UHV Surface/Interface Science: Exotic Phenomena of Low-Dimensional and Nanoscale Systems

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Scientific Requirements

In the coming decade, new nanoscale materials will be the main focus of science and technology. Because of their small dimensions, nano-scale materials exhibit intriguing new physical behavior that challenges our conventional understanding of solid state materials. They also present significant new challenges for materials synthesis. These materials are extraordinarily sensitive, because of their large surface to volume ratio, to their surrounding environment, including at the interface with their supporting surface. For this reason, it is essential to both fabricate and study nanoscale materials *in situ* in ultra-high-vacuum and to have the tools to investigate their surfaces and buried interfaces which so strongly influence their physical behavior.

Bright undulator beams from the Advanced Photon Source can play a pivotal role in revealing the fundamental physical behavior of nano-materials and their fabrication. In particular, x-ray scattering from surfaces and interfaces is the central tool needed for solving problems that *cannot* be addressed by other conventional surface science techniques. Traditionally, real-space scanning probe methods have been used for *in situ* studies of nano-materials; however, these methods have critical short-comings: they do not have the necessary atomic-scale precision, chemical specificity, or the ability to investigate buried interfaces. These are, however, precisely the strengths of x-ray scattering. Moreover, x-ray scattering methods provide outstanding and superior statistical accuracy, which is essential for understanding kinetic behavior. Time-resolved studies (such as during pulsed deposition) are basically impossible with STM. Another advantage of x-ray techniques is that in situ growth under aggressive environments can be easily studied. Numerous examples of how UHV *in situ* x-ray scattering methods *uniquely* solved important problems in nano-scale materials were presented during the X-ray Interface Science Workshop held at the Advanced Photon Source on January 10, 2012.

In looking towards the future and planning the new XIS sector, it is crucial to recognize that we cannot and should not design around a narrowly predicted future. Science and nature have a long history of surprising us with the unexpected. Therefore, it is important to develop a facility that is flexible and that can accommodate new materials and new ideas as well as be in a position to investigate unforeseen physical phenomena.

There are essentially two components to the overall design philosophy of the proposed UHV *in situ* facility. The first is to be able to utilize all of the surface/interface scattering techniques, including reflectivity, crystal truncation rods and diffuse scattering, while nano-materials are grown and perturbed *in situ*. The second feature centers on maximum flexibility: the growth

and analysis tools will be configurable and reconfigurable in order to meet the presently known needs of users as well as to be able to adapt to future scientific needs.

Extensive technical support for this facility is absolutely essential because of its technical complexity. At a minimum, all of the UHV tools require maintenance and configuration changes prior to x-ray scattering experiments. However, most *in situ* x-ray scattering studies will also require considerable preliminary measurements and calibrations prior to the assigned beam time. Importantly, if the APS wishes to welcome *non-Argonne users* of the facility, having adequate support is critical. Without it, external users will not come. On the other side of this argument, if the APS does a good job with its technical support it can be a model facility for the user community. If additional motivation for this support is required, one only has to look to other user facilities as an example. Neutron scattering facilities such as NIST, which typically do not have in situ tools as complex as a UHV system, have substantial user support; for synchrotron radiation, the ESRF is an excellent example where users are well supported and where the user community is involved with complex experiments.

Description of the Facilities

In order to achieve maximum flexibility without compromising functionality, the surface chamber will contain multiple stations, as shown in Fig. 1. There will be a main in situ chamber where the x-ray scattering takes place and a connected auxiliary chamber that will permit more complex functions.

The main in situ chamber will have a large Be window that allows access to a wide range of angles for the incident and outgoing x-ray beam. Large angular ranges are necessary for extended-range reflectivity and truncation rods as well as for high-Q measurements of strain effects at interfaces.

