High Temperature Materials Laboratory

HTML

The six user centers in the High Temperature Materials Laboratory (HTML), a DOE User Facility, are dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The HTML user program provides researchers from U.S. industries, universities, and federal laboratories with access to expert staff and to sophisticated, often one-of-a-kind instruments for materials characterization.

User Support

Instruments available at the six user centers have extensive capabilities for characterizing the microstructure, microchemistry, and physical and mechanical properties of materials over a wide range of temperatures.

Capabilities

- Diffraction (x-ray and neutron)
- Friction and wear testing, inspection
- Mechanical characterization
- Microstructural analysis
- Residual stress measurements
- Thermography and thermophysical properties determination

Professional Staff

HTML staff provide assistance to users and have the following responsibilities:

- Participate in the planning, performance, and interpretation of research, including co-preparation of technical papers and presentations from nonproprietary research;
- Assist users in the safe and efficient operation of user center research equipment;
- Maintain instruments to maximize their availability for research.

Facility Access

Before research begins, two documents must be in place:

- Standard user agreement between the user organization and UT-Battelle, LLC
- Approved research proposal

Proposals

Both nonproprietary and proprietary research is conducted within the user program. Prospective users must submit a research proposal for review and should consult with the appropriate center's staff concerning specific plans and time schedules for inclusion in proposals. Selection of proposals is based on the compatibility of the proposed research with the missions of both the HTML and DOE.

The proposal form is available for download at http://html.ornl.gov.

Nonproprietary

Nonproprietary research at the HTML is usually conducted at no direct cost to the user, although charges are assessed for special services. The HTML Proposal Review Committee meets monthly to evaluate proposals and to make recommendations on their acceptance. It also recommends priorities for use of instruments within HTML user centers, consistent with DOE missions and objectives. Applicants are notified of the committee's decision by e-mail.

Users conducting nonproprietary research must agree to submit research results for publication in the open, refereed literature no more than six months after research is concluded. Papers and presentations must be prepared jointly by users and HTML staff.

Proprietary

All proprietary research costs at the HTML are paid by the user. Fees are based on DOE guidelines for ORNL costs and must be paid prior to the beginning of research. Special care is taken to protect proprietary information.

HIGH TEMPERATURE MATERIALS LABORATORY

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User Centers and Representatives

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- X14A beamline at National Synchrotron Light Source

Materials Analysis User Center
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- Remote microscopy
- Scanning auger microprobe
- Hitachi HF-3000 FEG-STEM

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- Flexure test facility
- Nanoindenter

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- Neutron residual stress facility

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- Thermal transport
- Thermal analysis
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- Friction-reducing surface treatments
- Materials for wear-critical uses
- Subsurface defect structure
- Topographic imaging

Diffraction User Center

DUC

The Diffraction User Center (DUC) is part of the High Temperature Materials Laboratory (HTML), which is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The DUC provides world-class facilities and a staff of technical experts for determining the structure of materials, using x-ray, synchrotron, and neutron diffraction techniques.

Multipurpose X-Ray Powder Diffraction

This instrument provides either divergent- or parallel-beam optics coupled with high-temperature stages.

- Philips X´Pert Pro vertical θ/θ goniometer
- Cu or Cr x-ray targets
- Anton Paar XRK 900 high-temperature (HT) furnace with rotating sample stage (RT to 900°C)
- Anton Paar HTK16 HT furnace Pt strip heater (RT to 1600°C)
- Multipurpose RT sample stage
- Proportional detector or position-sensitive detector
- X´Pert Plus Crystallography and Rietveld software

Applications

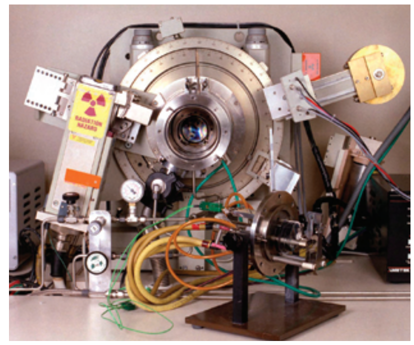
- HT phase transformations
- HT residual stress using parallel beam optics
- Thermal expansion using parallel beam or divergent optics
- Phase equilibria studies

High-Temperature X-Ray Powder Diffractometer

- Scintag PAD X vertical θ/θ goniometer
- Cu, Mo, Cr, Co, Ag, and Fe x-ray targets
- Buehler HDK 2.3 furnace with Pt, W, Ta, or Mo strip heaters (RT to 1600°C , or 2200°C+ in vacuum)
- Controlled environment (O_2, Ar, N_2, H_2, ambient, etc.), 1 atm , pO_2 sensing
- Peltier Si(Li) energy-dispersive detector
- Position-sensitive detector (rapid data acquisition)

Applications

- *In situ* process simulation
- Lattice thermal expansion coefficients
- Oxidation/reduction kinetics
- Phase equilibria studies
- Order/disorder transformations
- Rietveld analysis



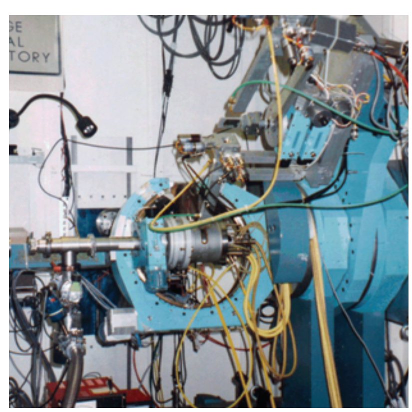
This instrument combines divergent optics with a high-temperature stage and atmospheric control.

