Microscopy for Nuclear Energy Materials

Studying the effects of extreme environments at the atomic level

Pacific Northwest
NATIONAL LABORATORY

The JEOL GrandARM scanning transmission electron microscope enables researchers to correlate atomic-resolution structural and element-specific compositional maps of irradiated materials.

Materials used in nuclear facilities—from power plants to fuel production—must withstand corrosion and radiation in high-temperature and pressure-extreme environments. Stress- and irradiation-assisted degradation results in changes at the atomic level, which can lead to premature replacement or even failure of equipment, components, or structures.

Microscopy enables researchers to observe and understand these changes at scales unobservable by eye. Scientists at Pacific Northwest National Laboratory (PNNL) have a long history and striking record of using state-of-the-art microscopy capabilities to study and understand how the extreme environments in nuclear facilities affect materials. Supported by leading experts in theoretical modeling and computational image analysis, microscopists can obtain and interpret unique high-quality data. Insights from these data can help inform the design of new materials for advanced reactor applications and to sustain the current nuclear power fleet.

PNNL has microscopy capabilities located in facilities capable of examining radioactive samples.

Microscopy of Radioactive Materials

The Radiological Microscopy Suite inside the Radiochemical Processing Laboratory—PNNL's Hazard Category II non-reactor nuclear research facility—enables researchers to examine and characterize radioactive materials with advanced microscopes. The suite consists of four specially designed laboratory spaces that house advanced microscopes and a sample preparation laboratory with a radiological fume hood to work with radioactive materials.

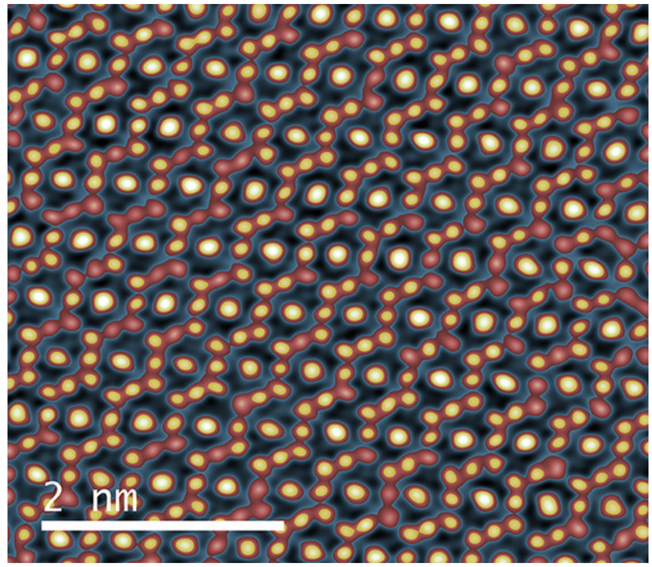
The Radiological Microscopy Suite contains a focused ion beam for precise sample preparation, an environmental scanning electron microscope allowing in situ observation of microscale processes, such as crystal growth, an atomic force microscope for surface analysis, and a JEOL GrandARM aberration-corrected scanning transmission electron microscope (STEM) with advanced analytical capabilities, such as energy dispersive x-ray spectroscopy and electron energy loss spectroscopy. The STEM and atomic force microscope are equipped for measurements at cryogenic and elevated temperatures, allowing researchers to preserve delicate structures or observe material behaviors under extreme conditions.

To facilitate extremely high-resolution measurements, the suite is "quiet" with reinforcement and isolation schemes to protect the instrument from disruptive vibrations, sounds, or electromagnetic interference. Adjacent to the quiet suite is a recently added radiation-capable, new-generation, local-electrode atom probe.

Atom Probe Tomography

PNNL currently has three atom probe instruments, the highest number at a single institution in the United States. Custom modifications of these instruments enable in situ chemical reactions in controlled gas environments at temperatures up to 500° C. Scientists can transfer environmentally sensitive and cryogenically preserved specimens directly into the atom probe for the highest resolution of 3-D chemical analysis.

Atom probe tomography is well suited to studying molecular-scale changes in the microstructure caused by degradation in real and simulated nuclear environments. It is also capable of studying hydrogen and its isotopes, which are practically impossible to observe at an atomic level through other approaches. Thus, atom probe tomography may prove to be the key to understanding the role of hydrogen embrittlement and tritium permeation in environmental degradation.



Above: Atomic-resolution STEM image of a Ni-Fe oxide spinel formed by corrosion of Ni-base alloys in nuclear environments.

Metallography Lab and Sample Preparation

For many measurements, the quality of the resulting data can be directly tied to the quality of sample preparation. PNNL has a fully staffed metallography lab with trained professionals who prepare both radioactive and non-radioactive samples for a wide range of microscopy studies. Sample preparation methods include mechanical, chemical, and electro-polishing techniques for bulk samples as well as focused ion beam and flash electropolishing for preparing microscopy samples.

Autonomous Microscopy

Researchers at PNNL are working to bring artificial intelligence and machine learning capabilities to microscopy, enabling more efficient data acquisition and analysis. AutoEM is an electron microscope platform that combines machine learning with automation to dramatically speed image acquisition and processing. AutoEM can achieve in one hour what used to take a team of humans 1,000 hours and was selected for a 2024 R&D 100 award. Beyond AutoEM, PNNL is leveraging expertise in both autonomous microscopy and

Atom probe tomography provides unique 3-D, atomic-scale elemental and isotopic maps of nuclear materials, including the permeation and trapping of hard-to-detect species such as tritium.

atom probe tomography to autonomously operate these instruments. To this purpose, scientists use AI/ML methods to both analyze large amounts of complex data during the experiment and make decisions about acquisition parameters on-the-fly.

PNNL's microscopy capabilities for nuclear materials are primarily based in the Material Science and Technology Building and the Radiochemical Processing Laboratory. Additional microscopes are located in specialized research facilities, including the Energy Sciences Center, Grid Storage Launchpad, and the Environmental Molecular Sciences Laboratory. In addition to conducting research funded by federal agencies including the Department of Energy Office of Nuclear Energy, PNNL's microscopy capabilities and expertise are available to industry and university partners.

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