Surface And Interface Studies At APS Endstation 5ID-C

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Abstract. We have instrumented the 5ID-C hutch of DND-CAT at the Advanced Photon Source (APS) for x-ray surface spectroscopy and scattering. A wide variety of experiments have already been carried out at 5ID-C, including studies on semiconductors, oxides, metals, nanodots, and biomolecular adsorption. We describe the beamline optics, data collection hardware and software, and both diffractometers, a five-circle kappa diffractometer for open-air and liquid-cell experiments, and a psi-circle diffractometer with an integrated ultrahigh vacuum (UHV) chamber, paying particular attention to the requirements of an XSW measurement.

INTRODUCTION

This paper describes the 5ID-C hutch of the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT). This hutch has been designed for a variety of *in situ* and *ex situ* surface-sensitive experiments, including x-ray standing waves (XSW), grazing-incidence x-ray diffraction, and surface EXAFS. XSW measurements place stringent constraints on the experimental setup, requiring a highly collimated x-ray beam and the simultaneous measurement of the intensities of the diffracted beam and of secondary particles (typically fluorescent x rays, Auger electrons, or photoelectrons). After describing the beamline optics and beam conditioners, the two diffractometers in the hutch are described. Then the data-collection electronics are presented, focusing on critical factors for successful XSW experiments.

BEAMLINE OPTICS AND POSTMONOCHROMATORS

The insertion-device source on the 5ID beamline is a typical APS "Undulator A." The high-heatload monochromator (HHLM) consists of a pair of Si(111) crystals, the first of which is cryogenically cooled; an automated script allows feedback control of the detuning of the second crystal via a solenoid. A pair of 75-cm-long, horizontally deflecting glass mirrors provides 1:1 horizontal focusing as well as harmonic rejection. The reflecting surface can be bare glass or a Pt or Rh stripe as desired. These optics are sketched in Fig. 1.

Additional beam-conditioning optics, as shown in Fig. 1, are located on a stationary optical table at the upstream end of the 5ID-C hutch. The optical table sits on sandboxes for vibration isolation. The optical elements attach to the table's breadboard via Newport X95 rails and can be easily configured according to the needs of the experiment. Here we describe in some detail the typical setup for an XSW experiment. The HHLM does not generally yield sufficient collimation to resolve the rocking curve of a sample's Bragg reflection, where Darwin widths can be on the order of an arcsec. Thus, additional elements based on perfect-crystal optics, called postmonochromators, reduce the beam's vertical divergence and wavelength spread to an acceptable amount, for a given Bragg reflection of the sample under investigation (see Ref. [1] for a recent review of the XSW technique).

The postmonochromator setup at 5ID-C employs two nondispersive, two-bounce channel-cut crystals. Each X-Z-θ stage holds three Si channel-cut crystals; a simple horizontal translation switches between channel-cuts with symmetric Si(*hhh*), Si(*hh*0), and Si(*h*00) reflections to best match the dispersion of the sample's Bragg reflection. Additionally, the gap of each channel-cut can be set to either 10 or 15 mm, extending the usable Bragg angle (i.e., energy range) of each channel cut. Sub-μrad angular resolution is achieved for each channel cut by a piezo-driven

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rotary stage with a flex-pivot torsion bearing. Angular position is stabilized using error-integrated feedback from an electronic monochromator stabilizer (MOSTAB) [2]. Detuning the second channel cut with respect to the first improves the collimation of the beam at, of course, the expense of flux. Typically, both channel cuts are held at a constant detuning by the MOSTABs while the sample is scanned in angle. But for high-resolution scans such as Si(008), which are very sensitive to angular drift, the sample can be held constant while the angle of the first channel cut is scanned; differentiation of Bragg's law shows this is a rocking scan through a narrow range of energy. In this case, the second crystal is still held at a constant detuning by its MOSTAB, following the first channel cut and maintaining a narrow beam divergence.

