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Soft X-ray spectromicroscopy beamline at the CLS: Commissioning results

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Abstract

The soft X-ray spectromicroscopy beamline (SM) at the Canadian Light Source (CLS) is a dedicated spectromicroscopy facility, consisting of an elliptically polarized undulator (EPU), a beamline based on a collimated PGM optimized for 100–2000 eV range and two end stations: scanning transmission X-ray microscope (STXM) and roll-in X-ray photoemission electron microscope (X-PEEM, from Elmitec GmbH). The overall system has achieved its design parameters with an on-sample flux of $\sim 10^8$ ph/s@$R = 3000$, 0.5 A in STXM and $\sim 10^{12}$ ph/s@$R = 3000$, 0.5 A in the PEEM, in each case at a spatial resolution exceeding 40 nm. It can also provide an energy resolving power above 10,000. A careful EPU calibration procedure enables advanced polarization measurements.

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Keywords: Synchrotron radiation; Soft X-ray beam line; Spectromicroscopy; STXM; PEEM; EPU; PGM

1. Beamline layout and instrumentation

Construction of third-generation SR centers has led to a rapid development of beamlines specialized in soft and hard X-ray microscopy. In addition to a spatial resolution 1–2 orders of magnitude better than optical, such facilities provide a wealth of information from elemental and chemical composition to molecular orientation and magnetic order in specimens, which often can not be measured by any other technique [1]. The 100–2500 eV energy range of the Canadian Light Source (CLS) spectromicroscopy (SM) facility encompasses the C, N, O K-edges, essential for measurements of biological, environmental and polymer samples; Si L- and K-edges, Al K- and all the 3d transition metal L-edges, which are important for modern research in semiconductors and magnetism; P K-, Ca L-, As M- and the N-edges of heavy actinides, which are of interest for environmental science. The two end stations are also chosen to complement each other. Photoemission electron microscopy (X-PEEM) is a surface sensitive technique with a sampling depth of ~10 nm in total electron yield mode and ~1 nm with electron energy selection. The custom built scanning transmission X-ray microscope (STXM) is more bulk-sensitive, with optimal sample thickness dependent on photon energy (typically ~100 nm at the C K-edge). As a photon-in/photon-out technique, STXM can measure wet specimens and buried interfaces. The CLS SM facility has placed special emphasis on exploitation of X-ray polarization properties by building a specialized elliptically polarized undulator (EPU) which can deliver both circular and linear (−90 to 90°) polarized light. A dedicated beam line was proposed [2] and built based on an entrance-slit-less plane grating monochromator (PGM) design (Fig. 1a–c). The M1 mirror (located inside the radiation protective hutch) has a sagittal cylindrical shape to deflect the beam horizontally and produce light that is almost parallel in the vertical

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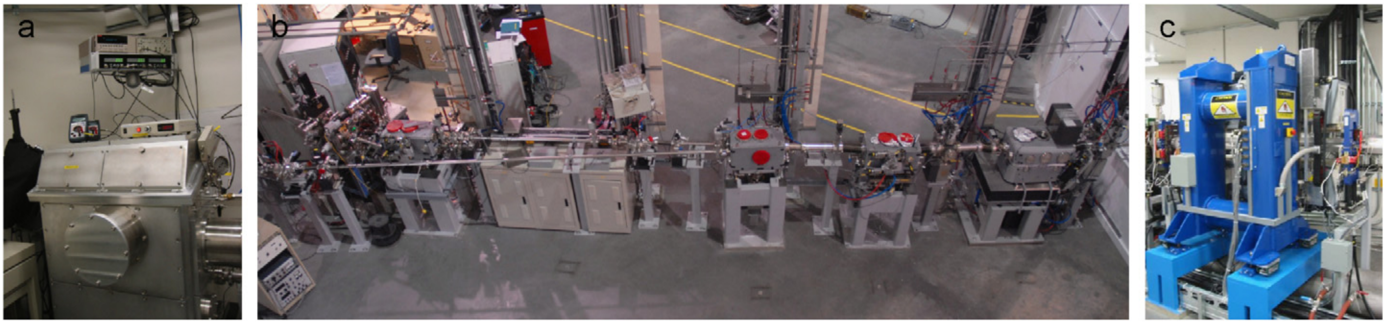

Fig. 1. SM beam line photos: (a) STXM; (b) SM beamline. SR light propagates from right to the left; (c) EPU.

direction. Since the PGM is illuminated with collimated light, a wide range of grating magnification (Cff) may be used. Three stripes (250, 500, 1250 mm) on a single grating substrate give an ultimate performance and phase space matching for the STXM. After the PGM, the beam splits into two lines. The M3PEEM movable mirror deflects the beam toward a short exit arm X-PEEM branch, while a stationary M3STXM mirror redirects the light into a long exit arm STXM branch when M3PEEM is retracted. Both M3 mirrors have a toroidal shape and focus light on the exit slits in both vertical and horizontal planes. The short exit arm favors the X-PEEM branch and provides strong compression of the horizontal beam size. The long arm (6 m) better matches the single diffraction mode of STXM operation. There is an additional M4PEEM demagnification mirror (ellipsoidal, with 3:1 ratio) which produces a small spot matched to the 50 μm nominal field of view in X-PEEM.

