2004

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Citation: AIP Conf. Proc. **705**, 454 (2004); doi: 10.1063/1.1757832
View online: http://dx.doi.org/10.1063/1.1757832
View Table of Contents: http://proceedings.aip.org/dbt/dbt.jsp?KEY=APCPCS&Volume=705&Issue=1
Published by the American Institute of Physics.

Performance Characteristics of Beamline 6.3.1 from 200 eV to 2000 eV at the Advanced Light Source

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Abstract. Bend magnet beamline 6.3.1 at the Advanced Light Source operates from 200 eV to 2000 eV, primarily used for x-ray absorption fine structure investigations. The beamline optics consist of a compact, entrance-slitless, Hettrick-Underwood type variable-line-spacing plane-grating monochromator and refocusing mirrors to provide a 25 μm x 500 μm spot at the focal point in the reflectometer end station. Wavelength is scanned by the simple rotation of the grating and illuminates a fixed exit slit. The LabView based beamline control and data acquisition computer code has been implemented to provide a convenient interface to the user. The dedicated end station is a reflectometer that is isolated from the beamline by a differential ion pump. The reflectometer can position samples to within 4 μm with an angular position of 0.002°, has total electron and fluorescence yield detectors, and pumps down in about 30 minutes. External end stations can be mounted downstream of the reflectometer as well. The versatility and simplicity of beamline 6.3.1 have made it useful for a wide range of applications such as the characterization of optical components, reflective coatings, and the investigation of a diverse range of materials in both the solid state and in solution.

INTRODUCTION AND DESCRIPTION OF BEAMLINE 6.3.1

The Center for X-Ray Optics of the Lawrence Berkeley National Laboratory has built a user-friendly bend magnet Beamline 6.3.1 at the Advanced Light Source (ALS), which operates between 200 eV - 2000 eV. Beamline 6.3.1 is shown schematically in Fig. 1 and Table 1 summarizes the optical parameters. Beamline 6.3.1 has a dedicated precision reflectometer end station and also permits the use of an external end station downstream of the reflectometer. The scientific programs of beamline 6.3.1 are dedicated to EUV and soft x-ray reflectometry, scattering, and x-ray absorption fine structure (XAFS) measurements.

The horizontal focusing mirror, M1, is a concave spherical mirror that images the bending magnet source at the focal point. The M1 mirror is side-cooled by a water-cooled stainless steel block with a thin film of Ga-In eutectic for thermal contact. The vertically focusing mirror, M2, is an uncooled concave spherical mirror and demagnifies the source five times on the exit slit. Both the mirrors are single crystal silicon substrates coated with ~500 Å of Ru. The four-jaw aperture at the front end consists of four, moveable water-cooled copper plates defining the horizontal and vertical acceptance of the beamline. The maximum horizontal and vertical acceptance as determined by the M1 and M2 mirrors are 2.4 mrad and 0.44 mrad, respectively.

The monochromator is a compact, entrance-slitless, Hettrick-Underwood type variable-line-spacing plane-grating (VLS-PGM) design [1,2]. The entrance-slitless design results in maximal flux collection and the stability of the ALS electron beam prevents wavelength shifts over long periods of time. Furthermore, it operates in converging light from M2 allowing the beamline to be very short, resulting in high mechanical stability. It also permits better f-number matching for optics following the fixed exit slit. The wavelength is scanned by simple rotation of the grating about its center. The large spherical aberration of M2 is corrected by the coefficients describing the

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CP705, *Synchrotron Radiation Instrumentation: Eighth International Conference,* edited by T. Warwick et al.

variation of grating ruling [3], which allows the monochromator to reach a resolution limited by the ALS vertical source size.

The monochromator has four interchangeable, mechanically ruled blazed gratings (fused silica substrates coated with Au) with central grove densities of 240, 600, 1200 and 2400 lines/mm. A DC motor under the control of a closed loop servo system, using a Heidenhain encoder, drives the grating sine bar. The short exit arm of the monochromator results in a small linear dispersion at the exit slit and precision exit slits are required. Adjustments for position, parallelism, and tilt are required for the best performance. To meet these requirements, a mechanism was designed in which each of the four slit corners is independently actuated and position sensed to 0.1 μm. Downstream of the exit slits but upstream of M3, is a filter carousel, in which thin foils are used for higher harmonic rejection and calibration.