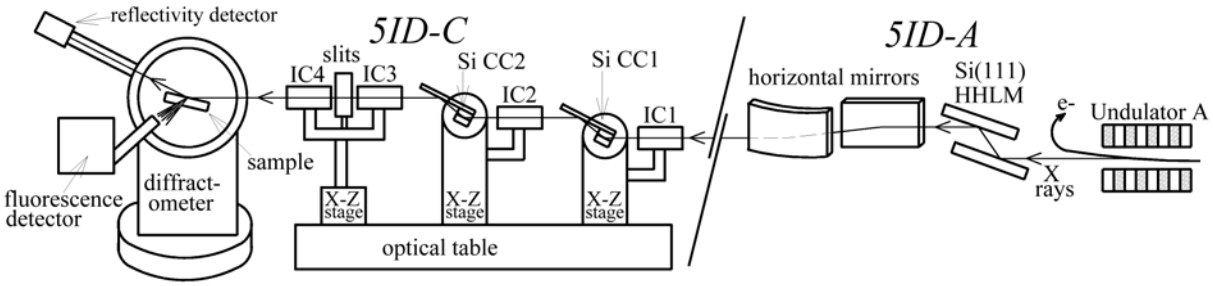

FIGURE 1. Schematic of XSW experimental setup: Beamline optics in 5ID-A; optical table containing postmonochromator elements, including Si channel cuts (CC's), ion chambers (IC's), and beam-defining slits; and diffractometer with reflectivity and fluorescence detectors.

FIVE-CIRCLE KAPPA-STYLE DIFFRACTOMETER

In addition to the specialized UHV diffractometer described below, we have commissioned a general-purpose diffractometer for open-air and liquid-cell experiments. This five-circle diffractometer is located immediately downstream of the postmonochromator optical table in the 5ID-C hutch, sitting on a heavy-duty kinematic table which, in turn, rests on sandboxes for vibration isolation. The diffractometer is a typical Huber Diffraktionstechnik kappa-style diffractometer that sits opposites a counterweight on a large fifth circle. The additional degree of freedom provided by the fifth circle is for performing surface and thin-film diffraction experiments at grazing incidence. Kappa geometry was chosen to maximize open space around the sample, both for positioning fluorescence detectors at a 90º scattering angle (to minimize Compton and thermal diffuse scattering) and for facilitating beam pipe installation when the downstream 5ID-D hutch is in use. Motorized XY and Z stages are available for the sample stage. Equipment on the detector arm (scintillator detector or ion chamber, slits, attenuator box, etc.) is attached with Newport X48 rail hardware.

The diffractometer is controlled by SPEC software [3] using *fivec* geometry code, whose convention we follow in referring to the angle names. SPEC allows a kappa diffractometer to be operated in standard Eulerian geometry by defining pseudomotors. To achieve acceptable angular resolution for typical scattering experiments, the larger circles (delta, k-theta, and mu) have 20:1 gear reducers. The smaller circles (kappa and k-phi) are typically driven with microstepping motor drivers for sufficient angular resolution to emulate a chi circle. In addition, the k-theta motor is highly microstepped in XSW experiments in order to perform fine scans through the sample's rocking curve. We have demonstrated the ability to perform XSW scans through the Si(008) reflection at 12.5 keV, where the Darwin width is 3.2 μrad [4].

PSI-CIRCLE UHV DIFFRACTOMETER

The premiere piece of equipment in the 5ID-C hutch is the UHV diffractometer. The design motivation and some features of this diffractometer were described previously [5]. The entire UHV chamber/diffractometer assembly sits on an X–Z table to center it in the x-ray beam, and can be rapidly and repeatably positioned in and out of the beam path by a 1 m translation on a pair of rails bolted to the floor.

The diffractometer (assembled by Blake Industries) has six rotational axes in a psi-circle configuration, also known as 4S+2D (so called for the four degrees of freedom for the sample and two for the detector), and is controlled with the *psic* geometry of SPEC [3,6]. Four of the axes (del, eta, mu, and nu) are high-precision angular

stages (Huber Diffraktionstechnik) with 20:1 gear reducers. The eta axis is typically microstepped for high-resolution rocking-curve measurements, which has been confirmed with autocollimator measurements; this motion is coupled to the UHV chamber through a two-stage, differentially pumped rotary feedthrough (model DPRF-400, McAllister Technical Services). The sample manipulator consists of a XYZ stage (model MB2002, McAllister Technical Services), a long, thick-walled tube, and the remaining two sample axes (model GB16, Thermionics Northwest). The two in-vacuum rotation stages, chi and phi, are useful for setting the orientation of the sample's surface normal as needed, e.g., switching between normal and off-normal reflections in an XSW measurements or switching polarizations in SEXAFS). However, because the chi and phi circles translate on the XYZ stage, phi is not in general collinear with eta for chi=0; therefore, chi and phi must remain fixed during a diffraction experiment (typically after the sample surface normal is set parallel to the eta axis). Detector-arm equipment is attached using Newport X48 rails, as with the kappa diffractometer, with the additional option of a two-circle analyzer stage.

