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### Title

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## A new Scanning Transmission X-ray Microscope at the ALS for operation up to 2500eV

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**Abstract.** We report on the design and construction of a higher energy Scanning Transmission X-ray Microscope on a new bend magnet beam line at the Advanced Light Source. Previously we have operated such an instrument on a bend magnet for C, N and O 1s NEXAFS spectroscopy. The new instrument will have similar performance at higher energies up to and including the S 1s edge at 2472eV. A new microscope configuration is planned. A more open geometry will allow a fluorescence detector to count emitted photons from the front surface of the sample. There will be a capability for zone plate scanning in addition to the more conventional sample scanning mode. This will add the capability for imaging a massive sample at high resolution over a limited field of view, so that heavy reaction cells may be used to study processes in-situ, exploiting the longer photon attenuation length and the longer zone plate working distances available at higher photon energy. The energy range will extend down to include the C1s edge at 300eV, to allow high energy NEXAFS microscopic studies to correlate with the imaging of organics in the same sample region of interest.

Keywords: x-ray scanning microscope

#### **BEAMLINE DESIGN**

Microscope illumination requires modest spectral resolution for NEXAFS spectroscopy. The goal is resolving power 3,000, as in the existing beamline [1], which covers the energy range 250eV to 600eV and at a similar facility at the Swiss Light Source [2]. In the new ALS facility the energy range will be extended to 2000eV. And further to 2500eV with diminished flux and diminished spectral resolution. In order to deliver higher energy photons the deflection angles of the beam line optics are shallower. Angles and arm lengths are constrained by the available space for beam line components and the location of the new microscope. This will be inside the same room as the existing microscope.

The phase space acceptance of the zone plate is small, so that only the on-axis coherent fraction of the illumination is useful. The beam line optical scheme is effectively paraxial, and the depth of focus of the grating is large. By choosing the grating radius carefully, and operating over a range of grating angles where the dispersion is low, it is possible to design a scheme where the grating rotation range for a given energy range is small and the defocus limit to the spectral resolution is better than R=3,000 over a fairly large energy range.

Flux and detector count rate is a concern. We need data rates at least comparable to those in the present instrument. In other words MHz detector count rates to give an image acquisition time of the order of a minute. The on-axis spectral brightness of the bend magnet source at higher energies is approximately the same as at lower energy (given the spectral bandwidth). The reflectivity is less, even at shallower angles. The transmission through the windows and the efficiency of the detector is improved.

Figure 1 shows the optical layout and parameters of the new beamline alongside the existing beamline.



FIGURE 1. Optical parameters, new beam line in light grey alongside existing beam line in darker grey.

The monochromator is designed by varying the grating radius and the arm lengths, with the 200lines/mm grating reaching down to 500eV and consistent with the space constraints shown above. The operational range is covered with low dispersion to keep the defocus resolution limit better than about R=3,000 from 500eV to 2000eV at 200 lines/mm. And better than about R=4,000 in the middle of this range. The entrance slits and exit slits are placed at precise distances from the grating. There is a corresponding slope error limit to the resolution. This leads to the slope error specification for the grating. We would like to maintain R=3,000 up to 2000eV so we need  $0.5\mu$ rad RMS grating tangential slope error or better. Figure 2 shows ray trace verification of the design.



**FIGURE 2.** Ray-tracing for 5 values of the photon energy across the range of the 200 lines/mm grating. Three spectral lines are included in each case, separated by 1:1,000. The monochromator is in-focus at 620eV and 1670eV. With this grating, R=3,000 is lost at 2500eV.

Figure 3. shows the estimated detector count rates at the zone plate focus with exit slit aperture set for the nominal spectral resolution of R=3,000. This curve includes an estimate for enhanced window transmission as the energy increases and a detector efficiency climbing to 100% at 1000eV. The zone plate diffraction efficiency is taken as 5%. Optical surfaces are gold coated. These rates are an order of magnitude higher than the estimates made (at lower energy) for the existing STXM. Reducing the exit slit size by a factor 4 in both directions to reach the diffraction limited spatial resolution will cost a factor 16 in detector count rate, still acceptable for imaging and absorption NEXAFS spectroscopy.



FIGURE 3. Estimated detector count rates at R=3,000.

#### THE NEW MICROSCOPE

Up-to-date stage technology will allow this new instrument to be compact, and openplan. We want to be able to dismount the fine-scan sample stage stack and replace it with as-yet unspecified heavy samples. For example, these might be in-situ reaction cells, or tomography stages. This will alternatively provide a standard fine-scan configuration for lightweight samples, as in the two existing STXMs at ALS and a flexible platform for mounting user-designed in-situ experiments.

Imaging by zone-plate scanning will be required for imaging when the fine-scan sample stage is dismounted. This can be accomplished over a restricted range ( $40\mu m$ ) within the overlap of the ZP central stop and the OSA.

The ZP scan assembly will carry one set of x/y interferometer reflectors and the sample scan assembly will carry the other set. The interferometers will monitor the relative transverse position of ZP and sample regardless of how the sample is positioned or which scanner is in use to make images. Early tests with a new interferometer show greatly improved precision over our prior STXM installations, see Figure 4.

The open architecture is shown on figures 5 and 6. This will facilitate fluorescence detection at the higher photon energies. We will follow the success at Elletra [3] and implement fluorescence yield detectors in a back scattering geometry for use with thick samples.



**FIGURE 4** First interferometer measurements of 20nm open-loop scanstage sinusoidal motion, showing vibrations of the order 2nm.



FIGURE 5 Test configuration of the scan stages



FIGURE 6 Layout of functional components

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#### REFERENCES

- A New Bend Magnet Beam Line for Scanning Transmission X-ray Microscopy at the Advanced Light Source T. Warwick, H. Ade, D. Kilcoyne, M. Kritscher, T. Tylisczcak, S. Fakra, A. Hitchcock, P. Hitchcock and H. A. Padmore, J. Synchrotron Rad. (2002) 9, 254-257
- PolLux: A new facility for soft x-ray spectromicroscopy at the Swiss Light Source" J. Raabe, G. Tzvetkov, U. Flechsig, M. Boge, A. Jaggi, B. Sarafimov, M.G.C. Vernooij, T. Huthwelker, H. Ade, D. Kilcoyne, T. Tyliszczak, R.H. Fink, C. Quitmann, Rev Sci Instruments 79, 113704 (2008)
- A Low Energy X-Ray Fluorescence Spectrometer for Elemental Mapping X-Ray Microscopy R. Alberti, A. Longoni, T. Klatka, C. Guazzoni, A. Gianoncelli, D. Bacescu and B. Kaulich, 2008 IEEE Nuclear Science Symposium Conference Record