# Soft X-ray Scanning Transmission Microscope Working in an Extended Energy Range at the Advanced Light Source

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**Abstract.** One of the ALS scanning transmission x-ray microscopes (STXMs) has been moved to the new ALS molecular environmental science beamline 11.0.2. The STXM is located on the microscopy branchline downstream of an elliptically-polarizing undulator delivering x-rays from 80 eV to 1900 eV. The wide energy range allows STXM operations with the current zone plate from 190 eV to about 1900 eV, the highest accessible energy with sufficient flux.

## INTRODUCTION AND DESCRIPTION OF THE ALS-MES STXM

Recently, a new molecular environmental science (MES) beamline (BL) has bean constructed at the Advanced Light Source (ALS) [1]. This elliptically-polarizing undulator (EPU) beamline delivers photons in the range from 80 eV to about 1900 eV to two branchlines: microscopy and spectroscopy. The existing scanning transmission x-ray microscope (STXM) from ALS BL 7.0.1 [2] has been moved and permanently installed in an isolated area on the ALS-MES microscopy branchline (11.0.2.2). The STXM branchline receives light from an LBNL variable angle-included plane grating monochromator [1] optimized for spectromicroscopy with an energy resolution ($E/\Delta E$) of ~7500. The beam is deflected and focused to the fixed exit slits of the microscopy branchline by an independent horizontal mirror. Between the exit slit and STXM is a fast shutter used to minimize sample radiation damage [3].

There were opportunities to implement several improvements and upgrades to the STXM as it was moved to the ALS-MES BL. The environment at the ALS-MES microscopy branchline is vibrationally more stable than at beamline 7.0.1 and coupled with the improvements to the mechanical stability of the STXM, has resulted in current STXM performance limited by zone plates rather than vibration. A new data acquisition and control system, similar to that of the beamline 5.3.2 STXM, was installed. Several mechanical stages were also replaced. The upgrade of the control system has permitted the ALS-MES STXM to adopt and utilize the same control program as the 5.3.2 STXM [1]. Thus, control and operation of the two STXMs at the ALS are identical to the user.

## STXM PERFORMANCE

The currently operating zone plate is 155 μm in diameter and the smallest zones are 35 nm wide. The zone plate was fabricated from a 100 nm thick Au layer on a 100 nm thick $Si_3N_4$ window. The central stop is 65 μm in diameter and is 1000 nm thick. The window area around the zone plate is masked by the continuous Au layer. The theoretical resolution for this zone plate is 43 nm. The zone plate was made by Center for X-ray Optics of the LBNL. Fig. 1 shows that thanks to the high quality of the current zone plate, 25 nm lines with 1:1 spacing are well

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resolved in the STXM image of another zone plate, used as a test pattern. The current zone plate is optimized for operation at 340 eV and has an experimentally measured efficiency of about 9% at this energy. The high flux of diffraction limited illumination from the undulator source and beamline optics, makes the ALS-MES STXM an excellent tool for spectromicroscopy in the 280 eV to 1000 eV energy range.

The wide energy range and high photon flux available at the ALS-MES beamline allows STXM operations with the current zone plate beyond the Si K edge but short of the P K edge. Despite the relatively low zone plate efficiency at higher energies, the ALS-MES STXM has sufficient flux for measurements up to about 1900˚eV. At this energy, the zone plate efficiency is about 1%. The negative side of such low efficiency is lower flux of the focused beam and a higher background. The measured background was about 8% of the flux at this photon energy.

For photon detection in the current ALS-MES STXM, a photomultiplier tube (PMT) with a scintillator material or a commercial Si photodiode is employed. Depending on the experimental requirements, one of two readily-available scintillator materials is used: P43 or P47 phosphors. P43 has a higher efficiency (50 % to 80 % higher than that of P47) but also has a relatively long persistence (about 1 ms). Thus, this scintillator material is utilized when radiation damage is a concern. Typically, the PMT works in photon counting mode, but the dead time starts to become significant at about 10 MHz. This high counting rate is easily achievable with diffraction-limited illumination of the zone plate at the ALS-MES STXM for photon of energies up to ~1500 eV. In practice, the exit slits of the ALS-MES microscopy branchline have to be closed to about 20 µm x 20 µm for STXM measurements from 400 eV to 1000 eV when acquiring in the photon counting mode and results in correspondingly high spectral resolution. The full photon flux of the microscopy branchline can be utilized by using the PMT in the analog mode.