High-Temperature Synchrotron X-Ray Diffractometer at the National Synchrotron Light Source

- Six-circle Huber diffractometer with split chi-ring; crystal analyzers (Si, Ge, LiF, graphite); CAMAC multichannel analyzer; pinholes > 10 mm diam; scintillation and proportional counters
- Focusing incident beam silicon monochromator
- Tunable wavelengths from 0.5 to 2.5 Å
- Buehler HDK 2.3 furnace with Pt, W, Ta, or Mo strip heaters (RT to 1600°C , or 2200°C+ in vacuum)
- Controlled environment (O_2, Ar, N_2, H_2, ambient, etc.), 1 atm
- Rotating capillary mount and optional furnace

Applications

- Oxidation/reduction kinetics
- Phase equilibria studies
- Order/disorder transformations
- Single-crystal diffraction
- Rietveld analysis



This high-flux beamline instrument is designed to permit the measurement of precise lattice parameters.

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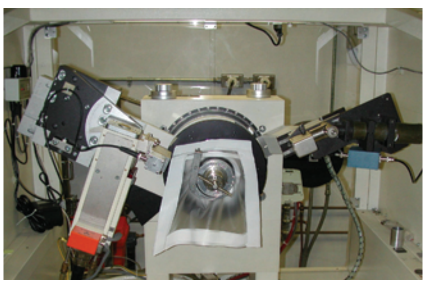
Low-Temperature X-Ray Powder Diffraction

- Scintag XDS2000 vertical θ/θ goniometer
- Cu, Mo, Cr, Co, Ag, and Fe x-ray targets
- Displex closed-cycle He refrigerator (10 K to 300 K)
- Cold sample loading
- Diffraction side graphite monochromator and scintillation detector

Applications

- *In situ* process simulation
- Lattice thermal expansion coefficients
- Oxidation/reduction kinetics
- Phase equilibria studies
- Order/disorder transformations



This instrument combines divergent optics with a displex closed-cycle He refrigerator.

High-Temperature Neutron Powder Diffractometer at the High Flux Isotope Reactor

- High-resolution 32-detector array
- Sensitive to light elements (H, C, O, S, etc.)
- Bulk diffraction rather than near-surface
- Temperatures up to 1600°C in vacuum furnace
- Temperatures from 2 K in He cryostat

Applications

- Phase equilibria studies
- Order/disorder transformations
- Rietveld analysis
- Microstress analysis in composites

Room-Temperature X-Ray Powder Diffractometer

Features

- PANalytical X´Pert Pro MPD vertical θ/θ goniometer
- Cu x-ray target
- Programmable slits
- X´Celerator RTMS detector with optional monochromator
- Single- or 15-position sample holder

Applications

- Structure refinement
- Lattice parameter refinement
- Quantitative phase analysis
- Crystallite size and microstrain determination

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Materials Analysis
User Center

MAUC

The Materials Analysis User Center (MAUC) is part of the High Temperature Materials Laboratory (HTML), which is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The MAUC provides world-class facilities and a staff of technical experts for characterizing the structure and chemistry of advanced materials. A special emphasis is using these tools to relate microstructure to materials performance.

The MAUC comprises a suite of labs that contain the latest-generation electron microscopes and surface analysis instruments, all of which are available to visiting researchers. Research specialties include characterization of nanophase materials such as catalysts, carbon nanotubes and nanoparticulates; structural ceramics; electron holography (e.g., for dopant profiling in semiconductors); and characterization of multilayer surface films.

MAUC Instruments

Field-emission transmission electron microscopy (FE-TEM) and aberration-corrected scanning transmission electron microscopy (STEM) instruments allow imaging and chemical microanalysis to the atomic level. The latest generation aberration-corrected electron microscope provides sub-Ångström imaging capability, e.g., for imaging single atoms in catalytic materials. Surface analysis is provided by a field-emission scanning Auger nanoprobe. The electron microprobe allows quantitative chemical analysis of bulk microstructures at the sub-micron level. An environmental SEM allows characterization of surface morphology and chemistry. The most modern specimen preparation techniques are available, utilizing instruments such as a focused-ion-beam miller with microsampling capability; a cryo-ultramicrotome; other ion-milling instruments; and assorted slicing, grinding, and polishing devices.

Analytical Instruments and Support Equipment

- JEOL 2200FS-AC aberration-corrected STEM/TEM with in-column Omega energy filter
- Hitachi HF-3300 TEM/STEM
- Hitachi S-3400 variable-pressure SEM with EDS
- JEOL 8200 5-spectrometer electron microprobe with JEOL EDS
- PHI 680 FE-scanning Auger nanoprobe
- Fischione low-voltage ion-beam miller
- Fischione Nano-mill
- Hitachi FB-5000 dual-beam focused-ion-beam miller with microsampling capability

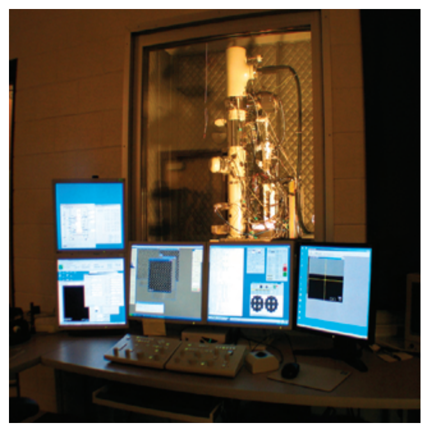
Hitachi HF-3300 TEM/STEM

- Cold field-emission electron source
- 0.19-nm point resolution in TEM; 0.2-nm in STEM
- Sub-nanometer spatial resolution for x-ray elemental analysis
- 2 Möllenstedt biprisms for advanced electron holography capabilities
- Energy-dispersive spectroscopy
- All-digital image acquisition and microscope remote control
- Available for remote microscopy