This main in situ chamber will contain the tools that are necessary for surface preparation and analysis during an in situ x-ray scattering experiment, such as several evaporators, ion sputtering, Auger/LEED, gas handling, etc. It is important to have a broad range of temperatures, typically 20 K at the low end to 1500 C at the high temperature end. The low temperature is required for interesting quantum phenomena of nano-islands, magnetic systems, as well as for freezing out surface diffusion. The high-end temperature is required for many surface preparation processes. For example, Si requires a flash anneal to 1200C; refractory materials such as SiC, and Ni require at least 1400 C anneals. Two in situ sample motions are also required. A sample azimuth motion is necessary for diffraction and an arc motion is required for orienting the sample with respect to various ancillary tools in the chamber (Auger, evaporators, etc); it can also be used for the diffraction measurements. We have proven experience in fabricating these high precision motions for a UHV environment.

A key design feature is to have a “cluster” chamber arrangement, shown in Fig. 1, which permits extensive flexibility in the types of experiments that can be configured. An auxiliary chamber can be connected, via a central load lock and two manipulator arms for sample transfer, to the main chamber. This auxiliary chamber can be easily attached/detached (without breaking

vacuum in any of the chambers) from the system or it can be interchanged with another auxiliary chamber that possesses other functions. Thus, multiple functions can be accommodated, depending on the type of processing or analysis that is required for the experiment. Importantly, such an arrangement also permits the facility to evolve with *future scientific needs*.

Note that it is not difficult to have the auxiliary chamber move with the main chamber during an x-ray scattering experiment, if that function is required (it can be chosen by the user). Considerable experience with this type of large moving diffraction instrumentation exists on variable incident-energy triple-axis spectrometers at reactor neutron sources.

For the sake of specificity, we give a few examples of how auxiliary chambers would be utilized. An important case is where it is necessary to fabricate a complex sample off-line without x-rays. Those who have done MBE growth are well aware that the fabrication details are sensitive to the instrument-specific characteristics of the UHV tools. Thus, one can establish the growth behavior and sample characteristics off-line, before the x-ray experiment. Samples can be characterized by RHEED and XPS/UPS, for example. Once the fabrication steps are well-understood, the sample can be efficiently and correctly fabricated when x-ray beam time is available and then transferred into the main in situ x-ray chamber. Another use for an auxiliary chamber function involves real-space scanning-probe tools. Often there is a disconnect between the wealth of data coming from STM, which probes only the top surface, and what is measured with x-rays. Having a low-temperature STM in an auxiliary chamber would allow the comparison of x-ray and STM measurements on the *same sample*. Finally, complex and potentially UHV-dirty processing could be performed in a special auxiliary chamber (MOCVD for example). Additional auxiliary chambers can be constructed as new research needs develop.

Because of the sample transfer arms and multiple chambers, a 6m x 7m hutch is required. The hutch must also be located on the end station, which will enable the significant and frequent off-line activity without x-rays. It will also need to be located some distance away from the storage ring in order to accommodate the large dimensions of the hutch. Figure 2 shows the hutch located across the walkway aisle, which is a position that had been previously proposed for this sector. This position would also be conveniently located to other useful facilities in the building, such as a wet laboratory, a mechanical/electronics laboratory, and access to a clean room. The hutch should be designed to handle toxic gasses typically used in materials growth processes.

X-ray Requirements

As a fixed-energy station with the options offered at the time of the Jan. 10, 2012 meeting, the optimal photon energy is 19.9keV. The proposed 10keV energy is too low as it would severely limit the amount of reciprocal space that could be covered. The proposed 30keV energy is too high as the experiments would suffer from low signal/background. If designs evolve, we could work with an energy range down to 16 keV. However, the energy must stay below the Mo K-edge (20keV) because UHV sample holders are made from Mo.

Although it is a fixed-energy line, it would be useful to have the ability to change energy by switching monochromaters. There are no particular polarization requirements for the beam. Large area detectors with fast read-out times are required for efficient data collection.

Because of the long distance from the ring to the experimental station, a pair of mirrors working in conjunction with a secondary source point is necessary for stabilizing the beam and for focusing. It is also desired to have the capability for a 10 μm beam size on the sample. This will enable one to probe inhomogeous nanoscale samples. It will also allow one to rapidly investigate thickness-dependent effects on wedge-shaped films that have varying thickness across the sample surface.

