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NANOSURVEYOR 2: A COMPACT INSTRUMENT FOR NANO-PTYCHOGRAPHY AT THE ADVANCED LIGHT SOURCE

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Abstract

The Advanced Light Source has developed a compact tomographic microscope based on soft x-ray ptychography for the study of meso and nanoscale materials [1,2]. The microscope utilizes the sample manipulator mechanism from a commercial TEM coupled with laser interferometric feedback for zone plate positioning and a fast frame rate charge-coupled device detector for soft x-ray diffraction measurements. The microscope has achieved scan rates of greater than 50 Hz, including motor move, data readout and x-ray exposure, with a positioning accuracy of better than 2 nm RMS and has achieved spatial resolution of better than 5 nm. The instrument enables the use of commercially available sample holders compatible with FEI transmission electron microscopes. This allows in-situ measurement of samples using both soft x-rays and electrons.

This instrument is a refinement of a currently commissioned instrument called The Nanosurveyor, which has demonstrated resolution of better than 20nm in both two and three dimensions using 750 eV x-rays. [3] The instrument has been installed on the new Coherent Scattering and Microscopy beamline at the Advanced Light Source and is in the final stages of commissioning. It will enable spectromicroscopy and tomography of materials with wavelength limited spatial resolution.

INTRODUCTION

X-ray ptychography holds the promise of overcoming the resolution, depth of field and efficiency limitations of imaging with conventional x-ray optics [1,2]. It instead relies upon phase retrieval from coherent diffraction data which can be measured to very high numerical aperture with high efficiency. The use of soft x-rays for microscopy, though limiting sample thickness to a few microns, provides very high contrast and exquisite sensitivity to electronic and magnetic states of matter. This, coupled with the high coherent flux available in the soft x-ray range at modern synchrotron sources allows for high spatial resolution with very short exposure times. The ability to image materials with near wavelength limited spatial resolution and functional systems with high time and chemical resolution has previously been demonstrated [2,3]. As x-ray source brightness increases, there is an ever increasing demand for high performance imaging systems. We present the operational instrument of an ultra-stable, high speed scanning x-ray microscope which is fully compatible with many electron microscopy sample holders. The microscope, called Nanosurveyor 2 (Fig. 1), enables wavelength limited x-ray microscopy and facilitate correlative imaging using x-rays and electrons.

MECHANICAL AND OPTICAL DESIGN

The nanosurveyor 2 instrument is derived from the sample area hardware of an FEI CM200 series TEM. We have modified the octagon sample area hardware to create a larger vacuum chamber incorporating both upstream and downstream sections in order to house the required beam conditioning hardware and the LBNL designed fast CCD [4]. This approach was taken in order to leverage the existing design of the TEM for high stability, which also utilizes air-side motor hardware for thermal stability, and to provide for ease of measurement of samples going between EM and XRM instruments. The sample is held and positioned by a standard FEI Compustage sample manipulator that has been upgraded to higher resolution optical encoders. Most functionality present in a standard TEM installation remains in our implementation of the system. This allows users to mount samples in commercially available sample holders designed for tomography, cryo-tomography and those holders specially designed for *in-situ* experiments.

The basic configuration of the system is similar to other scanning zone plate systems in use at the ALS in that the focusing optic is mounted into a high stiffness scanning piezo flexure stage with X and Y axes (orthogonal to the x-ray beam direction) [5]. The interferometer's fiber-optics heads are mounted to the fixed assembly which supports the sample goniometer such that mechanical path lengths to the sample are minimized. These interferometer beams are returned by a polished and gold coated surface on the zone plate mounting assembly in order to track position of the zone plate in XY while scanning. A compact, three axis order sorting aperture assembly is mounted within the frame of the piezo scanner in order to facilitate tracking of the focused x-ray beam with the order sorting aperture (OSA). The OSA assembly scans with the focusing optic. This enables us to utilize the entire 100 μm range of our XY scanning system rather than being limited to a fraction of the OSA diameter (typically 50 μm).

