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A high-performance double-crystal monochromator soft X-ray beamline

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A high-performance double-crystal-monochromator soft X-ray (DCMSX) beamline has been constructed at the Synchrotron Radiation Research Center (SRRC). This beamline delivers monochromatic photon beams with energies from 1 to 9 keV and a resolving power ($E/\Delta E$) of up to 7000. This beamline provides users with an opportunity to study many important materials, such as high-T_c superconductors, magnetic materials, catalysts, super-alloy compounds *etc.* Excellent EXAFS and NEXFS spectra have been routinely obtained from this beamline. Several interesting research projects are currently being conducted at this beamline. All the results show that this beamline has been constructed to meet its design goals.

Keywords: beamlines; soft X-rays; double-crystal monochromators.

1. Introduction

It has long been realized that there is much interesting science in the soft X-ray region. Soft X-rays can be used to study many important materials, such as aluminium, silicon, phosphorous, sulfur, chlorine and the first-row transition metals. These elements are very important in advanced materials development, heterogeneous catalysis, semiconductor industrial applications and physics and chemistry research. Therefore, several soft X-ray beamlines have already been developed at synchrotron radiation facilities (Paul *et al.*, 1995; Jones *et al.*, 1995; Robinson *et al.*, 1995; Schilling *et al.*, 1995; Yang *et al.*, 1992; Feldhaus *et al.*, 1986; Cowan *et al.*, 1986). SRRC has also constructed a soft X-ray beamline for both X-ray spectroscopy and scattering experiments. This paper briefly describes the beamline design and performance.

2. Beamline optical system

Two types of optical systems are most commonly used for soft X-ray spectroscopy beamlines: (i) in which a focusing mirror is employed before the crystal monochromator, and (ii) in which the monochromator is located between a vertical collimating mirror and a focusing mirror. The first optical system collects a wider horizontal radiation fan and also reduces the thermal loading on the monochromator crystal. However, the energy resolution is constrained by the divergence of the focused beam and the non-uniform beam footprint on the monochromator crystal. The second optical system requires an extra optical element, a vertical collimating mirror, but gains more flux and higher energy resolution primarily because the vertical collimating mirror can collect more photons in the vertical direction and, at the same time, eliminates the vertical beam divergence. Therefore, we have chosen the second type of optical design for the soft X-ray beamline at SRRC.

Fig. 1 shows the optical system layout of the soft X-ray beamline at SRRC. This beamline is designed to deliver photons with energies from 1 to 9 keV with an energy resolution of up to 7000. A double-crystal monochromator is located between the vertical collimating mirror and the focusing mirror. The beamline design parameters have been selected to match the radiation characteristics and to optimize the beamline performance. Details of the beamline design have been described elsewhere (Dann *et al.*, 1997). A set of carbon filters of various thicknesses are inserted before the collimating mirror to remove unwanted radiation (Feldhaus *et al.*, 1986) and reduce the thermal loading on the mirror and crystals. The filter can also cut down the background current and reduce unwanted reflections due to imperfect alignment of the crystal diffraction planes.

Two pairs of vertical slits are located before the monochromator to reduce further the beam divergence and hence increase the energy resolution whenever it is deemed necessary. The vertical slits can also effectively cut down the beam vertical divergence; however, they also seriously reduce the photon flux. Therefore, a collimating mirror is commonly used to reduce the beam divergence and thermal loading. The collimating mirror used here is a 700 mm-long water-cooled Glidcop mirror. There is a thermal load of 10 W deposited on the collimating mirror.

The double-crystal monochromator used in this beamline is a Cam-type manufactured by Kohzu Seiki Co. Ltd (Japan). The first crystal, which is water-cooled, only rotates when the second crystal is rotated and is moved to keep the exit beam fixed in direction and height. The angular scanning range is from 12 to 70°. The adjustments in 'pitch', 'roll' and 'Y' directions are performed with individual in-vacuum stepper motors. Fine adjustment in 'pitch' rotation for the second crystal is controlled by a PZT with a 60 arcsec adjustable range and 0.05 arcsec resolution. Three sets of crystals, Si(111), InSb(111) and beryl(1010), are used to cover photon energies from 1 to 9 keV. A pair of YB_{66} crystals will also be used for the 1–2 keV region (Wong *et al.*, 1995). Overall, the monochromator functions very well, although the chamber vacuum is only in the 10^{-8} torr region. Contamination of the crystal due to the residual gas has been observed.

A 1.4 m-long bendable toroidal mirror is located after the DCM, which is used as a focusing mirror. With the help of the *SHADOW* ray-tracing programs (Lai & Cerrina, 1986), we have found that the best focal point is at the 2:1 focusing condition. This is due to the aberration effect of the focus mirror (Forstner

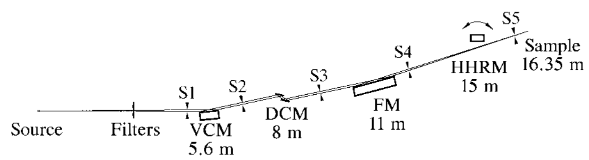

Figure 1

Optical layout of the DCMSX beamline. Source, synchrotron radiation from bending magnet. Filters, carbon filter set. VCM, vertical collimating mirror. DCM, double-crystal monochromator. FM, focusing mirror. HHRM, high-order harmonic rejection mirror. Sample, sample position. S1–S5, slits.

et al., 1992; Howell & Horowitz, 1975). The focused image size is about 1 × 0.5 mm (H × V). The mirror is coated with 2000 Å of gold for energies up to 9 keV, although a nickel coating is better for energies lower than 8 keV. A flat mirror is located before the end-station to remove the high-order harmonics. The end-station is mounted on an adjustable table to match the high-order rejection angle.

