Status of the 5.3.2 bend magnet polymer STXM

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INTRODUCTION

Over the past three years a qualitatively new design of Scanning Transmission X-ray Microscope (STXM) has been conceived, built and commissioned at beamline 5.3.2 at the ALS. A dramatic improvement in performance has been achieved by introducing laser interferometry to control the positioning of microscope components with an accuracy consistent with the optical resolution of the instrument, currently about 40 nm. The interferometer is a great advance over previous systems such as the old BL7.0 STXM at the ALS and the Stony Brook STXM at the NSLS. In the latter instruments, one could image at a fixed photon energy with a spatial resolution close to the diffraction limit of the zone plate utilized, but it was not possible to change the photon energy while maintaining the focused X-ray spot on the same place on a sample, or to image at successive energies over the same x-y frame. Hence, the spatial resolution was effectively degraded for chemical analysis. Over a typical C 1s NEXAFS scan from 280 to 320 eV the focal length changes by up to 300 μm, requiring displacement of the zone plate along the X-ray beam by this amount while simultaneously maintaining the lateral position of the zone plate relative to the sample with <20 nm precision. Mechanical systems achieving the required linearity of $>10^4$ are exceptional, and indeed, the experience at both the ALS and NSLS microscopes has been that spatial resolution in point spectral mode is rarely better than 200 nm and that one needs to use software to correct for image-to-image misalignment, with concomitant residual resolution degradation.

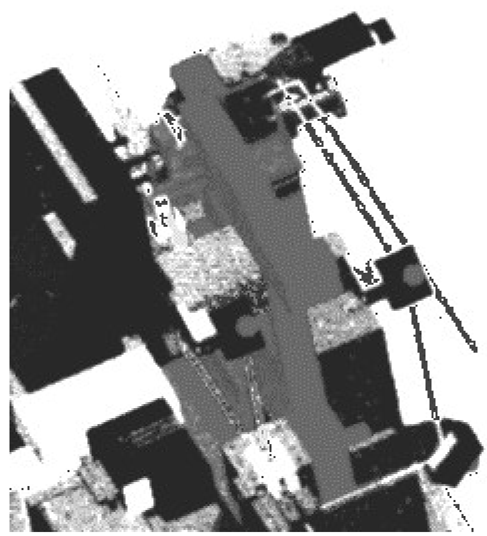

Fig. 1. View of the sample and zone plate stage region of the 5.3.2 STXM showing the paths of the laser beams used for 2-d interferometric control of relative position of the sample and the focused light.

EXPERIMENTAL

The solution that has been developed by a team from several institutions, adopted for both the 5.3.2 STXM and the 7.0 upgrade with somewhat different implementation, is to introduce a 2-d laser interferometer which continuously monitors the relative (x,y) position of the zone plate and the sample (see **Fig 1**). In the 5.3.2 STXM the interferometer is used as part of the feedback control loop for the PI piezo fast scan stage which is used to position and scan the sample when making images. This system provides stability of

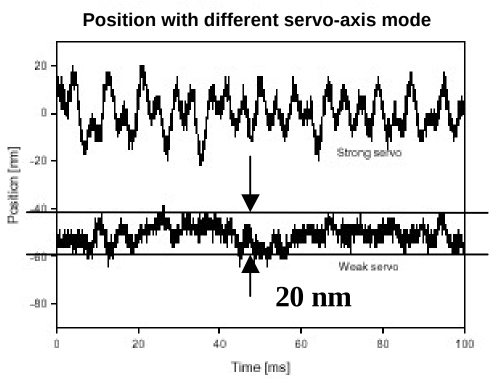

Fig. 2. Tests of positional stability over time of the sample position when under interferometric control (Tyliszczak, Feb-01)

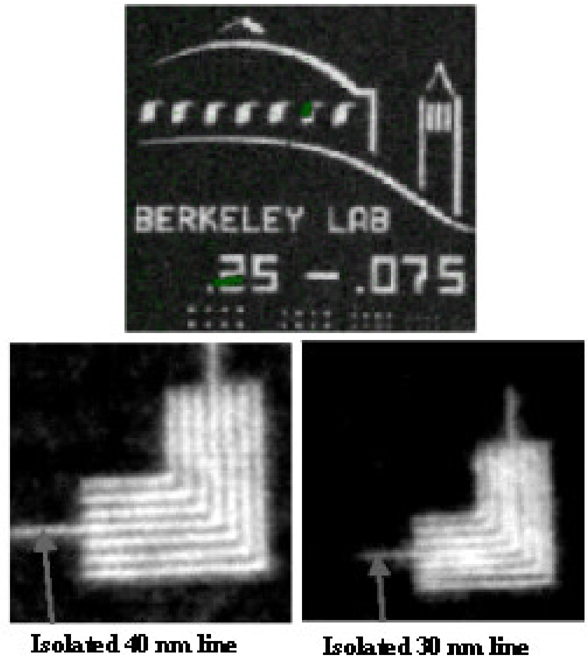

Fig. 3. Resolution tests using 1:1 Au lines on a silicon nitride substrate. The 40 nm pattern is fully resolved; the 30 nm pattern is partially resolved. (Each pattern consists of alternating lines and spaces of 30 or 40 nm). Zone plate and test pattern prepared by Erik Anderson *et al.* (CXRO, LBNL)

better than 20 nm with response times up to 100 Hz (see **Fig 2**) and thus it not only eliminates energy-to-energy image position errors, but also helps to desensitize the microscope to vibrational or other environmental noise.

