact laser surface topography measuring instrument.

Earl Shelton – technical support technician responsible for operation and CNC programming of the Cincinnati Milacron centerless grinder and the Sabre vertical grinding center.

(All technical support technicians are knowledgeable about the operation of all grinding machines that are available in our group.)

Jessie Whittenbarger – group administrative support.

Six types of numerically controlled grinders are available to guest researchers for their projects at MIRUC. The grinders were selected for their similarity to those used in manufacturing facilities throughout the United States. Grinders are instrumented to permit real-time measurement of key grinding process parameters including grinding forces, spindle power, spindle vibration, acoustic emission, and coolant temperature. Data may be collected, displayed, stored, and analyzed using specialized LabView™ programs and other analysis software. Major grinding equipment includes:

- Weldon Cylindrical Grinder
- Bridgeport/Harig Surface Slicer/Grinder
- Nicco Creep-Feed Surface Grinder
- Cincinnati Milacron Twingrip™ Centerless Grinder with high-speed spindle*
- Cincinnati Milacron Sabre™ Multiaxis Grinder with high-speed spindle*
- Trihedral tripod grinder with ultrasonic spindle

* These two pieces of equipment are owned by Milicron, Inc. and Cincinnati Tool, Inc., respectively. They were made available to the HTML and its User Program on a consignment basis.

Other available grinding equipment and related software include:

- Ded-Tru™ centerless grinding attachment for use with surface grinders to grind small components
- Compact Grindability Test System
- SmartCAM™ Production Turning Package
- SmartCAM™ Production Milling Package
- SurfCAM™ advanced 3D Computer Aided Manufacturing (CAM) software
- AutoDesk™ Mechanical Desktop with AutoCAD™
- AutoCAD™ LT 98 Windows-based 2D CAD software
- DADiSP™ (data analysis and display software)
- Statistica™ (statistical analysis software)
- ReLink software from ControlLink Systems, for analysis of Labview-generated datasets

The center also maintains state-of-the-art dimensional and surface texture measuring equipment for use by guest researchers, including:

- Electronic Measuring Devices (EMD) Legend™ Integrated Metrology Center, a precision coordinate measuring machine (CMM) with both contact and non-contact scanning capability
- Mahr/Perthen Formtester™
- Taylor-Hobson Talysurf™ Model 120 Stylus Surface Texture Measurement System with recently upgraded Windows-based controller and analysis software.
- EOIS Mini-moiré Sensor
- Nikon Optical Comparator
- Gage-Master Optical Comparator
- Rodenstock RM600 Laser Surface Texture Measurement System

Our technical staff is available to assist researchers in the operation of the more complex equipment, such as the centerless grinder, multi-axis grinder and the coordinate measuring machine. Our equipment is computer controlled, and inspection data can be easily exported to advanced analysis software and CAD/CAM software using a laboratory-wide network.

The group also operates two tribology laboratories. Physical testing and material analysis constitute a major portion of the work in these laboratories. Experiments are designed to screen materials, effect simulations of components, or study the basic relationships between the microstructures and compositions of surfaces and their friction and wear behavior. Available machines fall into three categories: (1) commercially developed testing machines, (2) machines designed under subcontract, and (3) machines designed and built by ORNL for special purposes. Most of the testing machines are aimed at sliding wear, but abrasive wear, impact wear, and rolling-contact wear tests are also available. Tribology testing at high temperatures and controlled atmospheres is also within the capabilities of the user center. Major tribology equipment includes:

- Reciprocating Friction and Wear Tester
- Repetitive Impact Testing System
- Friction Microprobe
- Cameron-Plint TE-77 Reciprocating Sliding Wear Tester
- Instrumented Scratch Tester
- High-Temperature Pin-on-Disk System

- Multimode Friction and Wear Tester
- Micro-Abrasive Wear Tester
- Sather Lubricant Load-Carrying Capacity Screening Rig
- Teledyne-Taber Portable Scratch Tester
- Wilson Microindentation Hardness Tester
- Talysurf 10 Surface Roughness Measuring System

Dedicated Workstation Acquired for Users' Office Area – An office area has been set aside for the use of visiting researchers who are participating in ceramic machining and inspection research under the HTML User Program. The office has been equipped with a PC-based workstation suitable for CAD/CAM applications and analysis of data collected during the grinding process. The SurfCAM™ CAD/CAM software package was acquired to provide a quick and simple means of generating complex numerically controlled tool paths for the grinding equipment.

Further Improvements Made to Compact Grindability Test System – The compact grindability test system, developed by Chand Kare Technical Ceramics, uses a diamond abrasive belt to measure the relative grindability of ceramic specimens. New instrumentation has been added to permit accurate calibration of grinding force, belt speed, and coolant temperature. A dedicated computer was added to the system and a customized database application was written for collecting grindability data.

Taylor-Hobson Talysurf Upgraded – The Taylor-Hobson Talysurf Model 120, Series I, surface texture measuring instrument was originally purchased with a DOS-based computer, which contained proprietary control components. Some of these components proved to be unreliable and subject to frequent repair. The computer sub-system was upgraded with a Windows-NT computer and new control hardware. The new, Windows-based analysis software is also a vast improvement over the old software.

EMD Legend Coordinate Measuring Machine (CMM) Upgraded – The performance of the CMM has been dramatically improved by upgrading to a Pentium-class computer with additional memory. During the upgrade process, the CMM was recalibrated and certified to standards that are traceable to NIST, and the latest version of analysis/control software was installed.