HF-3300 field-emission TEM/ STEM basic instrument

Aberration-Corrected Electron Microscope

The MAUC has installed the JEOL 2200FS-AC aberration-corrected STEM/TEM instrument (the "ACEM") in the new Advanced Microscopy Laboratory, located adjacent to the HTML. The instrument provides a nominal probe diameter of 0.7Å, with simultaneous annular dark-field and bright-field imaging in STEM mode. An in-column energy filter allows chemical species and bonding information to be obtained from single atomic columns. An information limit for TEM of 0.9Å has also been demonstrated.



View of the ACEM from the control room, showing monitors used for instrument control and aberration corrector operation.

Catalyst Characterization

MAUC personnel have experience in characterizing the structure and microchemistry of a wide variety of catalytic materials. We have imaged structures such as five-atom osmium clusters on MgO crystals, tri-rhenium carbonyl clusters on alumina, atoms and clusters of Pt on a variety of oxide supports, and bimetallic nanoparticulates such as Au-Pd, Pd-Zn, Pt-Fe and Pt-Co.

In Situ Electron Microscopy Capabilities

The ACEM and HF-3000 microscopes have available the latest *in situ* heating and environmental cell holders using Protochips Co. technologies for treatment of catalyst and other materials at elevated temperatures and under gas environments. The ACEM also provides a capability to treat specimens at room temperature and at atmospheric pressure in a gas, using a unique airlock reaction system. An *ex situ* reactor system is also available for special needs involving sample treatments in corrosive gas environments.

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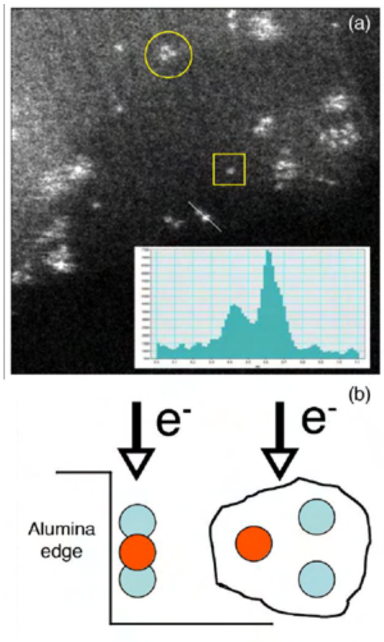
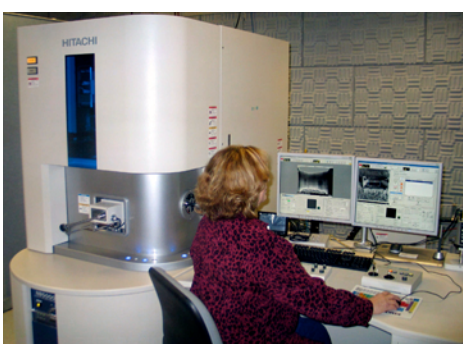


a) Dark-field image of tri-rhenium clusters on g-alumina. 3-atom cluster in circle; single atom in square. Apparent 2-atom cluster (arrowed) shows intensity profile inset. Brighter atom has double the intensity of dimmer atom, suggesting illustration in (b). This 3-atom cluster is edge-on to beam.

Special Group Capabilities

The MAUC offers a number of special capabilities, such as dual-biprism electron holography in the HF-3300 TEM, which allow the image phase information to be obtained from thin sample regions. This permits determination of the shapes of nanotube cross sections, the profile of dopants in p/n junctions, and the shape of electric and magnetic fields surrounding particles and structures. The Hitachi FB-5000 dual-beam FIB instrument also provides high-resolution SEM imaging, EDS for elemental analysis, and a backscattered electron detector for orientation imaging. Digital imaging on all instruments provides the apability to access instruments for remote microscopy research sessions.



Hitachi FB-5000 dual-beam focused ion beam (FIB) miller allows in-situ *microsampling for TEM specimen preparation and milling for micromachining at the submicron level. MAUC staff member D. Coffey at the controls.*

MAUC Staff

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Mechanical Characterization and Analysis User Center

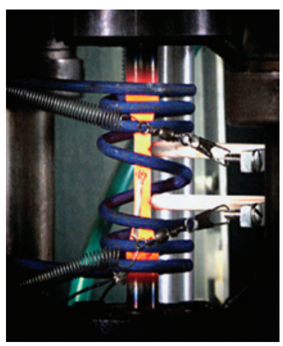
MCAUC

The Mechanical Characterization and Analysis User Center (MCAUC) is one of six user centers in the High Temperature Materials Laboratory (HTML), which is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The MCAUC provides world-class facilities and a staff of technical experts for the mechanical characterization of functional and structural materials.

The MCAUC performs mechanical testing and analysis and develops test methods and supplemental analytical techniques. Numerous mechanical test frames with uniaxial and multiaxial capabilities are available to visiting researchers from industry and academia to conduct tests in tension, compression, flexure, torsion, shear, and internal pressurization in controlled environments over a wide range of temperatures and strain rates using standard or customized specimens. Facilities also include equipment for micromechanical testing and instrumented indentation. Staff has expertise with a wide range of materials, testing configurations, analytical modeling, stress, failure and life-prediction analysis of materials and structures.