2. Achieved performance

The CLS-SM beamline has been operational since October 2005, but could only use upstream bend magnet radiation due to delays in completing and installing the EPU [3]. After the EPU was installed (May 2006), and the optical components properly aligned it was possible to reach an energy resolving power above 10,000 (Fig. 2). A new chicane scheme, which uses symmetrical deflection along the ID-10 straight, has been developed to allow optimal performance for both the SM and the future REIXS beamlines, as well as 2-in-1 modes [4]. Fine alignment of the EPU radiation along the beamline central axis was performed in June 2006. In order to achieve uniform illumination of the STXM zone plate, better than $\pm 30\,\mu$rad angular precision and an accuracy of mechanical positioning of the 1 ton STXM to within $\pm 200\,\mu$m is required. It took several iterations to get it right, but finally during September 2006 the first high-resolution STXM images were collected. Intensity measurements after the exit slit in the X-PEEM branch (Fig. 3a) and on sample in the STXM (Fig. 3b) confirm the flux is close to calculated performance: 3×10^{12} ph/s/0.5 A (X-PEEM) and 2×10^{8} ph/s/0.5 A (STXM), both for 3000 resolving power. The energy range was extended to the S K-edge (2500 eV) in the seventh harmonic. The EPU gap can be scanned synchronously with the PGM both in step-by-step and continuous (up to 10 eV/s) spectral acquisition. Excellent repeatability (~20 meV) and good absolute calibration (200 meV) of the PGM have been achieved. The second-order contribution was measured to be ~5% at 270 eV, but it can be reduced by using lower Cff or circular polarized light. PGM mirror to grating alignment leads to a beam lateral roll out lower that 20 mrad/deg. and M3 pitch adjustments are needed only when the photon energy is changed by more that 50 eV. After fine alignment of the STXM base plate, the Fresnel zone plate (FZP) translational stage and the interferometer mirrors the roll-out (lateral shifts during image sequences) is less than 200 nm over a 50 eV range. New FZPs (from the Center for X-ray Optics, LBNL) provide a STXM spatial resolution of better than 40 nm (Fig. 5a). Vibration and irreproducibility in nanocube piezo positioning is less than 10 nm. Other STXM upgrades include development of specialized devices, such as temperature controlled sample stages, a reliable cell for studies of liquids and wet samples, specimen holders with electrical contacts, as well as motorized stages for azimuthal and polar rotation, which are useful for orientation and tomography studies, respectively. The STXM sample support system is being modified to enable out of plane magnetism measurements. Future plans include development of a combined detector for dark field imaging; a CCD for X-ray scattering; and a fast avalanche photodiode for pump-probe measurements at 50 ps time resolution.

The X-PEEM microscope, purchased from Elmitec GmbH, was upgraded with the addition of an imaging energy filter, allowing photoelectron spectroscopy (XPS) and imaging with energy selected electrons. The energy analyzer provides up to 0.5 eV FWHM electron kinetic energy resolution and leads to enhanced chemical sensitivity. As a roll in instrument X-PEEM can be used at other CLS beamlines, but the high flux and small photon spot size of the SM beamline provides for very high-resolution PEEM imaging at X-ray energies (Fig. 4a).

The EPU energy and polarization are dependent on the gap and longitudinal displacement of the quadrants (phase).The gap and phase values for each condition were initially set to values computed from magnetic field

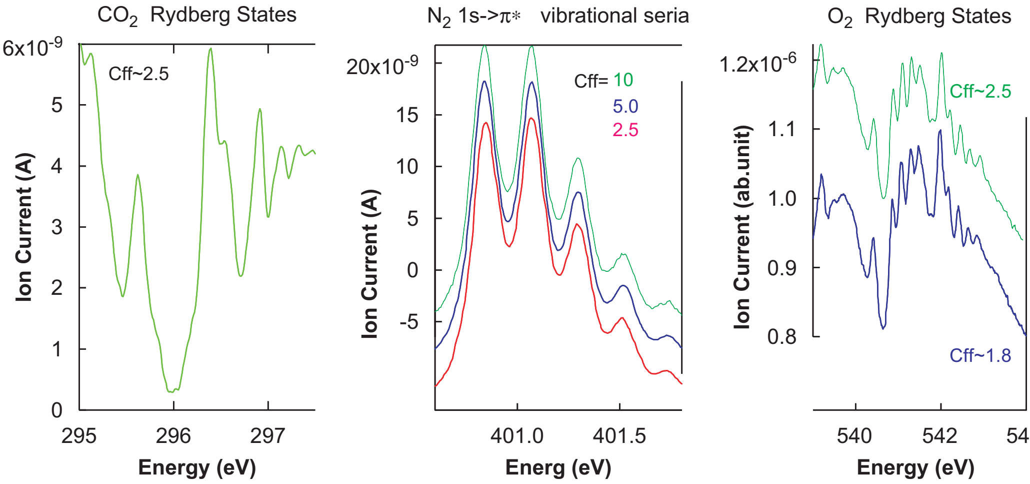

Fig. 2. Achieved energy resolution performance as measured by gas NEXAFS across C/N/O edges.