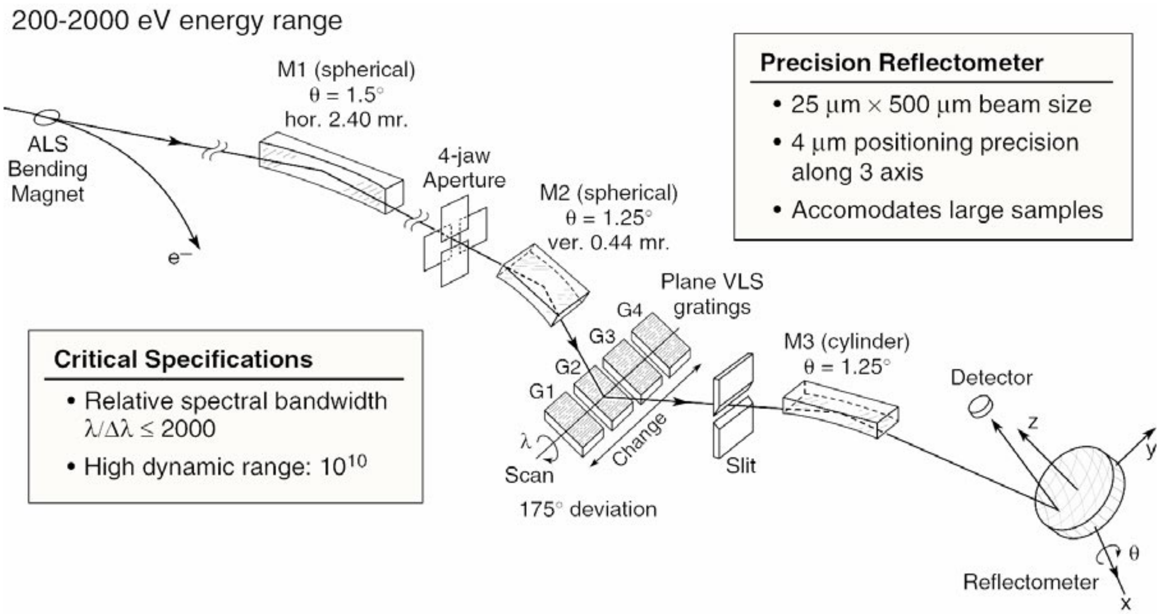

FIGURE 1. Schematic of beamline 6.3.1 at the ALS along with the most basic characteristics.

TABLE 1. Optical parameters of beamline 6.3.1.

Optical element	Distance (meters)	Angle θ (degrees)	Radius (meters)	Length (mm)	Width (mm)	Thickness (mm)	Comments
Source	0.0						
M1	10.95	1.5	487	1000	40	30	Bent
M2	16.0	1.25	244	400	38	10	Bent and profiled
Grating	18.0	2.5 (in ZO)	Plane	100	40	18	VLS
Exit slit	19.2	---	---	---	---	---	≥5 μm
M3	22.7	1.25	160	400	38	10	Bent
Sample	26.2	---	---	---	---	---	Focus of M1, M3

The final optical element, M3, is a vertically focusing concave spherical mirror with a variable curvature designed to image the exit slit at unit magnification at the center of the reflectometer chamber and also permits focusing further downstream. An unequal end couple mirror bender allows the mean radius of curvature of M3 to be varied and in addition provides a linear variation of the mirror radius and an image free from spherical aberration when M3 is used at other than unit magnification. M3 is a single crystal silicon substrate coated with ~500 Å of Ru.

The main experimental facility of the beamline is the dedicated reflectometer end station, which is a two-axis, vacuum system employing two Huber goniometers. The θ-motion axis accommodates the sample at the center of the reflectometer and the φ-motion supports the detectors [total fluorescence yield (TFY) and total electron yield] on a rotating arm. Linear motion translators position the sample in three directions (x, y, z) within 4 μm with an angular position of 0.002°. Although the reflectometer motions are primarily designed for measurement of precision optical components, they function as a sample manipulator with sufficient flexibility to allow a wide range of experiments. The sample holder can be removed and re-inserted with high precision into the θ-motion kinematic holder of the goniometer. The reflectometer section is isolated from the beamline by a differential ion pump, therefore the end station can be operated at pressures as high as 10^{-7} Torr with the beamline remaining at 10^{-10} Torr. The upstream end station isolation valve has a glass window, thus samples can be aligned using zero order light with the reflectometer at atmosphere. The reflectometer is equipped with a vibration-isolated 4000 l/s cryopump that permits 30-minute pump-down and the interchange of sample holders that mount multiple specimens.