Universal Test Facility

- Electromechanical and servohydraulic testing machines with load capabilities of up to 500 kN for monotonic or cyclic tests in tension, compression and/or torsion
- Actuator speeds up to 18 m/s and testing frequencies of 3000 Hz
- Load train configurations with fixed or self alignment
- Active and passive grips for a wide array of specimen geometries
- Digital controllers for load, displacement, or transducer-driven control, and computerized data acquisition



Dynamic Mechanical Analysis

- Dynamic mechanical analyzer with capabilities from -70°C to 600°C in air or controlled environment

Test Machine for Automotive Crashworthiness (TMAC)

- World's first integrated virtual and physical test system for high-force, high rate crashworthiness experiments on materials and structures
- Hydraulic system with load capacity of 267 kN dynamic, 490 kN static force at impact velocities greater than 8 m/s (18 mph)

Resonant Ultrasound Spectroscopy (RUS) Facility

- Characterization and inspection of mechanical integrity of test specimens and components
- Determination of the elastic constants of isotropic and anisotropic materials as a function of temperature and environment

Environmental Testing

- Environmental chambers for testing in inert environments, vacuum or pressurized simulated environments (e.g., steam) over a wide range of temperatures
- Infrared, induction and resistance heating

Mechanical Properties Microprobes

- Nanoindenters capable of operating at various load ranges and up to 400°C with constant monitoring of load and position of indenter
- Spherical, flat, and sharp indenter tips

Raman Spectroscopy

- Measurements of stress, phase content, and structure in
- nonmetallic materials with a spatial resolution of ~2 μm
- Laser and optics optimized for operation in UV, which
- allows for Raman spectral measurements up to 1500°C
- Loading fixture for in situ measurements and fiber optics
- for remote sampling

Fourier Transform Infrared (FTIR) Spectroscopy

- Optical spectrometer for FTIR with a range of 7,400–350 cm^{-1} and spectral resolution of 0.125 cm^{-1}
- System attached to an IR microscope with spatial resolution of ~10 μm

Fixtures, Accessories, and Special Configurations

- Fixtures for uniaxial and biaxial bending, Iosipescu shear testing, anti-buckling compression, and double-torsion for determination of fracture toughness

Additional MCAUC Capabilities

- Infrared imaging
- High-speed video extensometry
- Acoustic emission system
- Strain-gaging facilities
- Internal pressurization of tubular components

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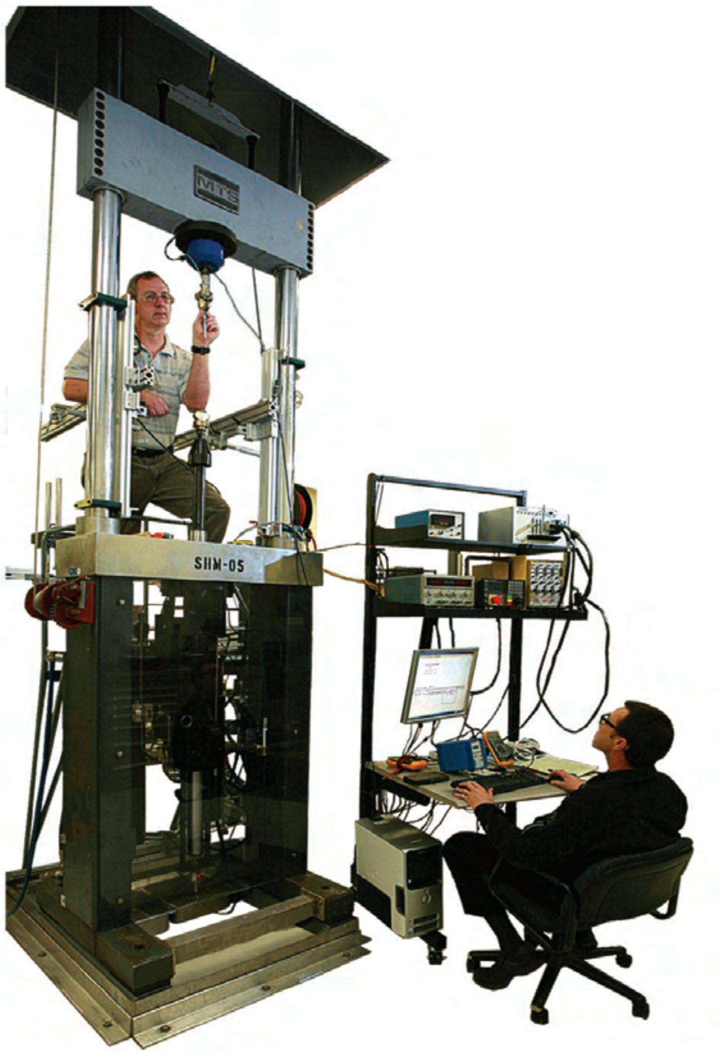

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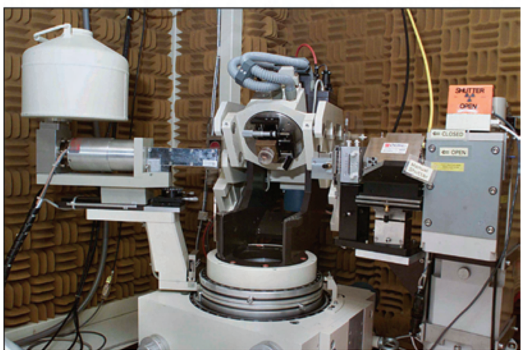
Residual Stress
User Center

RSUC

The Residual Stress User Center (RSUC) is one of six user centers at the High Temperature Materials Laboratory (HTML), which is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The RSUC provides world-class facilities and a staff of technical experts for characterizing both surface and through-thickness stresses with state-of-the-art x-ray, synchrotron, and neutron diffraction facilities.

