

Scanning transmission X-ray microscopy at a bending magnet beamline at the Advanced Light Source

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Abstract. During the last two decades, scanning transmission x-ray microscopy (STXM) has evolved into a powerful characterization tool. For best performance, STXM's are located at undulator sources at synchrotron facilities. The scarcity and expense of undulator sources and associated beamlines limits the number of available STXMs. We have successfully re-examined the use of bending magnets as a source for a STXM and implemented a interferometer controlled STXM with excellent performance at the beamline 5.3.2. at the Advanced Light Source. Near the carbon K-edge, periodic features with 30 nm half-period could be resolved with a zone plate that has a 40 nm outermost zone width with an energy resolution corresponding to 100 meV and an intensity of about 1 MHz. The design and performance of the microscope are described.

1. INTRODUCTION

The utility of Near Edge X-ray Fine Structure (NEXAFS) microscopy is now well established. The number of applications in a wide range of fields is steadily growing and thus there is a pressing need for more instrument access. NEXAFS microscopy can be readily accomplished on surfaces with an X-ray Photoemission Electron Microscope (X-PEEM) illuminated by either bending magnet or undulator beamlines [1,2]. Appropriately prepared bulk samples can be examined in a Scanning Transmission X-ray Microscope (STXM) [3]. The perceived need to operate a STXM on an expensive undulator and its associated beamline might have contributed to the limited number of STXMs that have been successfully operated in the past. In contrast, full field Transmission X-ray Microscopes (TXM) have been operated on (relatively) inexpensive bending magnet beamlines with very short exposure times. However, the presently limited energy tunability, low energy resolving power, and difficulty to operate near the carbon K-edge have resulted in very few NEXAFS microscopy applications with TXMs.

In order to provide much needed STXM capacity, we set out to explore a high brightness bending magnet (5.3.2) at the Advanced Light Source (ALS) as a possible source for a STXM. At the same time, we implemented a major technological advance in the STXM, namely incorporation of a differential laser interferometer between the sample and the microscope optics. This manuscript summarizes material presented in other publications [4,5].

2. THE 5.3.2 STXM: INSTRUMENT DESCRIPTION

The 5.3.2 STXM described here will be used primarily for NEXAFS spectro-microscopy at the C1s, N 1s and O 1s edges. This requires energy scanning and associated zone plate refocusing. We desire that image and spectral data from different photon energies are in registry at the spatial resolution limit of the microscope. A two-dimensional, differential interferometer that actively ensures image registry during refocusing, and also insulates the microscope from low frequency vibrations of system components and longer term drifts, was implemented. The conceptual design of the 5.3.2 STXM is not very different from prior STXM implementations; a stationary zone plate with a central stop and a pinhole define the

optics, the sample is mechanically scanned, and a simple detector measures the transmitted flux. Figure 1 shows 3-D CAD drawings of the major components of the 5.3.2 STXM. Annotated photographs of the zone plate and sample vicinity as implemented are shown in Figure 2.