3. Beamline performance

Due to a delay in the arrival of the collimating mirror, the beamline has been commissioned with the monochromator, focusing mirror and several pairs of slits. Several experimental results related to the beamline performance are briefly described below.

A flux of 3×10^{11} photons s^{-1} is obtained at the beamline when the ring is operated at 1.5 GeV with 200 mA stored current. The energy repeatability is about 0.1 eV, which is obtained at the absorption edge as the monochromator scans across the edge several times. The energy resolution of the crystal monochromator is checked by monitoring the absorption spectra of several typical elements at different energies. Fig. 2 shows the *K*-edge (3202 eV) absorption spectrum of argon gas, which was obtained with a double-ion-chamber system. The gas pressure was kept below absorption saturation. The energy resolution is better than the 0.4 eV reported by Cowan *et al.* (1989) using a similar Si(111) crystal monochromator. Fig. 3 shows NEXFS spectra of some representative elements and compounds whose *K*-absorption edges cover the range 1–9 keV. All the *K*-edges are plotted relative to their absorption edge for clarity. These near-edge spectra are among the best in the literature to date. Fig. 4 shows the silicon *K*-edge EXAFS spectrum taken with the InSb(111) monochromator crystals. The splitting of the 'white line' shown in the near-edge spectrum (as shown in the insert) caused by the density of unoccupied conduction-band states is clearer than in any other spectrum reported (Yang *et al.*, 1992; Kitajima, 1995). These results indicate that the beamline has been well constructed.

A user-friendly compact multi-purpose XAS experimental station has been constructed to make the experiments easy and enjoyable. This station is equipped with several detection methods such as electron yield, fluorescence yield and transmission detection. The sample can be cooled to 173 K and heated to 573 K. Gases, liquids and solids can all be easily investigated at this station. The detection system consists of three identical ion-chambers, a fluorescence-detecting ion-chamber, slits and a filter set. The sample chamber is vacuum-tight, and can either be filled with helium gas or pumped down to eliminate the absorption in the low-energy regions. Three identical ion chambers are used for improved beam intensity measurement and therefore make the normalization of the measured spectrum more accurate.

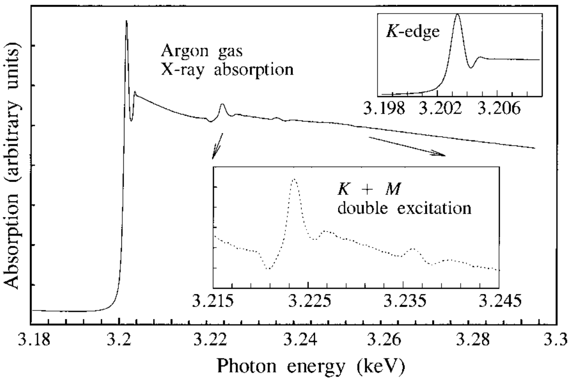

Figure 2
K-edge X-ray absorption spectrum of argon gas.

4. Conclusions

A high-performance soft X-ray beamline has been constructed at SRRC. This beamline provides a good monochromatic photon beam with energies from 1 to 9 keV and energy resolution of up to 7000. A multi-function X-ray absorption end-station has been established. Many excellent spectra have been routinely obtained from this beamline. Further analysis of these spectra is underway. A UHV soft X-ray scattering station has also been established (Hung *et al.*, 1997) and operated at this beamline. Preliminary results show that this beamline, despite being from a bending magnet, can provide excellent photons

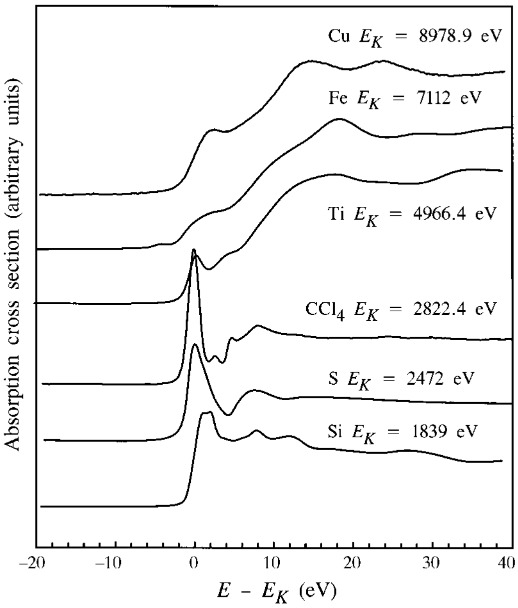

Figure 3
X-ray absorption spectra of various elements and compounds.

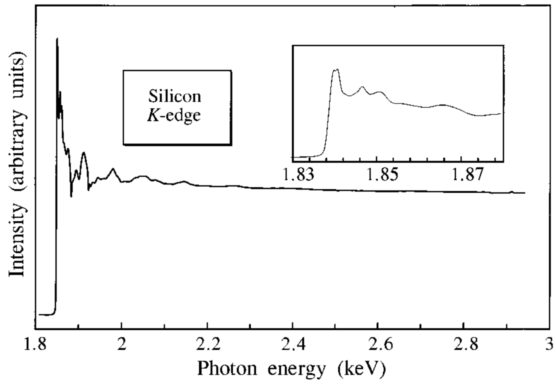

Figure 4
XAS spectrum of a silicon single crystal. The insert shows the detailed near-edge structure.

for soft X-ray scattering experiments. All the results show that this beamline has been well constructed to meet its design goals.

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