Technical Support

Given the complexity of UHV sample fabrication, it is essential to have adequate support, as discussed above. Further, in order to have *non-Argonne users* of the instrumentation, staff must be available to configure the instrumentation for experiments. There will also be a considerable amount of testing and preliminary experimentation required. Staff should also work with users during the beam time to ensure that the experiments run time-efficient and that problems with complex instrumentation are avoided. The *minimum* required staffing is envisioned to be *one scientific staff member, one technician and one postdoctoral fellow*.

Partnerships

With the appropriate technical support, it is believed that a significant user-base can be developed. Historically, the lack of adequate technical support has made it difficult for non-Argonne users to run productively on a UHV surface chamber beam line. However, with a good technical and scientific staff, a user base can be *cultivated* – and this should be in their job description. There is clear precedent for this at national neutron sources (non-UHV of course) and at the ESRF.

Financial partnerships are logical to pursue with DOE and NSF as well as with University institutions who participate in grants. A partnership with Argonne could come in the form of commitments to beam time, perhaps as PUPs, in return for participation in the development of new facilities. Also, part-time (or summer) appointments of University faculty should be considered in exchange for work at the APS. Other mechanisms could include student/postdoc support that would encourage a strong user base across the scientific community.

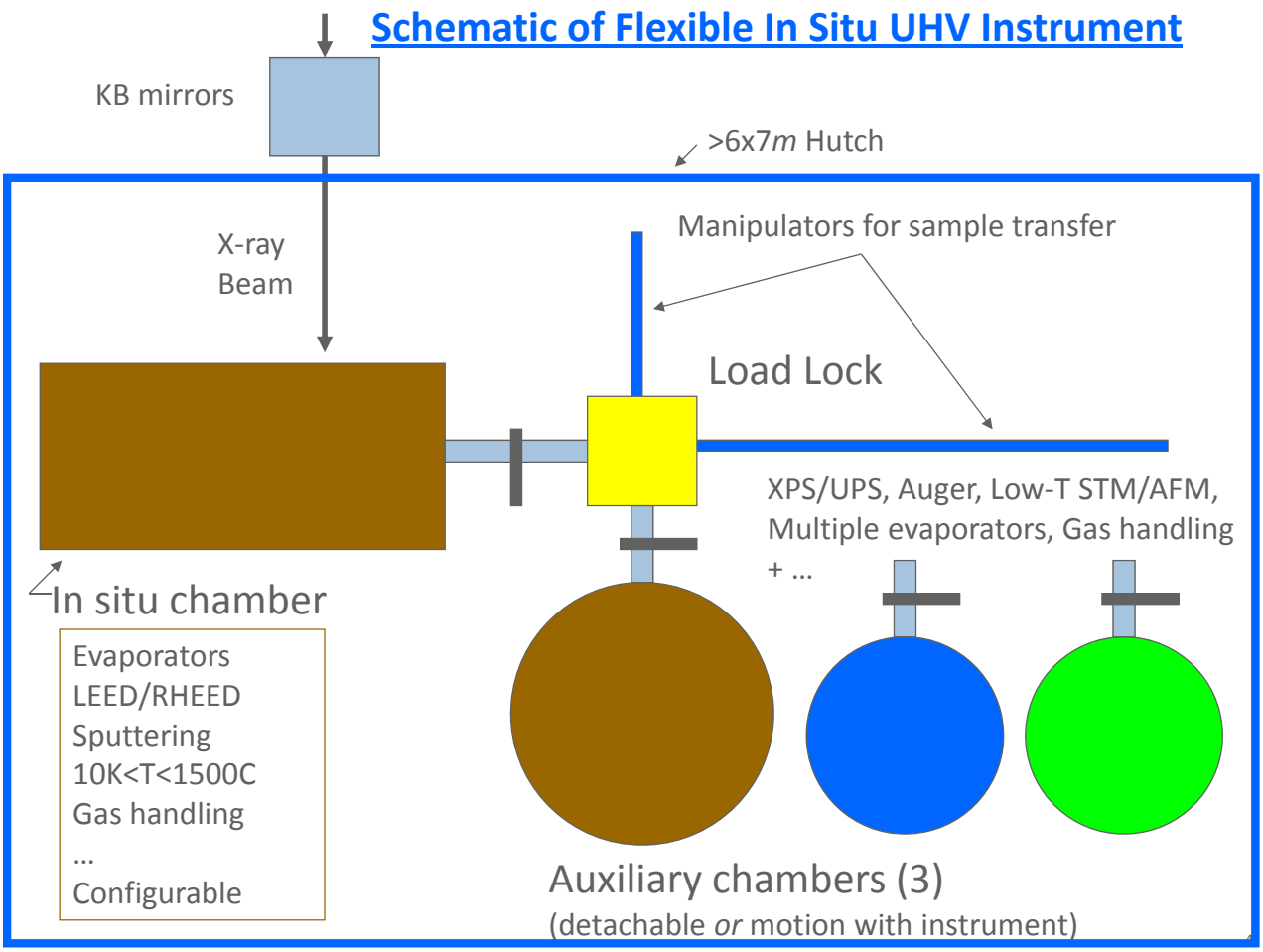
Schematic of Flexible In Situ UHV Instrument
KB mirrors
>6x7m Hutch
X-ray
Beam
Manipulators for sample transfer
Load Lock
XPS/UPS, Auger, Low-T STM/AFM,
Multiple evaporators, Gas handling
+ ...
In situ chamber
Evaporators
LEED/RHEED
Sputtering
10K<T<1500C
Gas handling
...
Configurable
Auxiliary chambers (3)
(detachable or motion with instrument)