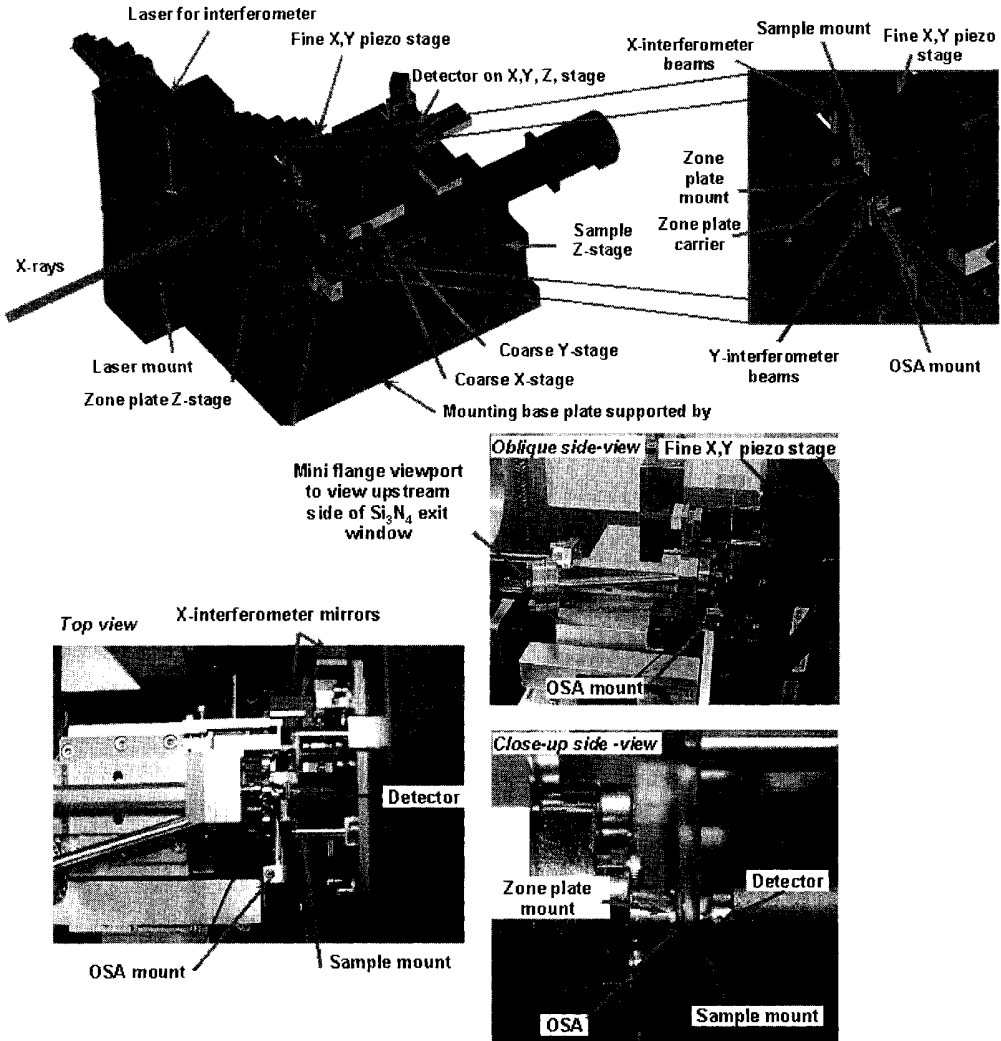


Figure 2. Photographs of 5.3.2. STXM.

Rather than isolate the instrument from floor vibrations, we have built the 5.3.2 STXM on a rigid polymer composite base - referred to as polymer granite or polymer concrete (Zanite®, Precision Polymer Casting) - avoiding resonant amplification at low frequencies. The microscope is built in such a way that only a small fraction of this amplitude (typically 10 nm) shows up as problematic transverse vibration of the zone plate lens relative to the sample. This arrangement on a solid base will maintain the long-term angular alignment required with respect to the optical axis much better than active or passive vibration isolation tables.

The microscope chamber seals to a metal plate embedded into the top of the granite, which does not move or deflect in any appreciable way as the pressure is changed. The chamber can be evacuated to 10^{-6} torr or be filled with helium. Compared to air, the helium environment provides more photons on account of lower absorption, results in smaller interferometer drifts due to its lower index of refraction, and eliminates oxygen from the environment to slow the degradation of polymeric materials during soft x-ray exposure [6]. All components, including piezo stage and stepping motors, are compatible with operation in vacuum.

Direct interferometric, differential measurement of the position of the zone plate with respect to the sample is implemented to achieve linearity, data registry during refocusing, and orthogonality. Two Agilent Technologies 10719A differential interferometers with 10889B PC Servo-Axis Boards are used with the mirrors mounted to the sample and zone plate carriers, respectively (See Figures 1 and 2.). The precision of this combination is 2.5 nm (least significant count). The orthogonality of the scan can be tuned by adjusting the sample mirror mounts. The servo-loop works with a selectable update frequency, up to 20 kHz. This high sampling frequency makes vibration reduction possible. The interferometer/piezo-stage servo loop is opened when the sample is moved with the sample x, y coarse stages. The loop is closed after the move and the stage is servo-ed onto the new interferometer values. In this way an accurate, precise and universal coordinate system is available over tens of millimeters.

The implementation of a laser interferometer also provides for a continuous, absolute coordinate system with a field of view of over 20x20 mm. The operation and selection of the coarse and fine motion are entirely under computer control, transparent to the operator. The large-field absolute coordinate system is also integral to a pre-indexing system based on visible-light microscopy. Areas of interest on a sample can be selected with a high-quality, visible-light microscope and the coordinates are stored under computer control for later use.

The typical scan mode records images one line at a time. The piezo stage is moved with a constant velocity, using buffered acquisition and synchronization of the channel advance with the velocity of the piezo stage set so as to achieve the desired per-pixel dwell. During the scan of each line, the starting point is positioned a sufficient distance before the user defined start-of-line to allow for stage acceleration. The servo-axis controller outputs a signal when the piezo stage is at the desired first point position, thus starting the buffered timed acquisition. Although this provides adequate precision at the current spatial resolution, we are developing an improved scheme that will provide on-the-fly use of the interferometer signal to gate the acquisition at an accurate position at each pixel.

