UHV Surface-Analysis Endstation with X-ray Scattering and Spectroscopic Capabilities

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Abstract: The design of a versatile ultrahigh vacuum (UHV) endstation for use at the Advanced Photon Source is described. The capabilities of the endstation include X-ray scattering and X-ray spectroscopic techniques for the investigation of surfaces, interfaces, and thin films. The UHV analytical chamber also includes facilities for surface preparation, thin film growth, and standard (non-X-ray) surface analyses. The endstation, which is inspired by previous successful implementations for surface scattering, incorporates several novel design features to facilitate the use of both scattering and spectroscopic techniques, and also allows the examination of small samples. Its capabilities include X-ray reflectivity and crystal truncation rod studies, grazing-incidence X-ray diffraction, X-ray standing waves, surface extended X-ray absorption fine structure, X-ray holography, and X-ray photoelectron spectroscopy.

INTRODUCTION

A component of the research mission of the DND-CAT at the Advanced Photon Source (APS) will focus on surfaces, interfaces, and thin films. This research thrust will exploit extremely bright undulator radiation, utilizing dedicated experimental enclosures (5-ID-C). This paper will describe endstation instrumentation capable of X-ray scattering and spectroscopic investigations of these systems in ultrahigh vacuum (UHV). The endstation will reside at the undulator, but will be transportable to a bending magnet beamline enclosure (5-BM-D). The purpose of the endstation instrumentation is to provide the full complement of X-ray analytical techniques useful for investigating surfaces, interfaces, and thin films. Additionally, the full suite of conventional (non-X-ray) techniques for surface preparation and analysis was desired. These requirements present a design challenge, since the conventional implementations of many of the desired X-ray techniques nearly preclude the use of any other technique. For example, UHV X-ray scattering requires that the incoming and outgoing rays will enter and exit through a Be window, so these windows generally subtend a large fraction of the solid angle visible to the surface. However, spectroscopic techniques also require access to a large solid angle. This paper will outline aspects of the design that overcome these conflicts, and will describe other novel features of the instrument.

DESIGN REQUIREMENTS

Desired capabilities of the instrument include X-ray scattering studies [including X-ray reflectivity, grazing-incidence X-ray diffraction (GIXD) studies of crystal truncation rods (CTR) and 2D crystallography], spectroscopies [including surface extended X-ray absorption fine structure (SEXAFS) and X-ray photoelectron spectroscopy (XPS)], and hybrid techniques [X-ray standing waves (XSW) and X-ray holography]. These techniques require a wide range of sample orientations with respect to the X-ray beam, as well as access to several detectors. For example, in-plane surface diffraction is typically carried out with the sample normal oriented horizontally, reflectivity is typically studied with the sample normal in the vertical plane, and XSW may require that the sample normal take on an arbitrary orientation. It is worth noting that some of these constraints are imposed in practice by the characteristics of radiation from a bending magnet; generally, there is a large horizontal angular divergence, leading to the above choices of orientation for surface diffraction and reflectivity. Although this instrument will
be primarily deployed on an undulator beamline, it is prudent to honor the conventional constraints so that the endstation can be used expeditiously on a bending magnet beamline.

Desired conventional surface analytical techniques and preparation facilities include: low energy electron diffraction (LEED), Auger electron spectroscopy (AES), temperature programmed desorption (TPD), ion sputtering, molecular beam epitaxy (MBE), and sample heating and cooling. Excellent pumping is required, as the expected base pressure will be \(5 \times 10^{-11}\) torr. Moreover, at a third generation source where beamtime is limited, a sample transfer system is imperative.

The overall system, then, requires a precision x-ray diffractometer that can be used with the sample oriented in any direction, a scattered x-ray detector with analyzing crystal, a fast energy-dispersive x-ray detector for fluorescent x-rays, and a fast electron energy detector. Also useful would be an x-ray detector with a large angular acceptance for XSW, such as an in situ PIN diode. Moreover, the extremely narrow (sub-arc-second) rocking curve widths encountered in XSW place extreme demands on the sample stability.