Residual stresses affect important properties such as fatigue life, fracture strength, onset of yield, and microcracking. X-ray and neutron diffraction methods are available in the RSUC to measure macro- and micro-residual stresses in polycrystalline and single crystal materials ranging from small specimens (thin films) to large industrial components.

Four-Circle Goniometers with18-kW Rotating Anode X-Ray or 2-kW X-Ray Tube Sources



X-ray diffraction permits measurement of texture and either biaxial or triaxial residual strains in ceramic and metal alloy surfaces. These instruments are designed to permit mapping of the residual stress state across the surface of a specimen and as a function of depth with electropolishing.

Common Features

- Scintag PTS four-axis goniometer for stress and texture analysis with unrestricted 2θ range (from –2 to +162°)
- Highly automated, flexible data-collection options (Ψ tilt or Ω tilt and multiple pole figure
- Solid-state detection of x-rays yields high peak-to-background ratios and excellent sensitivity
- Specimen dimensions up to 140 mm in diameter, 40 mm in thickness, and 5 kg in mass
- Software includes biaxial and triaxial stress analysis, pole figures, and orientation distribution function calculations from texture data
- Load frames for determination of diffraction elastic constants and hot
- Grazing incidence x-ray diffraction for depth profiling of stress and phases
- Parallel-beam optics for characterization of curved or irregular surfaces

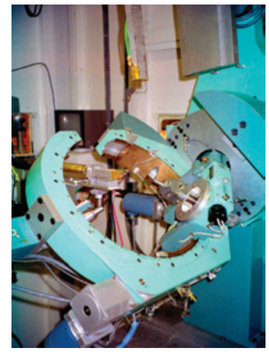
Cu Rotating Anode

- High-flux, high-brilliance rotating anode source
- Copper target
- Incident parabolic mirror and radial-divergence-limiting slits for true parallel beam optics

Tube Source

- Interchangeable 2-kW x-ray tubes
- Divergent-beam and near-parallel-beam optics

X-14A Synchrotron Beamline at the National Synchrotron Light Source

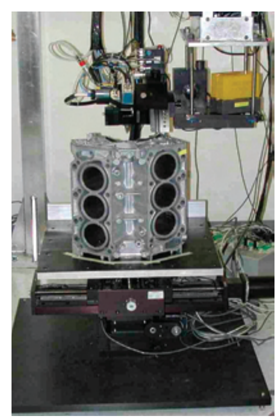


This high-flux beamline instrument is designed to permit the measurement of residual stress in a variety of ways, including grazing incidence at high temperature.

Features

- High-flux, high-brilliance source (9×10^{11} photon/s at 8 keV, 223 mA, 2.5 GeV)
- Tunable x-ray source (energy range 3.5–18 keV; energy resolution $\Delta E/E = 2 \times 10^{-4}$)
- Parallel x-ray source (total horizontal angular acceptance: 5 mradians)
- Six-circle Huber diffractometer with split chi-ring; crystal analyzers (Si,Ge, LiF, graphite)
- Pinholes > 10 μm diam
- Displex cryostat
- Buehler high-temperature stage
- Hemispherical Be dome furnace

Large Specimen X-Ray Diffractometer



This instrument allows x-ray examination of large specimens. Measurement of residual strain in ceramics and metal alloys and determination of retained austenite in ferrous alloys can be performed.

Features

- Cu, Co, and Cr x-ray targets
- Computer-controlled XY mapping of surface
- $Sin^2Ψ$ residual stress and retained austenite analyses
- Sample fixturing able to be custom-designed to accommodate needs
- 120-ft^2 x-ray enclosure
- Gantry system providing overhead translation of x-ray source and goniometer
- Two automated XY sample translation stages for large and small specimens

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Neutron Diffraction Residual Stress Mapping Facility

Neutron diffraction permits measurement of triaxial residual strains within the bulk of alloys and ceramics. Diffraction elastic constants are used to convert strains to stresses. The high flux at HFIR permits mapping through many millimeters thickness of most materials.

Features

- Highest flux thermal neutron source in United States
- Selectable wavelength with multiwafer doubly focusing monochromators
- Position-sensitive 7-detector array
- Specimen-positioning equipment
  - X, Y, and Z translations
  - Specimen rotations
  - Large capacity (1000 lbs)
- Peak position measurement to ±0.003° 2θ
- 3-D strain mapping
  - Gage volume of <1 to 40 mm^3
  - Automated data acquisition
  - Real-time data analysis
- Triaxial strain measurement and stress analysis
- Load frame for uniaxial loading to 15 kN
  - Fracture mechanics strain mapping under load
  - Diffraction elastic constants
  - Anisotropic intergranular response
  - Multiphase response to load of composites

Micro Residual Stress Neutron Powder Diffraction Facility

This high-flux instrument permits the determination of the grain-to-grain residual stresses in multiphase samples.