The 5.3.2 STXM is operated through a single computer that controls all instrument and beamline parameters. The user interface provides capabilities to control 12 dimensions of motion of the various microscope stages (ZP, OSA, sample, detector), along with 4 aspects of the monochromator (grating angle, entrance slit, and horizontal and vertical exit slit dimensions), and a fast acting (1 msec open-close time), in-vacuum shutter. The control and acquisition software has a flexible scan engine that allows single and multi-dimensional axis scanning using similar user interfaces. The graphical user interface is structured so that only information necessary to the current task is provided. The user front panel gives equal weight to image and spectral presentation and manipulation. Optimization of the number and contents of separate threads of the software code was critical to achieving high performance.

The 5.3.2 STXM uses phosphors to convert x-rays to visible light. The visible photons are counted by a photo-multiplier (PMT) with count rate capability in the tens of MHz. The front of a lucite pipe is covered by a thin layer of P43 phosphor deposited by sedimentation from a suspension.

3. RESULTS

The laser interferometer allows the direct measurement of the relative vibration of the zone plate and the sample. Ambient vibrations of the zone plate relative to the sample are seen with typical amplitudes of tens of nm. Operated in a closed loop mode, vibrations in both scan directions can be reduced to about 10 nm peak-to-peak. A typical measurement is displayed in Figure 3. This measurement shows that the dominant modes have a few well-defined frequencies. Based on these results, the performance of the microscope depends only on the quality of the zone plate and the illumination conditions utilized. These

measurements show that the 5.3.2 microscope, with closed-loop control, is ready to use zone plates with spatial resolution better than 20 nm.

The 5.3.2. beam line follows design principles developed at beamline X1A at the NSLS [7] and provides very flexible illumination [5]. Due to the large phase space available at a bending magnet, independent control over a relatively large range of the zone plate illumination parameter (p) [7] and resolving power ($E/\Delta E$) has been achieved. For typical operating conditions, the resolving power at the carbon, nitrogen and oxygen edge is in excess of 2000, but can be improved to >5000 [5]. For high spatial resolution imaging, an illumination parameter between $p=0.5$ and $p=1$ is selected, corresponding (at 310 eV) to exit slits of 10 μm and 20 μm , and half Airy disk and full Airy disk illumination criterion, respectively.

Various test patterns fabricated in Au with electron beam lithography techniques and supported on Si₃N₄ membranes have been imaged with a 180 μm diameter zone plate with outermost zone width of 40 nm and a central stop of 80 μm . Results from periodic features with a 1:1 or 1:2 line-to-space ratio are displayed in Figure 4. The contrast for 40 nm and 30 nm features is 24% and 11%, respectively. The microscope is limited neither by mechanical vibrations nor by the use of the laser interferometer. The total overhead time of the image scan is about 1 ms/pixel, similar to the dwell time used for navigation (typically <1 ms/pixel) but small compared to that used for high quality images (typically 3-5 ms/pixel) or spectra (typically 30 ms/point). No astigmatism could be detected. Opening the vertical and horizontal exit slits greatly improves the intensity, at some cost in spatial and spectral resolution. An extreme case is

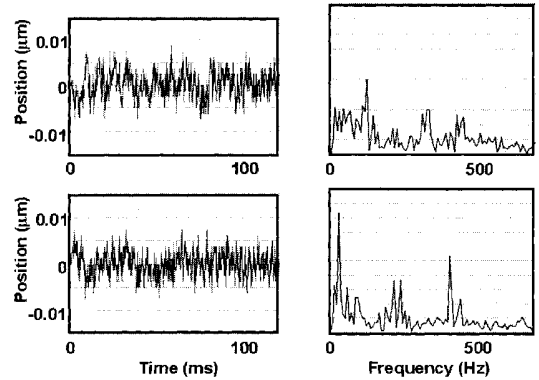


Figure 3. Ambient vibrations (left) and powerspectrum (right) of the relative position of the zoneplate and sample measured with the interferometer. Top: x-axis, bottom: y-axis