**SPECIFIC DESIGN FEATURES**

**A. Diffractometer Geometry**

Perhaps the most fundamental choice to make in designing the endstation is that of diffractometer geometry. Over the years, several approaches have been implemented for UHV diffractometers. (See Refs. [1,2].) Earlier schemes featured small- to moderate-sized UHV chambers mounted directly on a standard four-circle [3,4] or z-axis [5,6] diffractometer. More recent implementations have favored using feedthroughs and/or bellows to couple some of the precision motions into a stationary vacuum envelope [7-9]. Since it is not practical to carry a large, multiple-use endstation completely on a precision diffractometer, only the latter will now be considered.

A remaining fundamental choice is how many degrees of freedom (DOF) will be carried on the sample, and how many will be carried on the detector. Although it is possible in principle to specify the momentum transfer with a total of 3 DOF, a total of 4 DOF are almost universally employed at the minimum; this over determination then allows the experimenter to choose, for example, the angle between the sample surface and the incoming x-ray (\(\alpha\)), or outgoing x-ray (\(\beta\)) [10]. The standard four-circle geometry features 3 DOF on the sample and one on the detector. The z-axis geometry [5] places 2 DOF on the sample, and 2 independent DOF on the detector. (Note, however, that in the z-axis geometry the two detector goniometers are mounted on, and rotate with, one of the sample rotations.)

Later, more complex implementations contain more than 4 DOF. For example, a rotation of a standard four-circle about a vertical axis has been used to create a five-circle (which has 4 DOF on the sample, and 1 independent DOF on the detector) [11,12]. Finally, the full combination of the motions allowed by the four-circle and z-axis geometries has also been implemented, resulting in a six-circle with 4 DOF on the sample, and 2 independent DOF on the detector [13-15].

A refinement of the basic z-axis layout has been proposed [16] and independently constructed [17] that completely mechanically decouples the DOF of the sample and detector. This diffractometer, having 2 DOF on the sample and 2 fully independent DOF on the detector, is termed the "S2D2 geometry." This arrangement is simple and has many practical advantages. It is particularly well suited to the requirements of this endstation, as discussed below, and is presently under construction [18].

The S2D2 geometry is depicted in Fig. 1. (The angular nomenclature differs from Ref. [17], and was chosen to most closely integrate the nomenclature used for all the principal diffractometer geometries.) The sample is rotated about a horizontal axis \(\omega\), which in turn rotates about a vertical axis \(\mu\). (The UHV chamber also rotates with \(\mu\).) The detector is rotated about a horizontal axis \(\delta\), which in turn rotates about a vertical axis \(\gamma\). The other angles depicted, \(\phi\) and \(\chi\), are not precision motions used for scanning the diffractometer, and will be explained later. For surface diffraction, \(\chi\) will be set to zero, placing the surface normal in the horizontal plane.

![Fig. 1 Schematic of the S2D2 geometry (a) with all diffractometer angles at zero and (b) with all angles offset to a small positive value.](image-url)
With this arrangement, only one precision DOF needs to be coupled into the UHV chamber, greatly simplifying the vacuum interface. In the present design, as in many others, the principal axial rotation is coupled by a differentially pumped rotary seal [19]. Unlike geometries that encompass the standard four-circle arrangement [7,8,13,14], however, it is possible to mount the rotary seal so that no torque is placed on the expensive bellows attached to the rotary seal.

As pointed out in Ref. [17], the S2D2 geometry allows the greatest possible reciprocal-space access for a given size of Be window. This is important in the present application, because the instrumentation necessary for spectroscopy and surface science limit the size of allowable Be windows. Also, in the S2D2 setup, the sample normal and the UHV chamber rotate as a unit during x-ray diffraction scans. This feature facilitates the use of techniques that combine scattering and spectroscopy (e.g., x-ray holography). Finally, the sample is not rotated about an external \( \chi \) arc, and so the sample may be physically far from the \( \omega \)-circle without limiting reciprocal-space access. As discussed in the next section, this ability to incorporate a lengthy manipulator arm to hold the sample is advantageous for the design.

To provide positioning of the diffractometer with respect to the x-ray beam, the entire assembly will reside on a heavy-duty, motorized translation table [20]. The table will allow precise translations both vertically and transverse to the incident beam direction. The diffractometer will have a mass of 1700 kg, and the table is capable of supporting over 3000 kg.