An auxiliary end station for beamline 6.3.1 has been developed to perform XAFS measurements under atmospheric pressure and with liquids from 400 eV to 3000 eV using both transmission and TFY detection techniques [4]. Measurements using this end station are carried out under a He atmosphere at room temperature. The experimental setup consists of two optical shutters and a Si_3N_4 membrane at the end of the beamline, with vacuum on one side and atmospheric pressure on the other. Optical shutters are interlocked to the acrylic case that encloses the sample holder and whenever the case is opened, the optical shutters close to prevent exposure to x-rays. The beamline valves are interlocked to prevent venting the beamline should a Si_3N_4 membrane fail. For example, XAFS measurements of thin films deposited on Si_3N_4 membranes are done in transmission mode and the sample holder has a provision to hold the detector closely behind the sample. The sample holder is mounted on a stage where it can be moved along the beam direction; hence the sample is placed close to the Si_3Ni_4 membrane at the end of the beamline. For solutions, liquids, and solids, measurements are done in TFY mode using a photodiode. Solid samples are mounted at 45^o to the x-ray beam using carbon tape. Liquids and solutions are placed in a 6 mm diameter plastic cylinder that has a 1.5 mm - 2 mm slot. The liquid surface at the cylinder slot is exposed to the beam and the fluorescence is collected at 90^o with respect to the beam. The sample and the detector are kept close to the Si_3N_4 membrane to minimize absorption from the He atmosphere of the apparatus.

The beamline and reflectometer are controlled by a Sun workstation through a VXI crate. All monochromator, reflectometer (except sample holder interchange) and data acquisition functions are controlled from and displayed at the workstation. The software provides the capability for scanning wavelength (λ) or energy (E); scanning any of the reflectometer motions θ, φ, x, y, z; making special scans such as $\varphi = 2\theta$; and 2-dimensional scans using any pair of motions.

BEAMLINE 6.3.1 PERFORMANCE

Beamline 6.3.1 has been installed, aligned, and operational since March 2000. Recently, the full re-characterization of the resolution, flux, and wavelength calibration has been carried out following the improvement of an optical element. The beamline photon flux is presented in Fig. 2(a) and is on the order of 1×10^{11} to 2×10^{9} photons/sec/0.1%BW from 200 eV to 2000 eV. For measuring the monochromator resolution, a gas cell was used in the experimental location downstream of the reflectometer. Figures 2(b) and 2(c) show the XAFS spectra at the $L_{2,3}$-edge of Ar gas and K-edge of Ne gas collected using the 600 lines/mm and 1200 lines/mm gratings, respectively. These spectra have a spectral resolution of $\lambda/\Delta\lambda \approx 2000$.

The Mg K-edge extended-XAFS (EXAFS) spectrum from a Mg metal thin film capped with 10 nm of Pd using the auxiliary end station with the 2400 lines/mm grating and collected in transmission mode is shown in Fig. 2(d) [5]. The TFY XAFS spectra from single crystals of MgO and MgF_2 collected in the reflectometer are also included in Fig. 2(d). The XAFS spectra show characteristic edge shifts based on the bonding environment in the Mg metal, MgO, and MgF_2. The spectra of both MgO and MgF_2 show a sharp near-edge peak at ~1311 eV and 1314 eV, respectively, which are attributed to the 1s to 3p transition whose intensity depends on the degeneracy of the 3p states. This feature occurs at 1304.5 eV for metallic Mg. The interatomic distances and the coordination determined from the Mg metal EXAFS are in good agreement with crystallographic data up to the second shell [5].

The Al K-edge EXAFS spectra for crystalline $NaAlO_2$, $Al_2(SO_4)_3$, $Al(NO_3)_3$, and Na[Al(EDTA)] collected in TFY mode using the auxiliary end station with the 2400 lines/mm grating are shown in Fig. 2(e) [4]. The spectra in the near-edge region are characterized by two main features at ~1568 eV and ~1572 eV for the compounds studied. An additional feature at ~1564.5 eV is present for $NaAlO_2$. The feature at ~1568 eV is characteristic of six-fold Al coordination with oxygen for $NaAlO_2$, hydrated $Al(NO_3)_3$, and $Al_2(SO_4)_3$. Similarly, the feature at ~1564.5 eV for $NaAlO_2$ is characteristic of Al in four-fold coordination. The feature at ~1572 eV for all of the compounds results from a long-range-order correlation beyond the nearest-neighbor shell. Detailed analyses of the Al K-edge EXAFS are in progress and will be published elsewhere.