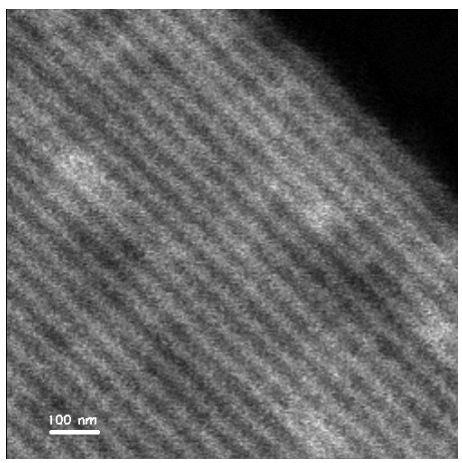


**FIGURE 1.** The STXM image of 25 nm zones of a zone plate. The image size is 1 µm x 1 µm.

## EXAMPLES OF HIGH ENERGY IMAGING

The ALS-MES BL, although operational and regularly performing scientific investigations, is just emerging from the end of the full commissioning phase. During this period of final commissioning, the ALS-MES STXM has been used extensively for the investigation of a wide and diverse range of scientific topics. The major focus is on MES investigations of systems of increased complexity. One new type of study enabled by the EPU, a specially-designed magnet, and the capability to scan the sample at 30¡, is characterization of the nanoscale magnetization of materials. The high energy capabilities provide new opportunities for spectromicroscopy studies of the later transition metals, environmentally-relevant elements such as As and Se, and important elements such Al and Si.

### Al K absorption edge

The first spectromicroscopy studies at the Al K edge with STXM have been performed at the ALS-MES BL. These experiments have focused on the characterization of Al model materials that will serve as references for

environmental investigations. Fig. 2 shows boehmite particles in a hydrated organic film measured in a STXM wet cell [4]. The STXM performance at the Al K edge has spatial resolution better than 50 nm and the high flux permits rapid collection of images. High quality near-edge spectra from the 200 nm boehmite particles were obtained.

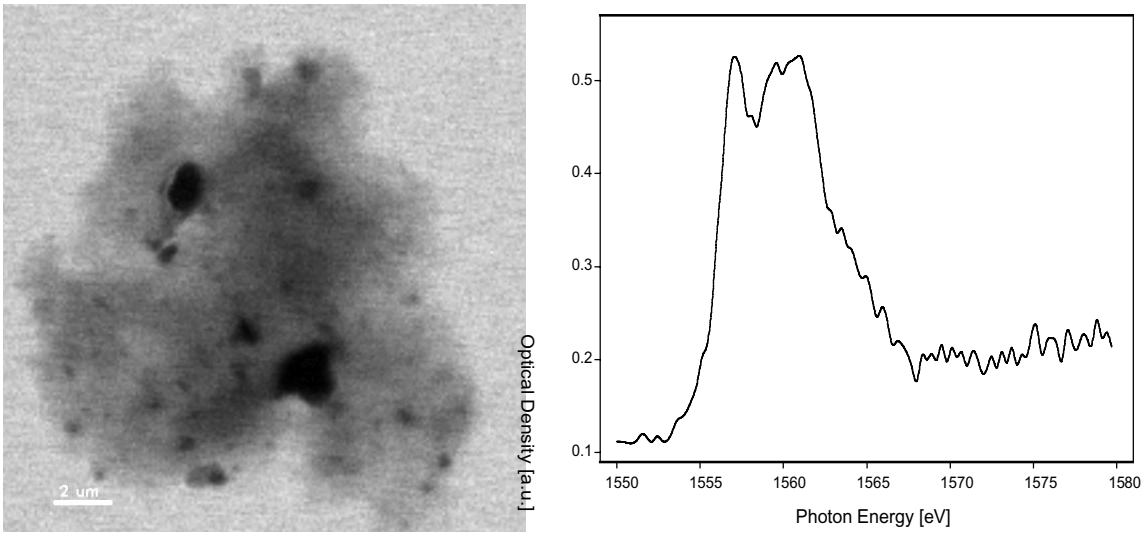


**FIGURE 2.** (a) STXM image of boehmite particles embedded in a hydrated organic film obtained at the Al K edge (1557 eV) [4]. The image is 18 µm x 18 µm (300 x 300 pixels, 670 µs per pixel) taken with the ALS operating at 250 mA. (b) Al NEXAFS spectrum of one of the small particles (200 nm). [Sample from T. H. Yoon and G. E. Brown, Jr., (Stanford Univ. and SSRL)]

## Si K absorption edge

Silicon is currently the highest energy K edge (1840 eV) obtainable at the ALS-MES STXM and Fig. 3 shows an image collected from Black Sea particulates [5]. Even at the Si K edge energy, the photon flux is greater than 1 MHz and this is sufficient for high quality imaging with 1 ms per pixel acquisition time. There is no abrupt contrast from these particles, thus the spatial resolution of this image is difficult to assess but is better than 100 nm.