**B. UHV Chamber Layout**

There are two critical issues to consider for the UHV chamber layout. The first consideration is how the sample will be moved from a position where the Be windows are accessible (the "x-ray spot") to a position where the conventional surface analyses are accessible (the "surface analysis spot"). If the sample is moved with respect to the diffractometer, the sample alignment will be lost. Although providing an internal kinematic mounting for the sample can help to recover the orientation quickly [7], a better approach is to hold the sample fixed with respect to the diffractometer and to translate the chamber instead [12].

The next consideration is whether to locate the surface analytical instruments closer to the goniometer ("inboard"), or farther away ("outboard"), than the x-ray spot. In any design that uses an external \( \chi \) arc, the distance from the x-ray spot to the goniometer should be minimized to maximize the range of \( \chi \) available. Almost universally, then, the x-ray spot is placed inboard, and the surface science equipment is located farther out, away from the goniometer. To access these, either the entire chamber or the individual instruments are translated inward so that the sample is at the focal point of the analytical apparatus. This arrangement is inappropriate for this case, however, because the spectroscopic detectors require unrestricted access to the sample, and will occupy the space opposite the sample support arm. Thus, in the chosen layout, the large fluorescence detector and hemispherical electron energy analyzer are located at the most-outboard location, the Be windows defining the x-ray spot are adjacent, and the surface analytical instrumentation is in the most-inboard position.

Most surface diffraction chambers utilize a semi-cylindrical strip of Be brazed or welded into a section of the chamber wall [7]. This gives the incoming and outgoing x-rays access to 180° angular range in the traditional scattering plane, as well as a much smaller range (\( \approx 30° \) typical) in the out-of-plane direction. As appealing as that layout is, it could not be incorporated for several reasons. Firstly, the hemispherical electron detector is heavy and needs to be close to the sample; a traditional Be strip window would not allow the port for that detector to be located close enough to the sample position. Secondly, the replacement of a large section of the stainless steel chamber wall with thin Be would substantially weaken the chamber; it was feared that the torque that the detector could (and inevitably would) apply to the chamber would likely rupture the Be window or open a leak at the weld. Finally, the desired inclusion of a full array of analytical and preparation instruments inboard of the x-ray spot forced the sample to reside far from the diffractometer; it is highly desirable to minimize this distance for better sample stability, and this goal was achieved by “nesting” the surface analytical instruments with several discrete Be windows.

The resulting chamber layout is visible in Fig 2. There are three Be windows [21] mounted on ConFlat®-type flanges. The horizontal entrance window, visible in the center of Fig. 2(a), is mounted on a 222 mm \( \times \) 76 mm rectangular flange; with inner dimensions of 193 mm \( \times \) 41 mm, this window will accept x-rays at incidence angles of \( \mu < 46° \). The principal exit window is formed by a large (203 mm diameter) Be window mounted on a 254 mm OD round flange, which is prominent in Fig. 2(b). The port is offset so that the straight-through beam would exit near the periphery of the window.
This arrangement allows x-rays to exit at up to 45° in the vertical plane (δ), and up to 35° out-of-plane (γ - μ). Lastly, there is a secondary, rectangular exit window, visible near the top of Fig. 2(a), which spans angles of 90° < δ < 143° for out-of-plane angles of 8° to 20°. The S2D2 geometry allows these two compact exit windows, coupled with a wide entrance window, to provide access to a sufficient amount of reciprocal space to conduct surface diffraction studies.

Other notable features of the UHV chamber include two ports for laser-alignment of the sample; these ports are located 180° apart from one another, with a shared focus at the x-ray spot. Another port, aimed at the focus of the hemispherical analyzer, will contain an electron gun for exciting the sample for AES analysis. For MBE growth, up to three effusion cells or e-beam evaporators can be placed in ports with water-cooled jackets and shutters, all mounted on a demountable 203 mm flange. Lastly, spare ports that can contain deposition sources are focused on the x-ray spot to allow studies of real time, in situ growth.

