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

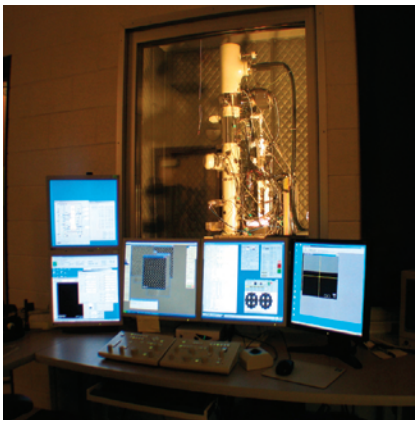
Hitachi HF-3300 TEM/STEM

- 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

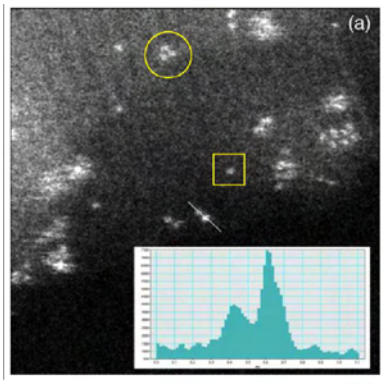
MAUC personnel have experience in characterizing the structure and microchemistry of a wide variety of catalytic materials. We have imaged structures such as five-atom osmium clusters on MgO crystals, tri-rhenium carbonyl clusters on alumina, atoms and clusters of Pt on a variety of oxide supports, and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

Web: http://html.ornl.gov

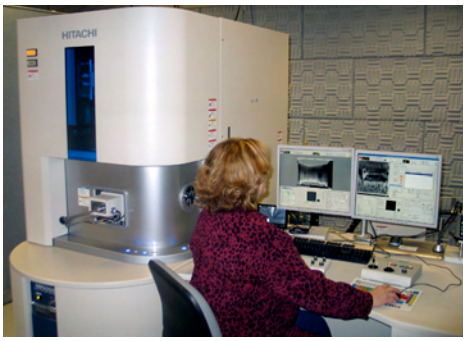
html HIGH TEMPERATURE MATERIALS LABORATORY



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.*

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