Current Features

- Phase sensitive
- Monochromatic beam
- 32 ^{3}He detectors with Soller slits
- Pattern from 10 to 130° 2θ
- High resolution ($\Delta d/d = 2 \times 10^{-3}$)
- Room for sample enclosures and attachments
  - High-temperature furnace (1600°C in vacuum)
  - Controlled atmosphere furnace
- Rietveld analysis codes
- Powder diffraction analysis codes

Scan Arm and Laser Tracker

Metrology and reverse engineering of samples can be achieved with the Laser Tracker and Scan Arm. With associated software packages (CAM2 and GeoMagic Studio) the instruments can be used to generate “as-is” engineering drawings for comparison with the design drawings and identification of locations of distortion. The instruments are also used to pre-align specimens and pan neutron diffraction strain mapping experiments using the SScanSS visualization software.

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Thermography and Thermophysical Properties User Center

TTPUC

The Thermography and Thermophysical Properties User Center (TTPUC) is one of six user centers in the High Temperature Materials Laboratory (HTML), which is a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The TTPUC provides world-class facilities and a staff of technical experts for both determining thermophysical properties such as thermal conductivity, diffusivity, expansion, or specific heat; and also characterizing thermal stability, high-temperature reactions and compatibility, and high temperature oxidation and corrosion properties.

Materials studied at the TTPUC include metals, ceramics, superalloys, glasses, sand, paper, thermal barrier coatings, carbon materials, carbon composites, ceramic composites, metal matrix composites, and thick and thin films. In addition, high-performance infrared (IR) cameras are used in various thermography applications, including mapping temperatures and properties, monitoring processes, and nondestructively evaluating materials and structures. The thermography capability is portable and may be used for offsite user projects.

Thermal Analysis Facilities

Simultaneous Thermal Analysis (STA)

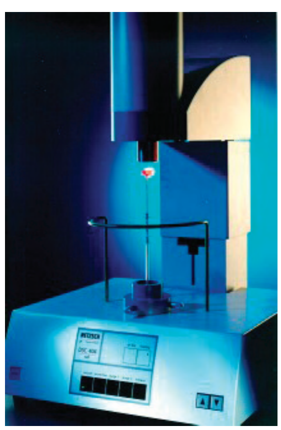
The STA technique is comprised of both differential thermal analysis (DTA) and thermogravimetry (TG). STA can be used to follow the course of chemical reactions, thermal decompositions, or phase changes as a function of temperature, heating rate, and atmosphere.

Features

- DTA and TGA measurements from 25 to 1450°C
- All systems fully computer controlled

Differential Scanning Calorimetry

The operation of a differential scanning calorimeter (DSC) is based on measurement of the thermal response of an unknown specimen as compared with a standard when the two are heated uniformly at a constant rate. A DSC is used to measure specific heat capacity and heats of transition, and to detect phase changes and melting points.



Features

- Operation of instrument completely automated and computer controlled
- Small sample volumes required (~1 to 50 mm^3)
- Widely accepted method for determining specific heat capacity

Netzsch DSC 404C

- Specific heat measurements to 1200°C
- Operation in air, inert gas, or vacuum to 1650°C

TA Instruments Q2000 DSC

- Operation in inert gas from - 180°C to 400°C
- Automatic sample changer
- Moduated DSC
- Pressure cell for pressures to 7MPa (1000 PSI)

High-Temperature, High-Mass Thermogravimetric Analyzer

This instrument is used to examine the time-temperature-environment relationships for high-temperature materials. The ability to accommodate large samples allows the testing of real components in aggressive environments, simulating actual processing or operating conditions.

Features

- 20–1700°C operation
- TG balance capacity up to 100 g with sample diameters up to 30 mm
- Mass change determinations to ±1 μg
- Heating rates up to 100°C/min
- Controlled environments, including vacuum, inert, and corrosive gases
- All systems fully computer controlled

Dual-Push-Rod Dilatometer

A differential dual-push-rod dilatometer measures the linear thermal expansion from 20 to 1600°C and can be used to detect various physical or chemical changes such as phase transitions in a specimen.

Features

- Measurements from 20 to 1600°C
- Differential length changes measured to ±1.5%
- Measurements in both inert gas and vacuum oxidizing environments
- Dedicated computer control of time-temperature programs for the samples
- Length change data automatically acquired, analyzed, and plotted

Quench Dilatometer

A quench dilatometer measures the change in length of a specimen as it is heated or cooled at programmed rates or held isothermally for a programmed time. It can be used to detect various phase transitions in a specimen such as equilibrium and metastable transitions in steels (development of CCT and ITT curves), and precipitation in age-hardenable aluminum and magnesium alloys.

Features

- Rapid heating at controlled rates in vacuum using RF induction coil
- Rapid cooling at controlled rates in vacuum or by forced helium assisted quenching
- Quench rates in excess of 150°C/s obtainable
- Option of deformation of specimen prior to cooling

Thermography: IR Cameras

High performance IR cameras are used in the following areas:
- Generating thermal diffusivity maps of bulk materials, especially composites
- Temperature mapping of devices, components, human, and biological objects
- Nondestructive evaluation of materials and components
- Process monitoring, high-speed imaging, and machine-vision application

Near-IR (0.9–1.7 µm) Camera features
- Snapshot mode
- 320 x 256 pixel InGaAs focal plane array (FPA)
- Frame rate up to 346 Hz (full frame) and 38 kHz (line)

Mid-IR (1.5–5 µm) Camera features
- Snapshot mode
- 320 x 256 pixel InSb focal plane array (FPA)
- 0.012 K temperature resolution
- Frame rate up to 346 Hz (full frame) and 38 kHz (line)
- IR microscope lens: 1.4 x 1.4 mm field of view, 5.4 µm per pixel
- Hyperspectral lens (3–5 µm)

Long IR (7–14 µm) Camera features
- Compact size (4.3 cm x 4.3 cm x 10.7 cm)
- 160 x 128 pixel FPA microbolometer
- Frame rate: 30 Hz
- 0.1 K temperature resolution

Thermal Transport Facilities

The TTPUC offers several techniques for the measurement of thermal transport properties numerous classes of materials over a wide temperature range. In addition, electrical resistivity and Seebeck coefficient are measured up to 1000°C.