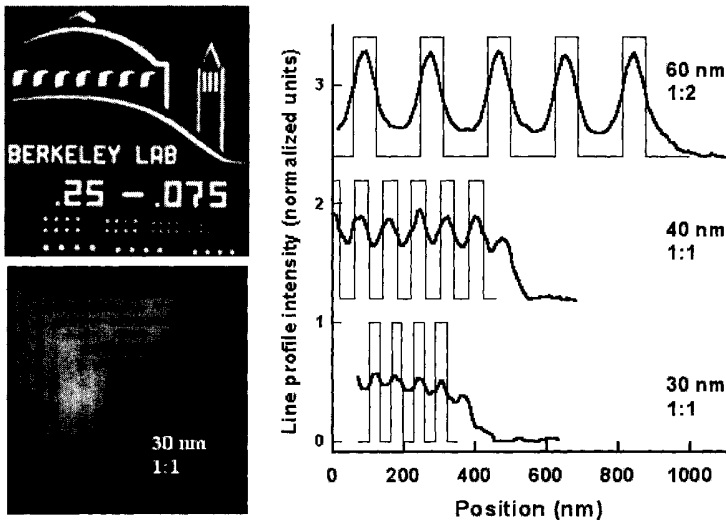


Figure 4. Images of test patterns and their contrast. Top left: Berkeley logo and sequence of dots whose size ranges from 250 nm down to 75 nm. Bottom left: 30 nm angular features with 1:1 mark:space ratio. Right side: line profiles of periodic structures with feature size and mark:space ratio as indicated.

$p=4$ and $E/\Delta E=1500$, for which count rates in excess of 20 MHz could be achieved at the carbon K-edge while still resolving features 120 nm in size.

The interferometer servo-control works in both the x and y direction. Image sequences ('stacks') [8] register, one to another, with very little spatial jitter. In order to demonstrate the precision of the registration, a sequence of images of a high contrast test object (the 10 μm diameter pinhole) was analyzed to extract quantitative shifts. This test 'stack' is a sequence of 31 images taken with varying photon energy from 360 eV to 390 eV, with the ZP translating longitudinally a total distance of 175 μm to stay in focus. A range of 175 μm is sufficient for a

NEXAFS scan at the carbon, nitrogen, and oxygen edge. Figure 5 shows the residual alignment shifts in x and y from image to image of the sequence. These values were derived by tracking shifts in the peak of the 2-d Fourier transform of images. High frequency misregistration is controlled to about 20 nm. Residual, still to be corrected misalignment in the y-direction amounts to less than 40 nm. If the interferometer is properly aligned, it will be possible to acquire images sequences, line scans, and point spectra without degradation in spatial resolution. The longterm stability of this interferometer alignment in response to heavy use by many different researchers is still being assessed.

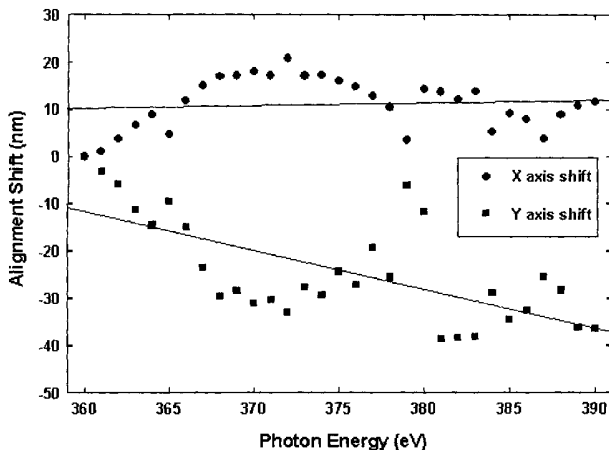


Figure 5. Residual alignment shifts in a servo controlled image sequence. The shifts are extracted from correlation analysis of an image sequence.

4. CONCLUSION

The laser interferometer-controlled instrument described here represents a qualitatively new design that has improved the capabilities of soft x-ray STXM technology, and in particular the facilities at the ALS. The approach presented provides for highly efficient data acquisition and minimizes data analysis and post processing. Avoiding the use of active vibration isolation provides stable long-term positional and angular alignment with the x-ray beam. Small amplitude vibrations of the zone plate with respect to the sample have been achieved. We observe only 10 nm peak-to-peak vibrations when the interferometer servo-loop is active. This shows that the hardware of the 5.3.2 microscope is ready to take advantage of zone plate improvements that are anticipated in the near future.

The successful operation of a STXM at bending magnet beam line 5.3.2 has important implications. The ability to operate a STXM successfully at a bending magnet beam line significantly lowers the overall costs for these instruments. In addition, the 5.3.2 STXM is presently the only STXM with dedicated illumination, available for STXM experiments whenever electrons are stored in the ALS ring.

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