One key design feature of this compact assembly is that the UHV chamber can translate independent of the diffractometer and sample, thus allowing one chamber position for x-ray scattering studies and a second chamber position for UHV tools that require a large solid angle. The UHV chamber connects to the diffractometer through large bellows; an air piston system counteracts the vacuum force through a set of pulleys and cables. In the scattering position, x-ray fluorescence can be measured by a solid-state Ge detector with a UHV interface, and electron spectroscopy can be measured using a Staib Instruments double-pass cylindrical mirror analyzer. A collimator and filter assembly eliminates background fluorescence from the chamber walls and elastic scattering from the Be windows from entering the Ge detector. Large Be windows on Conflat flanges allow x rays to enter and exit the chamber; a large, semicylindrical Be window is not welded to the chamber, as is traditional for surface diffraction chambers, due to space constraints and concerns of stress on the window due to heavy UHV instruments on nearby flanges. One rectangular Be window allows x rays to enter the chamber over a wide range of the angle mu. A 10-inch-diameter Be window allows a wide range of angles for x rays to exit the chamber, while another, smaller rectangle allows higher-angle scattering at mu and nu ~0º. For XSW experiments, an *in-vacuo* three-grid mesh detector fills in the range of scattering angles not accessible via the Be windows. Samples can be introduced and stored in UHV by transfer to a load-lock chamber. Additional sample preparation and characterization tools include radiative heating (to 1100 K), cooling (to 115 K), a Physical Electronics differentially–pumped ion sputtering system, molecular beam epitaxy via Knudsen cells and electron-beam evaporators, a reverse-view Omicron SpectaLEED low-energy electron diffraction system, a Staib Instruments DESA-100 retractable double-pass cylindrical mirror analyzer for Auger and x-ray photoelectron spectroscopy, an SRS RGA300 residual gas analyzer, an Omicron soft x-ray source, and a Staib Instruments electron flood gun. The base pressure of the chamber is $\sim 1.5\times10^{-10}$ Torr.

DATA-COLLECTION ELECTRONICS AND SOFTWARE

The wide range of experiments performed in 5ID-C necessitates specialized equipment to handle the various aspects of the measurements. A block diagram of the electronics is shown in Fig. 2. In the rest of this section we detail the data-collection hardware and software.

A well-thought out scheme for data collection is essential for synchrotron experiments, especially when several x-ray detectors operate simultaneously. A Joerger Enterprises 16-channel scaler is the input source for most of the detectors, which may include a scintillator, avalanche photodiode, or ion chamber (whose output signal runs through an SRS570 current amplifier and a voltage-to-frequency converter). Two methods have been used to collect full fluorescence spectra in a multichannel analyzer (MCA) for XSW and EXAFS experiments. Analog electronics can process fluorescence spectra from a single-element Ge solid-state detector, with the output sent to a PCA-3 MCA card, but such a scheme is inefficient for multielement detectors and the high count rates available at a synchrotron. Presently, fluorescence spectra are typically collected with a Canberra multielement UltraLEGe detector with a thin Be window and optional vacuum interface. Digital pulse-height analysis is performed by X-ray Instrumentation Analysis DXP-2X boards in a CAMAC crate; this system is controlled by Labview-based software called MESA. After initializing and calibrating each detector element with MESA, relevant parameters such as gain and shaping time are accessible by SPEC. We note that solid-state detectors must be electronically isolated to avoid noise-pickup originating from the microstep motor drivers.

Custom macros have been written to perform XSW scans in SPEC. At each point of a rocking-curve scan, the full spectrum can be collected from every detector element. When each scan is completed, typically after 32 points, a drift-control routine judges whether the rocking curve is acceptable and adjusts the center of the following scan. If

the current scan is accepted, the data are retained and the scan is repeated to build up statistics. After a given number of scans, the summation of data from good scans for is written to a file for each detector element. Data from the various elements are kept separately because in general they see different count rates and therefore have different deadtimes, and may also have varying depth sensitivity due to differences in take-off angle. The lowest channels in the MCA are preceded by data from the scaler and various information from the DXP, such as the calculated "livetime" and single-channel analyzer counts for preliminary analysis.