Figure 1.

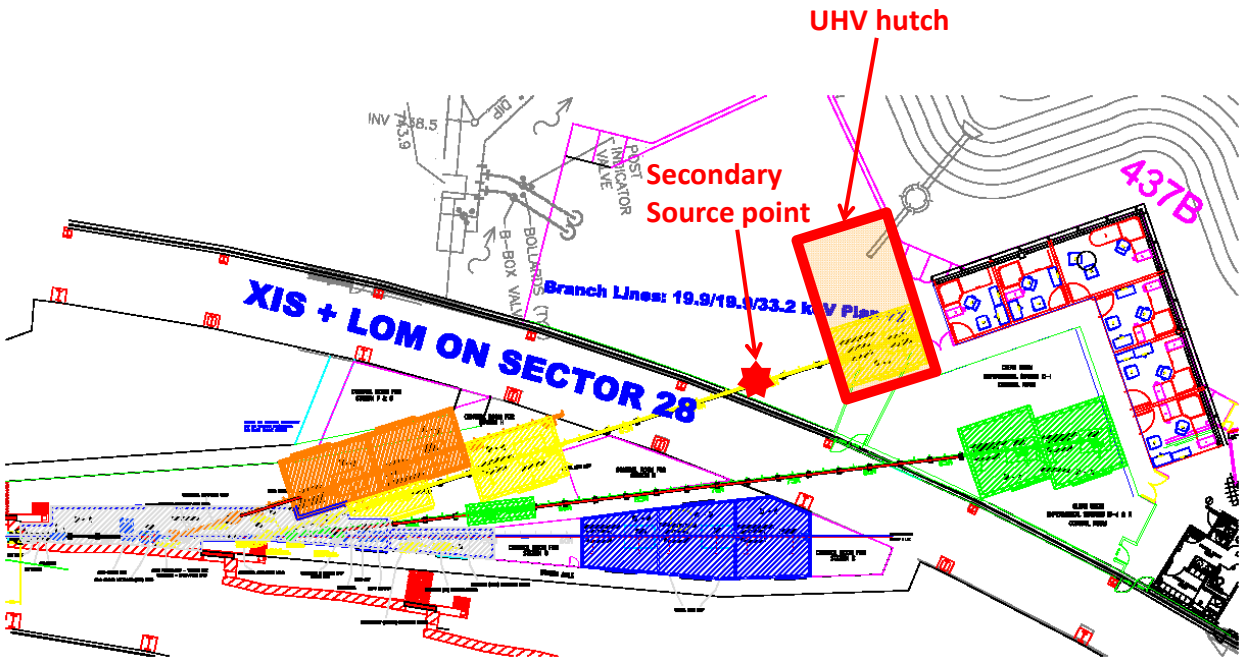

Figure 2.