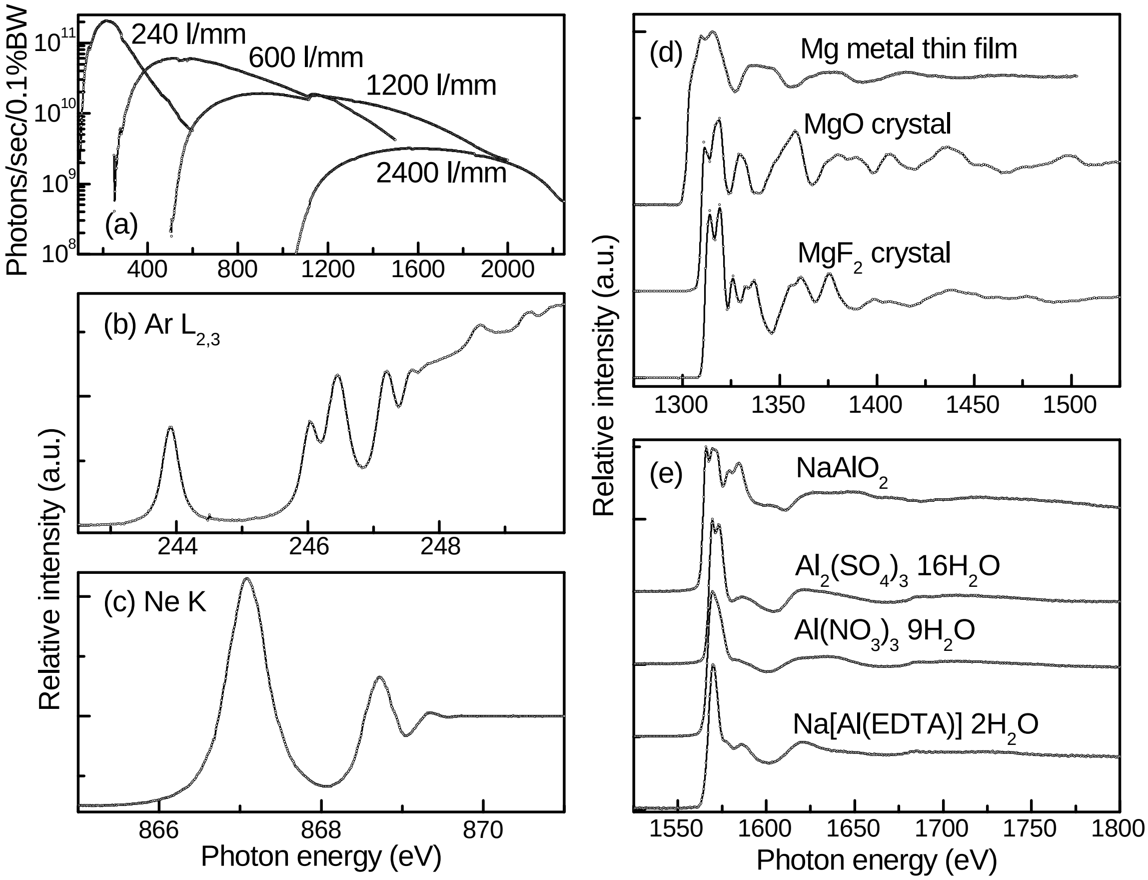

FIGURE 2. (a) Measured photon flux from the beamline for gratings having central grove densities of 240, 600, 1200 and 2400 lines/mm. (b) Ar $L_{2,3}$ and (c) Ne K-edge x-ray absorption spectra (0.5 Torr). The resolving power ($\lambda/\Delta\lambda$) is estimated to be $\approx$ 2000. (d) Mg K and (e) Al K-edge x-ray absorption spectra collected in total fluorescence-yield method except for Mg metal thin film which was collected in transmission mode.

ACKNOWLEDGMENTS

This work was supported by the Nevada NSF EPSCoR RING-TRUE program under Award No. EPS-9977809; the Nevada DOE EPSCoR State-National Laboratory Partnership program under Award No. DE-FG02-01ER45898; the Director, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences for the operation of the ALS and the Division of Chemical Sciences, Geosciences, and Biosciences of the proceeding U.S. DOE office, both under Contract No. DE-AC03-76SF00098 at the Lawrence Berkeley National Laboratory.

REFERENCES

1. Hettrick, M. C., and Underwood, J. H., in Short Wavelength Coherent Radiation: Generation and Applications-1986, edited by D. T. Attwood and J. Bokor, AIP Conference Proceedings 147, American Institute of Physics, New York, 1986, p. 237.
2. Underwood, J. H., and Gullikson, E. M., J. Electron Spectrosc. Relat. Phenom. 92, 265 (1998).
3. Hettrick, M. C., and Bowyer, S., Appl. Opt. 22, 3921 (1983).
4. Nachimuthu, P., Matsuo, S., Farangis, B., Lindle, D. W., Wakita, H., and Perera, R. C. C., J. Alloys and Compds., (2003) (in press).
5. Farangis, B., Nachimuthu, P., Richardson, T. J., Slack, J. L., Perera, R. C. C., Gullikson, E. M., Lindle, D. W., and Rubin, M., Phys. Rev. B 67, 085106 (2003); Richardson, T. J., Farangis, B., Slack, J. L., Nachimuthu, P., Perera, R., Tamura, N., and Rubin, M., J. Alloys and Compds. 356-7, 204 (2003).