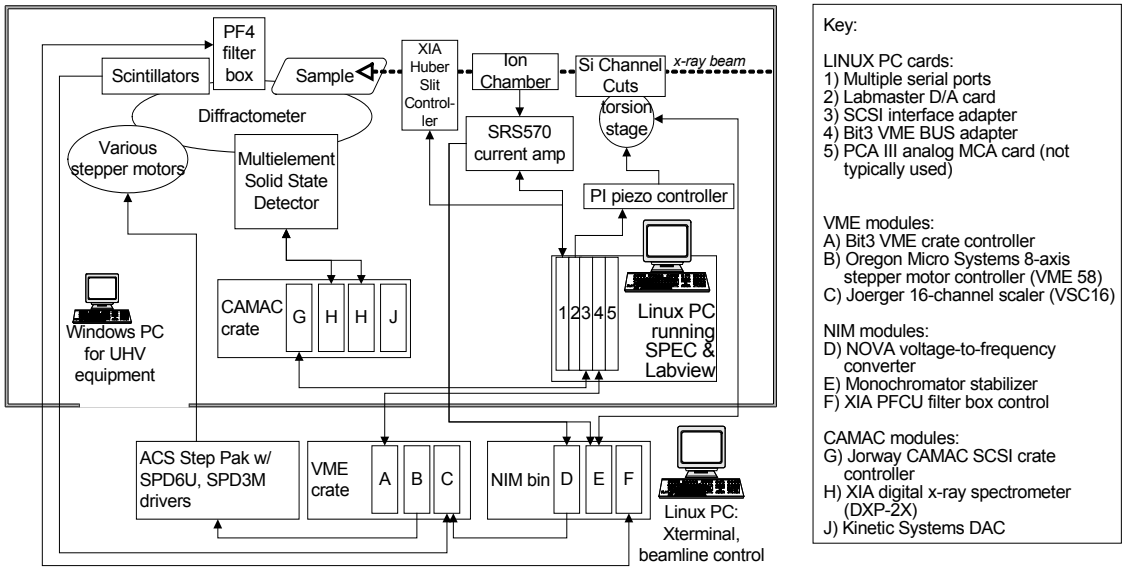

FIGURE 2. Block diagram of the data-collection and experiment-control electronics at 5ID-C. The hutch walls and x-ray beam are shown schematically. The LINUX computer inside the hutch is the primary control computer; the LINUX computer outside the hutch serves as an X-terminal to the inside computer and controls beamline operations through the SCIPE protocol [7]. The interface cards and computer boards are described in the key at right.

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REFERENCES

1. Bedzyk, M.J., and Cheng, L., "X-ray Standing Wave Studies of Minerals and Mineral Surfaces: Principles and Applications," in *Applications of Synchrotron Radiation in Low-Temperature Geochemistry and Environmental Science,* edited by P. Fenter, M. Rivers, N.C. Sturchio and S. Sutton, Geochemical Society, Washington, 2002, pp. 221-266.
2. Krolzig, A., Materlik, G., Swars, M., and Zegenhagen, J., *Nucl. Instrum. Method Phys. A* **219**, 430-436 (1984).
3. Certified Scientific Software, Cambridge, MA.
4. Tinkham, B.P., Goodner, D.M., Walko, D.A., and Bedzyk, M.J., *Phys. Rev. B* **67**, 035404 (2003).
5. Lyman, P.F., Keane, D.T., and Bedzyk, M.J., "UHV Surface-Analysis Endstation with X-ray Scattering and Spectroscopic Capabilities" in *Synchrotron Radiation Instrumentation: Tenth US National Conference*, edited by E. Fontes, AIP Conference Proceedings 417, American Institute of Physics, New York, 1997, pp. 10-14.
6. You, H., *J. Appl. Cryst.* **32**, 614-623 (1999).
7. Quintana, J.P.G., *J. Synchrotron Rad.* **5**, 600-602 (1998).