EMSL

Capabilities Series

Deposition and Microfabrication

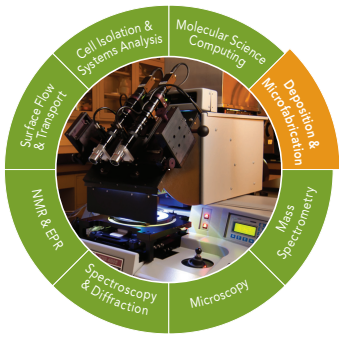
Designed to augment research important to a variety of disciplines, EMSL's Deposition and Microfabrication Capability tackles serious scientific challenges from a microscopic perspective. From deposition instruments that emphasize oxide films and interfaces to a state-of-the-art microfabrication suite, EMSL has equipment to tailor surfaces, as diverse as single-crystal thin films or nanostructures, or create the microenvironments needed for direct experimentation at micron scales.

EMSL's deposition and microfabrication instruments are part of an unparalleled collection of capabilities designed to support research that integrates experimental and computational tools. These tools enable novel advances in EMSL's science themes of Biological Interactions and Dynamics, Geochemistry/Biogeochemistry and Subsurface Science, and Science of Interfacial Phenomena. Research includes:

- **Catalysis** – developing thin-film catalysts to modify chemical reaction rates for applications including biomass, CO_2 conversion, and electrical–chemical interconversion
- **Subsurface flow and transport** – generating a better understanding and predicting behaviors of microbial communities or contaminants (metals, radionuclides, chemicals) within the subsurface
- **Analytical microdevices** – performing complex, multiple-step processes in integrated microfluidic devices that offer efficient chemical separations, minimize reagent consumption, and reduce sample loss
- **Geochemistry and biogeochemistry** – controlling environmental interfaces, such as oxide minerals, as substrates for heterogeneous surface organic, inorganic, and biochemical interactions
- **Complex oxides, surfaces, and interfacial phenomena** – preparing high-quality pure and doped oxide thin films, surfaces, and interfaces to study complex interrelationships between composition; structure; and photocatalytic, magnetic, electronic, and electrical transport properties.

CAPABILITY DETAIL

Deposition



Discovery® Deposition System
- hybrid system provides multi-target sputtering, MBE, electron beam deposition, and RHEED characterization

Pulsed Laser Deposition System
- can grow uniform, multilayer, or compositionally spread combinatorial complex-oxide thin films

Molecular Beam Epitaxy
- features a customized MBE chamber for synthesis and *in situ* characterization of novel oxide, ceramic, and mineral materials as crystalline films.

Microfabrication

Aligning Wafer Bonder
- heat, pressure, oxygen plasma, and electric field combination welds multiple wafers

Deep Reactive Ion Etching System
- bores patterns into silicon and glass substrates as small as a few tens of nanometers with a large aspect ratio

Direct-write Lithography System
- creates the photomask or can directly pattern features on varied substrates

Dual Focused Ion Beam/Scanning Electron Microscopy
- combines FIB and SEM for high-resolution microscopy, as well as fabrication and manipulation of structures at the nano scale

Photomask Aligner
- provides ultraviolet light source for photolithographic patterning and enables alignment of features for multilayer devices

Rapid Thermal Annealing Furnace
- quickly heats wafers in gases to facilitate chemical reactions and grow thin films

Thermal and UV Nanoimprint Lithography System
- uses pressure, heat, or light to transfer patterns from a master to another substrate.

WHY USE EMSL'S DEPOSITION AND MICROFABRICATION CAPABILITIES?

- In one location, EMSL's Deposition and Microfabrication capability unites a suite of instruments designed to tailor surfaces atom by atom.
- Opportunities for integration with other EMSL resources provide a unique multidisciplinary research environment. For example, the high-resolution X-ray diffraction, electron microscopy, XPS, and ion-beam analysis capabilities allow detailed characterization of specimens.
- Expert staff members and distinguished users share their extensive scientific knowledge and expertise in using EMSL's instruments and resources, such as mass spectrometry, microscopy, spectroscopy, diffraction, and the high-performance computing capability, to unite experiment and theory.

ABOUT EMSL

EMSL, a U.S. Department of Energy national scientific user facility located at Pacific Northwest National Laboratory, provides integrated experimental and computational resources for discovery and technological innovation in the environmental molecular sciences to support the needs of DOE and the nation. EMSL's distinctive focus on integrating computational and experimental capabilities, as well as collaborating among disciplines, yields a strong, synergistic scientific environment. Uniting experts and an unparalleled collection of state-of-the-art instruments under one roof, EMSL has helped thousands of researchers use a multidisciplinary, collaborative approach to solve some of the most important and complex national scientific challenges in energy and environmental sciences.