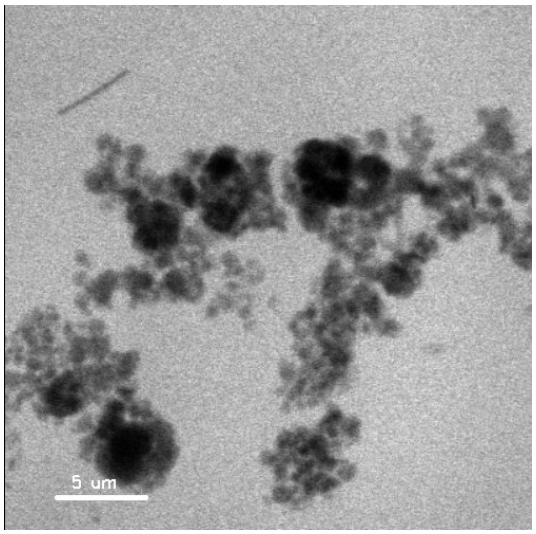


**FIGURE 3.** The STXM image of Black Sea particulate matter collected near the Bosporus inflow collected at Si K absorption edge (1843 eV) [5]. The image is 30 µm x 30 µm (400 x 400 pixels, 1.1 ms per pixel) with the ALS operating with a 240 mA storage ring current. [Sample from B. Tebo (UC San Diego- Scripps Institute of Oceanography]

## Cu L absorption edge

One of the unique applications of the ALS-STXM is to magnetic materials since it is downstream of an EPU, has excellent flux in the region of the transition metal L-edges, and has a flexible experimental arrangement. The combination of high resolution spectromicroscopy and beamline characteristics has permitted successful STXM studies of a complex magnetic microdevice even under two-bunch operating mode with low storage ring currents. This shows that the timing structure of the ALS can be used to investigate time-dependent processes using STXM. Fig. 4 shows an image of a seven-layer magnetic structure composed of Pt, $SiO_2$, Co, Cu, Co, Fe, and Cu. Although, this image was obtained at the Cu $L_3$, all of the relevant absorption edge spectra and images were collected from this structure as well as the x-ray magnetic circular dichroism of the Co and Fe layers (not shown) [6].

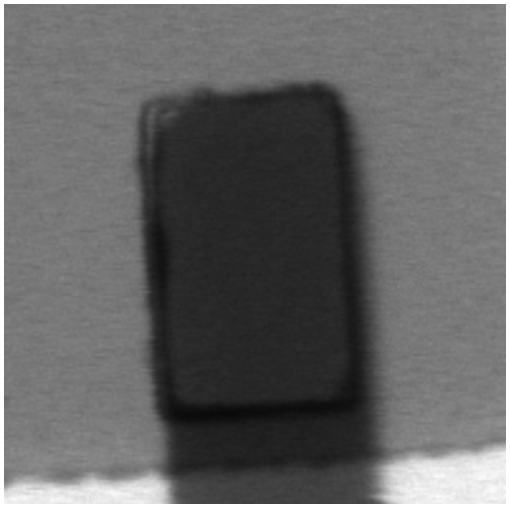

**FIGURE 4.** STXM image (4 µm x 4 µm, 300 x 300 pixels, 600 µs per pixel) of a microdevice connection pad consisting of Pt, Co, Fe, and Cu layers obtained at the Cu $L_3$ edge (928 eV) [6]. The ALS was operating in two-bunch mode at 20 mA. The edges show thicker Cu layers than the main part of the structure. [Sample from Y. Acremann and J. Stohr, (Stanford Univ. and SSRL)]

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## REFERENCES

1 T. Warwick, N. Andresen, J. Comins, K. Kaznacheyev, J. P. McKean, H. A. Padmore, D. K. Shuh, T. Stevens and T.˚Tyliszczak, New Implementation of an SX700 Undulator Beamline at the Advanced Light Source, this volume.
2. A. L. D. Kilcoyne, T. Tyliszczak, W. F. Steele, S. Fakra, P. Hitchcock, K. Franck, E. Anderson, B. Harteneck, E. G. Rightor, G. E. Mitchell, A. P. Hitchcock, L. Yang, T. Warwick, and H. Ade, J. Synchrotron Rad. 10, 125 (2003).
3. A. L. D. Kilcoyne and T. Tyliszczak, Fast Soft X-ray Beam Shutter, this volume.
4. T. H. Yoon, S. B. Johnson, C. S. Doyle, K. Benzerara, T. Tyliszczak, D. K. Shuh, and G. E. Brown, Jr., "*In-situ* Characterization of Nanometer-sized Al-containing Particles Using Aluminum K-edge Soft X-ray Spectromicroscopy. *Langmuir* (submitted).
5. The sample is courtesy of B. Tebo (Scripps Institute of Oceanography-University of California San Diego).
6. S. Andrews, V. Chembrolu, J. Strachan, Y. Acremann, and J. Stohr, (Stanford University and SSRL), to be published.