X-Ray Interface Science Workshop: Materials Synthesis Report

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Scientific Requirements

In situ, x-ray synchrotron measurements provide a unique window into the dynamic processes occurring during materials synthesis, processing and use. Design and synthesis of new materials requires answering questions of what materials process occur, where they occur, when they occur, and why; and using that information to develop predictive models of both material properties and pathways through metastability to the desired new materials. The capabilities proposed for the new XIS sector will enable the efficient and effective use of x-ray techniques to understand multi-scale mechanisms that are critical to the synthesis of new materials and to their use in realistic, active environments. The participants in this workshop agreed that an acceptable balance between the ability to use state-of-the-art x-ray techniques and extended access to valuable APS x-ray capabilities has been achieved with the proposed new XIS facility.

Facility Requirements

Modern growth techniques are sophisticated and require significant laboratory space to be productive. For the XIS facility, the required space will include both space in the x-ray hutch and adjacent (or at least nearby) laboratory space.

Hutch Requirements

The consensus of the group was that a minimum hutch size was roughly 4×6 meters and that an asymmetric beam location would improve hutch utility. This space is necessary for growth equipment, for x-ray optics and detectors, and for supplemental probes such as optical probes. The hutches will need to be equipped to handle reactive and toxic chemicals, and this necessitates significant exhaust capacity. Since growth systems often require significant amounts of liquid nitrogen and boil-off nitrogen, integrating these requirements into the hutch design would be good. In addition, it is desired to maintain a relatively clean environment with the ability to provide local regions of at least Class 1000 cleanroom conditions. These requirements will require infrastructure and practices that are a significant departure from those currently used at synchrotron facilities including the APS.

In addition to x-ray characterization, we anticipate that many growth systems will include other in situ analysis tools. These additional tools will be used both independently and in conjunction with x-ray tools. We anticipate that the tools will

include optical spectroscopies, light scattering, optical microscopies, electron spectroscopies (e.g. XPS) and electron diffraction (e.g. RHEED).

Laboratory Requirements

The growth experiments will need access to a variety of sophisticated laboratories for sample preparation, ex situ analysis and growth system maintenance. For sample preparation and growth system maintenance there will be a significant need for a well-designed chemistry laboratory with facilities for HF, concentrated acids, and solvents. A scanning electron microscope, an atomic force microscope and a high end light microscope will be necessary. In addition, the availability of UHV surface analysis capabilities such as Auger and x-ray photoemission spectroscopy would be very useful.

It will be necessary to have storage for a significant supply of compressed gas cylinders of reactive gasses, bubblers for growth precursors and a variety of less standard chemicals. These supplies often have significant lead-time for purchase and delivery, and must be stored between experiments.

X-Ray Requirements

It is anticipated that most experimental measurements in materials synthesis will be performed between roughly 4.5 keV and 30 keV. Below 4.5 keV the experiments become difficult because of the resultant short x-ray penetration distance. Above 30 keV detectors and optics become much more challenging. However, there are some materials synthesis measurements (e.g. bulk crystal growth) that might benefit greatly from even higher x-ray energies. Coherent x-ray scattering measurements are emerging as a useful tool to explore materials synthesis. Coherent techniques on a storage ring are most useful at lower photon energies (perhaps below 9 keV) because of the strong dependence of coherent fraction on photon wavelength. However, for the fixed energy beamlines, the participants had a weak preference for the 19.9/19.9/33.2 KeV option.

A strong need for small x-ray focused beams is not anticipated. The participants generally thought that a 50μ×50μ primary focal spot was a good goal. Space for in-hutch focusing should be reserved for experiments that require smaller focal spot sizes.

An x-ray energy resolution of 10^{-4} ($\Delta E/E$) is generally suitable for most x-ray measurements including resonant studies near absorption edges. The broad bandpass of multilayer monochromators (e.g. 10^{-2}) can be very useful time-resolved measurements. Multilayer support would be useful on the fixed energy beamlines if the technical issues can be solved.

Higher performance x-ray detectors are crucial for the real-time measurements planned for this facility. In particular, fast readout is necessary to capture transients in thin film and surface structure during the initial steps of processing. Since many of the experiments will operate at high photon energies, the development of high quantum efficiency (i.e. stronger x-ray absorption) sensors at 30 keV could yield up to an order of magnitude of performance. This performance increase is particularly crucial since it comes with no increase in the radiation dose and associated damage or process modification.

Technical Support

Growth systems are typically complex with intrinsic hazards (e.g. high-power lasers), generally use a variety of hazardous chemicals (e.g. pyrophoric), and require great attention to detail in order to generate the reproducible measurements crucial to advancing synthesis science. To operate a user facility safely and efficiently will require a number of dedicated beamline scientists supported by technical experts in areas such as chemical safety, vacuum systems, and ex-situ characterization. We estimate that two staff members will be necessary for each station although some specialized skills may be shared within the facility (e.g. chemical safety).

The goal of this facility is to greatly improve synthesis science. This requires real-time x-ray measurements and real-time data visualization. These real-time x-ray measurements will rely on higher performance x-ray detectors that will often be operated in an on-the-fly data acquisition mode. Such operation will generate rapidly acquire large amounts of data. Both the instrument control and data visualization software tools to accomplish these goals do not currently exist and the APS will need to take a leading role in developing these capabilities. We anticipate that this development will be continuous. That is, as progress is made on synthesis science, the types of measurements (e.g. coherent x-ray imaging) required to make progress will evolve and the hardware and software requirements will evolve as well. Thus, the facility will need on-going development of software tools.

Partnerships

Strong partnerships with science programs within Argonne and with nearby universities will greatly strengthen the scientific program for the XIS facility. In particular, the panel believed that mechanisms to access the extended capabilities of Argonne including, but not limited to, the Center for Nanoscale Materials and the Electron Microscopy Center, as part of the beamtime proposal would be advantageous. For example, in synthesis studies it is often necessary to anneal a sample at high temperature in a controlled environment. Such facilities for a wide variety of materials characterization and ex situ processing exist at Argonne but are currently difficult for users to access.