To learn more about EMSL, the science conducted at EMSL, as well as the instruments and expertise available to users, visit www.emsl.pnl.gov.

BECOME AN EMSL USER

Researchers are invited to access the world-class capabilities and collaborate with internationally recognized experts at EMSL via its peer-reviewed proposal process. To submit a proposal, follow the five steps outlined on the User Access section of the EMSL website (www.emsl.pnl.gov). Current and potential EMSL users are encouraged to respond to Calls for Proposals, which are announced each spring. However, unique research proposals that fall outside the Calls for Proposal focus may be submitted at any time.

Applicants are encouraged to submit proposals to use EMSL's capabilities with an emphasis on integrating computational and experimental tools. In general, most users whose open research proposals are accepted may use EMSL resources free of charge. Open research generally is defined as science and engineering research for which the resulting information is published and shared broadly within the scientific community.

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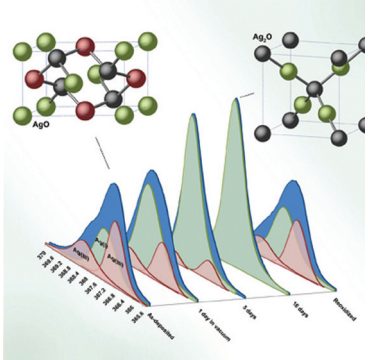
RESEARCH HIGHLIGHTS

Silver's Surprising New Signature

A routine instrument calibration involving EMSL's X-ray photoelectron spectrometer (XPS) and highly oxidized silver led to a game-changing discovery when scientists saw something entirely unexpected—the true XPS signature of rare Ag(III). Using high-resolution XPS within a vacuum deposition chamber, scientists deposited silver metal on a substrate. During deposition, they exposed the silver film to oxygen plasma, oxidizing the silver. Then, they created different films using varying levels of silver and oxygen. Because EMSL's XPS is connected *in situ* to the deposition chamber, scientists measured the position of peaks produced and determined the oxidation state without exposing silver oxide samples to air or varied temperatures that contaminated previous studies. Scientists observed a surprising XPS peak position and determined it was from Ag(III), contradicting the signature accepted since 1973 and expanding the potential for new silver data in catalysis, electrochemistry, nanomaterials, and other fields. This work was featured on the December 2010 cover of *The Journal of Physical Chemistry C*.



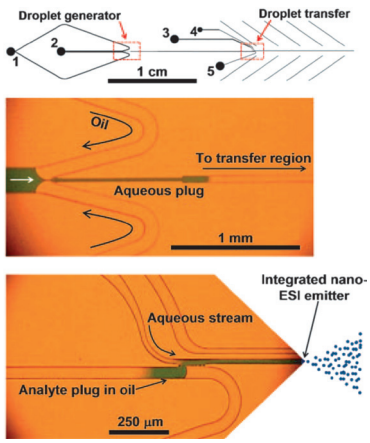
Using deposition by MBE and *in situ* XPS at EMSL, scientists discovered Ag(III)'s true XPS signature.

EMSL Users: PNNL and University of North Texas, Department of Chemistry.

Kaspar et al. 2010. *The Journal of Physical Chemistry C* 114(49):21562-21571. DOI: 10.1021/jp107914e.

A Concentrated Effort

Oil-encapsulated microdroplets serve as ideal reaction and storage vessels for ultra-small samples, such as individual cells. However, detection methods have largely been limited to optical methods that probe the contents of intact droplets. In a breakthrough solution, PNNL scientists researching properties of droplet-based microfluidics used EMSL's ultra-sensitive electrospray ionization (ESI) mass spectrometer to examine droplet contents by transferring an oil-encapsulated droplet (plug) into the instrument's continuous-flow source that terminated at an integrated nano-ESI emitter. The device's microchannel design afforded stable transfer of the droplet into an aqueous stream, assuring hydrophobic solvent did not pass the apertures and analytes were sent to the microchip. This method accelerated transfer time and allowed researchers to analyze picoliter-level samples with essentially no dilution and a gain in sensitivity of about three orders of magnitude versus earlier work. Ultimately, the process could better support proteomics and single-cell analysis, as well as promote microchip capillary electrophoresis technology. This innovative research was featured as an online news spotlight in *Analytical Chemistry* and a research highlight in *Lab on a Chip* (The Royal Society of Chemistry).



Microfluidic Device Design

EMSL Users: PNNL.

Kelly et al. 2009. *Angewandte Chemie International Edition* 48(37):6832-6835. DOI: 10.1002/anie.200902501.