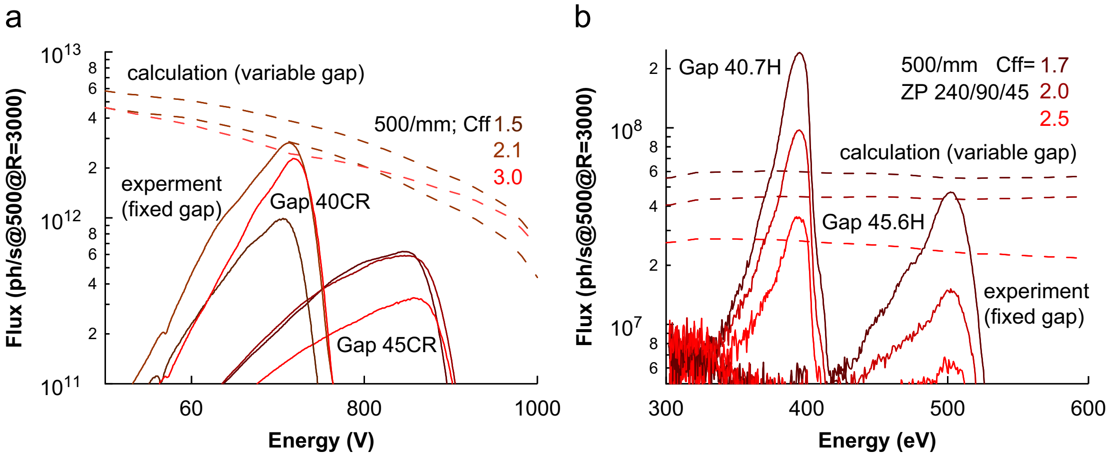

Fig. 3. On-sample flux curves, calculated (dashed) vs. measured at fixed gap (solid) for (a) PEEM and (b) STXM branches.

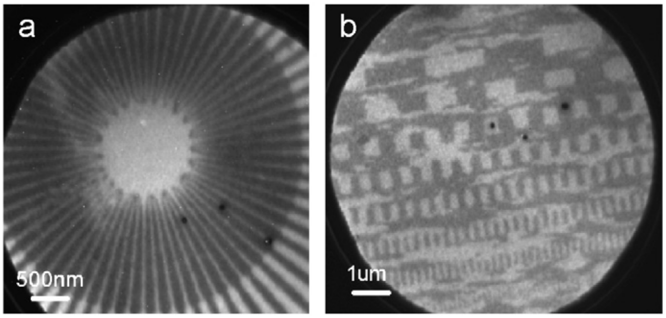

Fig. 4. (a) X-PEEM image of a nickel test pattern, prepared by Applied NanoTools Inc (ANT), recorded at the Ni 2p3/2→3d transition. The lines at the center of this pattern are 25 nm wide. (b) PEEM Image of a magnetically patterned hard drive surface (sample courtesy Andreas Moser, Hitachi) recorded at the Co 2p(3/2)→3d transition with left circular polarized X-rays.

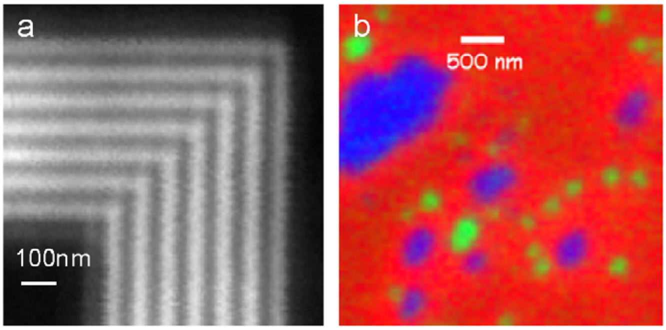

Fig. 5. (a) Demonstration of sub-50 nm resolving power of STXM (1:1 40 nm test pattern, developed by ANT). (b) Chemical analysis of #355 filler-particle polyurethane sample from a C 1s image sequence (red = polyurethane matrix, green = PIPA, blue = SAN filler particles).

measurements. Although a good shimming was achieved (first integrals were suppressed below 0.5 Gm, second do not exceed 1.5 Gm^2 and phase errors are below 7°), the moderate absolute accuracy of Hall probes does not provide sufficient accuracy for the EPU gap and phase parameters. Extended spectroscopic measurements followed by parametric fitting were used to further optimize the gap and phase look-up values. This reduced the deviation of predicted and optimal experimental gap to ~100 μm and provides excellent polarization performance

(Fig. 4b). SM beamline has reached or exceeded design parameters, passed required marquee experiments (Fig. 5b) and is open for general users.

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