With this mechanical/optical/control system, accompanied by advanced control and acquisition software, as well as excellent performance of the 5.3.2 spherical grating monochromator beamline [1], the 5.3.2 STXM is able to achieve diffraction limited spatial resolution. The images of a Au test pattern (**Fig 3**) indicate a spatial resolution of better than 40 nm, which is the theoretical diffraction limited resolution of the zone plate used. More significantly for analytical applications, the interferometry allows the 5.3.2 microscope to retain image registry at all photon energies. As an example of the level of positional stability achieved with the interferometry system, **Fig. 4** compares a linescan data set acquired at BL 5.3.2 in August 2001 on a polyurethane sample containing nanoscale filler particle with a data set on a similar sample recorded over a similar spatial and photon energy range, acquired with the old ALS beamline 7.0 STXM. In each case the raw data - position along line versus photon energy - is plotted. Clearly the ability to determine the NEXAFS spectra at high spatial resolution is greatly enhanced by the improved performance of the 5.3.2 STXM.

Further work on both the beamline and the mechanical and controls system has further increased stability and reliability of the 5.3.2 STXM [2] relative to its performance when these test measurements were performed.

REFERENCES

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2. A.L.D. Kilcoyne *et al.* J. Synchrotron Rad. In preparation

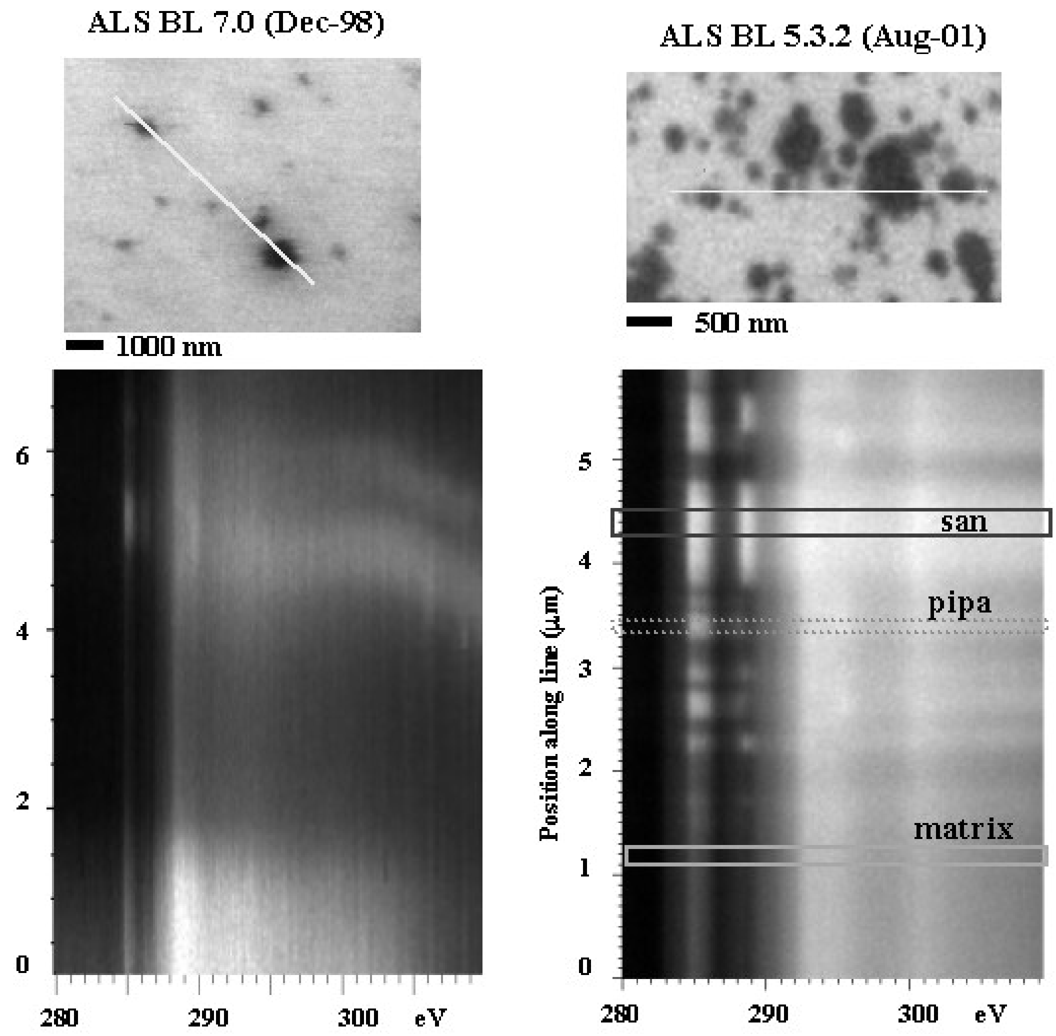

Fig. 4.
the old BL7.0 STXM and the new 5.3.2 STXM using data from filler particles (pipa) in a low-water polyurethane sample. The two lower 'images' plot ‘linescans spectra’ measured with each instrument. The display shows photon energy horizontally and position vertically - successive horizontal lines are the spectrum at pixels nominally along the line indicated in the true OD image of the samples displayed on the top (BL7.0: pipa only; BL5.3.2: pipa and san). The waviness of the signal in the horizontal direction in the left hand linescan is caused by drift of the X-ray spot on the sample due to mechanical limitations. In contrast, in the right hand linescan, there is negligible waviness in the horizontal direction. This is because the interferometric control in the 5.3.2 STXM provides precise zone plate - sample positioning at all photon energies. It also provides significant dynamic compensation for vibrations. Vibrations were present in the old 7.0 STXM and degraded its spatial resolution.

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