All instruments have been used in the study of factors affecting the thermal transport properties of materials.

Flash Thermal Diffusivity System
Flash thermal diffusivity systems provide for studies at a wide range of test conditions. The three systems are a multiple-station laser flash system, a xenon flash system, and an IR-camera-based diffusivity mapping system.

Multiple-Station Laser Flash Diffusivity System
- Temperature range: cryogenic to 2500°C
- Bulk and layer sample measurements
- Vacuum or inert atmospheres
- Six samples per run measurement capability
- Thermal contact resistance between two layers
- Finite pulse-width and heat-loss corrections

Xenon Flash Diffusivity System
- Optimized for rapid room-temperature thermal diffusivity measurements
- Measures bulk and layer samples
- Accepts a wide range of specimen sizes and shapes

Hot Disk System
This technique measures thermal conductivity. The hot disk sensor is usually sandwiched between two pieces of sample during measurement. The sensor consists of an electrical conductor in the shape of a double spiral laminated between two thin sheets of insulating material (Kapton or mica). The sensor is used both as a heat source and as a dynamic temperature sensor.

LaserPIT System
This technique measures in-plane thermal diffusivity of a wide range of materials, including CVD diamond, metals, ceramics, glasses, plastics and thin film coatings on a substrate. Typical sample size is 30 mm long, 2.5 to 5.0 mm wide, and 3-500 µm thick. Measurements are made at room temperature.

Seebeck Coefficient and Electrical Resistivity System
This technique measures Seebeck coefficient (thermopower) and electrical resistivity of bulk and thin films from room temperature to 800°C. For bulk samples, typical samples are 3 mm x 3 mm x 15 mm. Thin film samples should be on insulating substrates, and the substrate needs to be at least 1 mm thick and 3 mm wide.

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TRUC

Tribology Research User Center

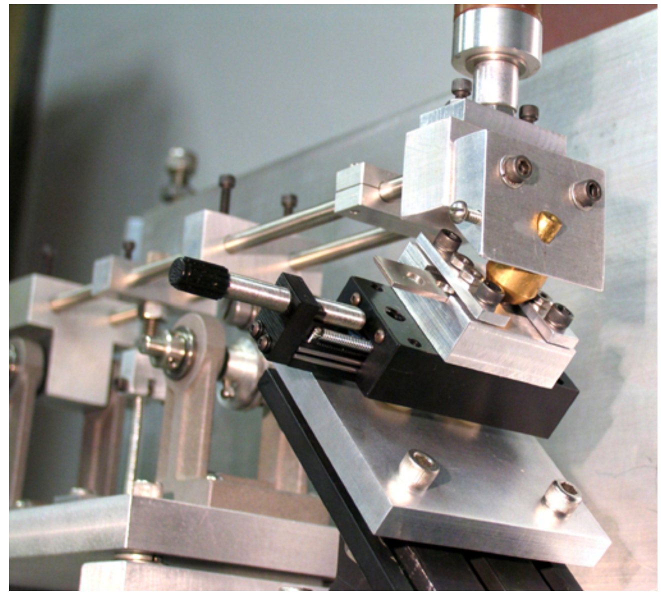
The Tribology Research User Center (TRUC) is one of six user centers in the High Temperature Materials Laboratory (HTML), a DOE User Facility dedicated to solving materials problems that limit the efficiency and reliability of systems for power generation and energy conversion, distribution, storage and use. The TRUC provides world-class facilities and a staff of technical experts for characterization of material properties and performance of load-bearing, engineering surfaces and the materials that separate them.

The mission of the TRUC is to provide a collaborative environment and a variety of tools and techniques to investigate the response of materials to friction- and wear-causing environments. Consequently, our vision is to be a national resource for providing solutions to technology-enabling barriers related to the friction, wear, and lubrication characteristics of high-performance materials. Primary customers are U.S.-based companies and universities, but the TRUC staff has worked with U.S. government agencies as well. In 2007, the new TRUC was formed to redefine the scopes of the two user centers that preceded it: the Machining, Inspection, and Tribology User Center (MITUC) and the Friction, Wear, and Machinabililty User Center (FWMUC).

The word tribology stems from the Greek 'tribos,' which means "rubbing." It describes a broad interdisciplinary field of science and technology ranging from studies of frictional interactions on the nanometer scale to massive earth movements on the megascale. The TRUC facilities support tribological research and characterization of new materials, coatings, and surface treatments for diverse applications like energy-efficient engines, brakes, farming equipment, medical implants, and manufacturing. Users vary from university faculty involved in fundamental studies of the nature of friction in thin films to applications engineers investigating wear-resistant facings for rolling mill rolls, emission-control valves in diesel engines, and down-hole oil well casings. The diversity of past users has given us a unique experience base from which to help future users solve their tribology problems effectively.

