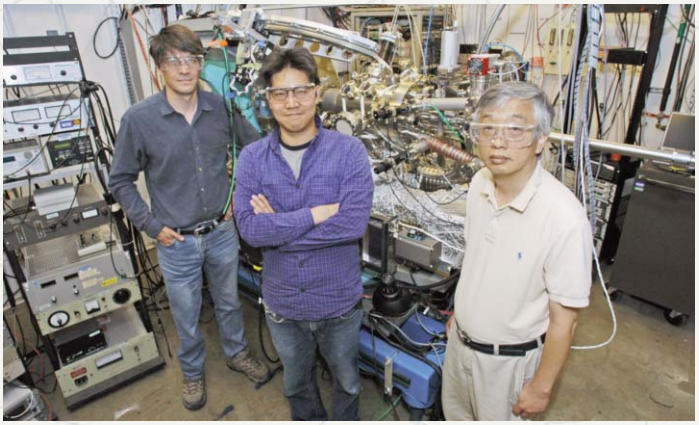
The Advanced Photon Source
The *In situ* Oxide MBE System at the APS

Under the Argonne National Laboratory's Materials for Energy Strategic initiative, the world's first *in situ* oxide molecular-beam epitaxy (MBE) system has been built at X-ray Science Division (XSD) Sector 33 of the Advanced Photon Source (APS). It will play an important role in helping move forward Argonne's development of functional layered oxides.

The layered complex oxides consisting of stacked oxide sheets offer a broad range of functionalities for meeting many outstanding energy challenges. Some have demonstrated excellent mixed ionic-electronic conducting properties, making them highly attractive candidates for intermediate-temperature solid oxide fuel cell cathodes. Others have a unique structure that makes them ideal for a wide range of catalytic reactions, e.g., photochemical water splitting. With the recent advances in *ab initio* modeling techniques and computational power, we can now design layered oxides that display novel functionalities. Researchers have been constrained in their ability to synthesize such layered materials because of limited control over composition and structure during deposition, even with the most powerful tool available for layer-by-layer oxide film growth: oxide MBE. For materials with highly tunable properties, like the complex oxides, loss of stoichiometric control is tantamount to unintentional doping, turning what was to be an excellent dielectric material into a conductor. Such synthesis issues have impeded the widespread applications of complex oxides to the many energy technologies in which they have so much potential. Scientific/technological progress in this field demands the realization of exceptional compositional and structural control during synthesis.

While researchers have some control over deposition parameters, nature controls crystal growth. We need to not only monitor growth, but also understand what atomic-scale processes take place, along with their associated energies and time scales. For example: Is the deposited structure thermodynamically stable? Does dynamic surface segregation of one of the cation species occur during deposition? Do the interfaces naturally interdiffuse at the growth temperature? Many such questions can be addressed by harnessing state-of-the-art *in situ* synchrotron x-ray techniques to provide a heretofore unknown level of insight into the synthesis of complex materials. Integrating the oxide molecular-beam epitaxy system into a high-flux, hard x-ray beamline will enable us to track layer-by-layer structure and composition in real-time.



Left to right: John Freeland (XSD-Magnetic Materials Group), June Hyuk Lee, and Hawoong Hong (both XSD-Surface Scattering & Microdiffraction Group) in the 33-ID-E enclosure with the oxide growth upgrade to the existing molecular beam epitaxy system.

In order to synthesize the complex composition profile for layered oxide materials, the MBE chamber accommodates multiple elemental sources with integrated shutters for layer-by-layer growth control. Each source also has a vacuum gate valve that enables source maintenance without affecting the base pressure of the ultra-high-vacuum chamber. Film oxidation is accomplished using a commercial ozone generator/distiller that delivers a jet of pure ozone to the growth position. This high oxidizing power of ozone enables the growth of a wide class of oxide materials in ultra-high-vacuum environment. Additional features include a new ozone-compatible sample holder design, a vacuum load-lock system for quick interchange of samples, and a reflection high-energy electron diffraction system for offline growth. This allows detailed growth studies before the x-ray experiments to optimize growth conditions. Most importantly, all this functionality can be controlled remotely, which is necessary for *in situ* studies where the conditions can be actively manipulated during the x-ray experiment. While this instrument is currently under development, it is expected to be available to the general-user community beginning in 2013.

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Call for APS General-User Proposals

The Advanced Photon Source is open to experimenters who can benefit from the facility's high-brightness hard x-ray beams.
General-user proposals for beam time during Run 2013-1 are due by Friday, October 26, 2012.
Information on access to beam time at the APS is at http://www.aps.anl.gov/Users/apply_for_beamtime.html or contact Dr. Dennis Mills, DMM@aps.anl.gov, 630/252-5680.

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