C. Sample Manipulator

A key feature of the endstation design is the introduction of a sample manipulator, having several DOF, interposed between the sample and the α-circle. The manipulator encompasses an X-Y-Z stage [22] and two angular DOF [23], corresponding to φ and χ in a conventional four-circle diffractometer. These angular motions have precision of ≈0.01”, and thus do not represent precision motions that can be used to scan a sample during diffraction measurements (although, as noted by Robinson [1], the requirements on the precision of χ are comparatively modest). In any event, the angular DOF provided by this setup provide two very important functions. Firstly, the χ rotation allows the sample to be mounted with its surface normal oriented horizontally (for surface diffraction), vertically (for reflectivity), and anywhere in between (for XSW and magic-angle SEXAFS). Secondly, the combination of (even relatively imprecise) φ and χ rotations overcomes a shortcoming in the S2D2 and z-axis geometries: if the sample normal is not perfectly aligned with the α axis, the incident angle α cannot be held at a fixed value during a diffraction scan [17]. In essence, one can use the internal φ and χ rotations in the same way as a "goniometer head" is used on a conventional, non-vacuum diffractometer, namely, to align the plane of interest with a rotational DOF. Moreover, aside from diffraction considerations, these rotations allow the sample to be oriented to face the conventional surface analytical instruments, and to facilitate sample transfer.

The analogy to the goniometer head extends to the X-Y-Z stages of the manipulator as well. As on a conventional diffractometer, translations of the goniometer head allow an arbitrary point on the sample to be placed at the center of rotation of the diffractometer. Such an ability is vital for conducting diffraction studies of small samples with a focused beam. X-ray-based analyses of surfaces have, up until now, been almost exclusively conducted on large, homogeneous samples, and it mattered little which point lay at the center of rotation. With the advent of third generation sources, however, the exciting possibility of employing microbeams for surface and interface work can be realized. There has been a rising interest in microdiffraction; to the authors' knowledge, this instrument will be the first surface diffractometer equipped with the sample translations necessary for microdiffraction.

The sample manipulator also incorporates facilities to heat the sample (by irradiation) to 1300 K, and to cool to 125 K. Cooling is accomplished by flexible Cu braids attached to an in vacuo LN2 reservoir. Moreover, the sample sits on a transferable platen, allowing for quick exchanges of samples. This is vital in a new facility where beamtime will be limited.

There are practical difficulties in constructing a sample manipulator with all these features. The foremost concern has been with stability and rigidity of the sample support, and many steps were taken to stabilize the sample. The rotational gears are preloaded by springs to limit sample vibrations. The manipulator probe assembly is supported by a thick-walled, 64 mm OD tube for rigidity. The X-Y stages slide on highly preloaded, crossed-roller-bearing slides with hardened sways. The Z stage slides on preloaded, full-area-contact linear bushings.

D. Detectors

To effectively conduct spectroscopic studies at a third-generation insertion device, fast detectors are required. The hemispherical electron energy analyzer [24] incorporates a multielement detector: a multichannel plate amplifies each detected photoelectron, and the resultant electron shower is directed onto a 16-wire anode. Sixteen separate sets of pulse-counting electronics can provide a collective count rate of over 1 MHz. The x-ray fluorescence from the sample will be detected by a multielement solid state detector with fast electronics, allowing a composite count rate of several hundred kHz.

For x-ray diffraction, a standard 2-circle crystal analyzer will be mounted on the end of the detector arm; a fast scintillator detector accommodating count rates up to 1.2 MHz will be used. For XSW, precise knowledge of the scattering angle is less important, and it is convenient to use a detector with a large solid angle, which should have a 2θ range of 0° to close to 180°. This will be accomplished by placing a PIN photodiode on a moveable track inside the UHV chamber.
CONCLUSIONS

With little compromise to any of the techniques desired, a versatile UHV endstation with capabilities for surface diffraction and spectroscopy has been designed. This diffractometer incorporates the most desirable elements from several other successful surface scattering endstations (e.g., S2D2 geometry, movable UHV chamber), as well as novel features (spectroscopic detectors, outboard sample position, highly adjustable sample manipulator) to achieve these goals. Experiments are planned to commence with this system in the winter of 1997-98 at the APS.

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REFERENCES

[18] Blake Industries, Scotch Plains, NJ.
[19] Model DPRF-400, McAllister Technical Services, Coeur d’Alene, ID.
[22] Model MB2002 X-Y-Z stage, McAllister Technical Services, Coeur d’Alene, ID. The X and Y stages have a range of ± 13 mm, and the Z stage has a total stroke of 38 mm.
[23] Model GB16, Thermionics Northwest Laboratories, Port Townsend, WA. The azimuthal (φ) and flip (χ) have ranges of ± 90° and -5°/+95°, respectively.