Helping Material Developers and Designers

Selecting appropriate materials, surface treatments, coatings and/or lubricants for friction- and wear-critical applications can present a formidable challenge for designers and material developers. Sometimes an existing material will perform satisfactorily, but under severe operating conditions and cutting-edge designs, new high-performance materials may be needed. The path to a solution often involves laboratory scale testing, data collection, and subsequent analysis. Experienced TRUC staff members advise users on the best approach to their specific friction or wear problem, whether it involves basic research or applies to a particular type of machinery. ORNL's experience in friction and wear research spans over a quarter century and encompasses a wide variety of materials, including ceramics, metal alloys, carbon materials, composites, solid lubricants, polymers, and intermetallic alloys. Customized test apparatus are available to produce various forms of wear such as unidirectional sliding, reciprocating sliding, scuffing, repetitive impact, belt abrasion, and slurry abrasion.

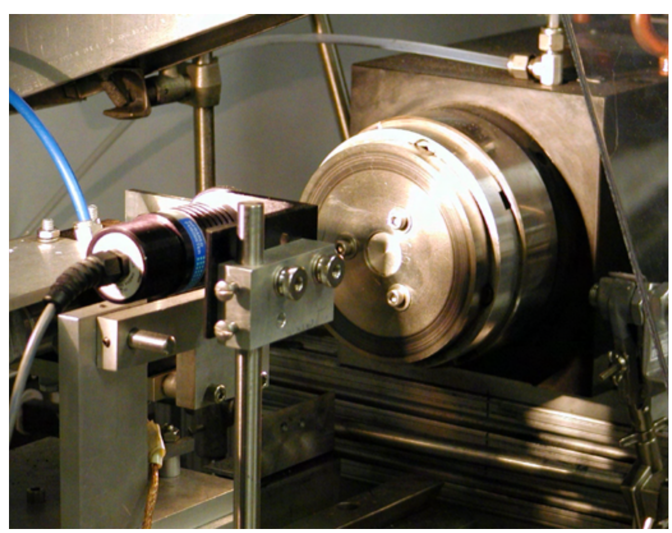


Repetitive impact test apparatus also imparts slip.

Tribotesting Capabilities

- Customized friction and wear testing apparatus for a variety of specimen types
- Lubricated and non-lubricated contact conditions
- Instrumented scratch testing for hardness and adhesion studies
- Hot hardness testing
- High-temperature friction and wear testing up to 1000°C
- Abrasive wear and grindability evaluations related to machining
- Repetitive impact testing with varying slip

More than fifteen kinds of friction and wear testing configurations are available, including a high-speed sub-scale disc brake apparatus and a high-temperature repetitive impact test system designed to study the durability of diesel engine exhaust valves at temperatures to 850°C. Supporting instruments include a mini-viscometer, microindention hardness tester, optical and measuring microscopes with image analysis, and a micro-abrasion system to measure coating thickness.



High-speed sliding test apparatus for brake materials

Web: http://html.ornl.gov

html HIGH TEMPERATURE MATERIALS LABORATORY

Setting the Standard

Over the years, ORNL has led the development of three new ASTM standards for wear and friction testing (G 133, G 171, G 181) and has participated in the development of several others (G 99, G 164, G 174). One of these standards (G 181) is a key method to measure the friction of piston rings and cylinder liners in engine-conditioned oil and is used by a major diesel engine manufacturer.

The use of standards, where applicable, helps to ensure the repeatability of our data and the ability to compare our results with past work under similar test conditions. We recognize the importance of understanding the attributes and limitations of each test method in order to more fully appreciate the implications of the data. Working with the standardization process provides a firm grounding when it comes to helping TRUC users interpret their results, and it strengthens the relationship between researchers and end-users.

Dimensional Metrology Instruments

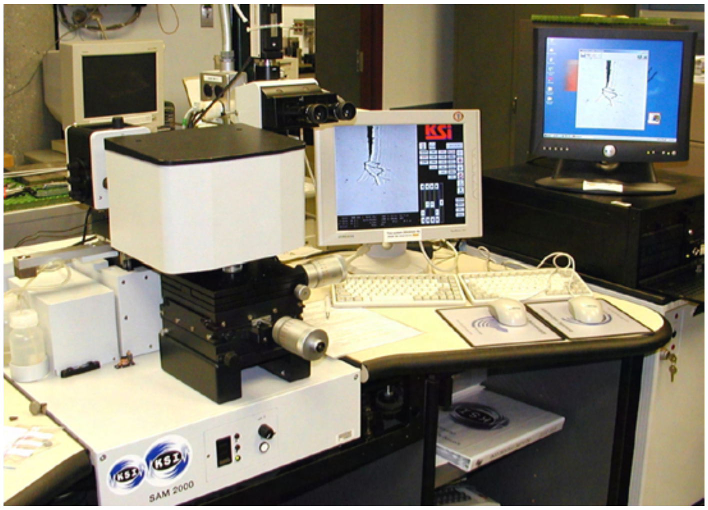
The TRUC maintains outstanding dimensional metrology and surface texture measuring equipment to assist HTML guest researchers to characterize the size, shape, and surface roughness of their samples. Our highly qualified technical staff is available to assist researchers in the operation of the more complex equipment, such as the coordinate measuring machine and atomic force microscope.

Measurement Capabilities

- Contact (stylus-based) and non-contact (laser-based) surface topography measurement instruments
- Image analyzer for high-magnification feature measurements
- 3D optical imaging apparatus for micro-topography
- Unique scanning acoustic microscope (SAcM) for imaging subsurface flaws with high frequency sound waves

Most of the metrology instruments are computer controlled, and data can easily be exported to advanced analysis software. Users are also provided with image files for use in presentations and publications.

Overall, the TRUC represents a unique national resource that supports advances in materials science and technology by providing expertise in tribology, metrology, and the micromechanical response of surfaces.



Unique Scanning Acoustic Microscope can probe beneath surfaces for the origins of wear.

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