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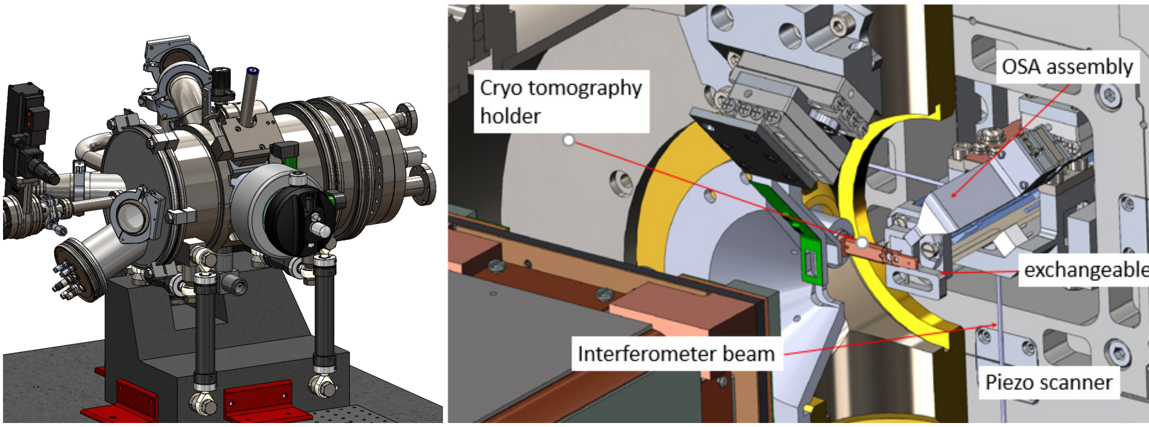

Figure 1: CAD models of the Nanosurveyor 2 instrument showing Gatan cryo tomography sample holder and internal zone plate optic scanning hardware with reference to TEM sample holder.

The focusing optic travels along the beam axis on a novel stage that integrates the motion stage with the interferometric mirror surfaces and the mounting system for the zone plate optic. This system is mounted within the open frame of the XY piezo scanner. The design of this linear travel stage is centered around an octagonal piece of sapphire that has been polished and had designated reflecting surfaces coated for use as mirrors for the interferometric tracking system. Other faces act as the bearing surfaces for stick/slip piezo actuators that provide the motive force to translate the optical elements along the beampath. This assembly is both lightweight and very stiff, leading to a high resonant frequency in the scanning system. The design specification for the system was to allow for positioning the beam on the sample at a rate matching the maximum frame rate of the fast CCD.

VIBRATION AND THERMAL STABILITY

Vibration control and stability of the entire endstation assembly is critical to reaching the ultimate planned system resolution of 1nm. To this end, the exit slit assembly is incorporated into the structure of the endstation support in order to remove effects of experimental hall floor motion. The slit assembly is installed on a cantilever section of tubular steel that is mounted to the endstation support structure (Fig. 2). Both the endstation and exit slit frames are filled with epoxy concrete (Zanite) for vibration damping and increased thermal stability. The cantilever was designed to maximize horizontal stability of the exit slits with respect to the focusing optics in the endstations. The two endstations that comprise the COSMIC Imaging station are mounted on a single 1500kg casting of Zanite. This casting is isolated from the support frame by an array of engineered viscoelastic urethane polymer isolation pads (Sorbothane) which provide passive vibration isolation. This has resulted in motion transmissibility of 19 Hz in the vertical (Y) and 5 Hz in the lateral directions (X,Z) (Fig. 3). Damping of motion is aided by the center of gravity of the experimental system being located 50 mm below the top surface of the Zanite filled steel assembly.

The endstation assembly is contained within an acoustically dampening enclosure which is thermally stabilized. The temperature of the enclosure is maintained by a recirculating, closed-loop HVAC system which has been demonstrated to maintain temperature within 0.1 degrees Centigrade over long periods of time (Fig. 4).

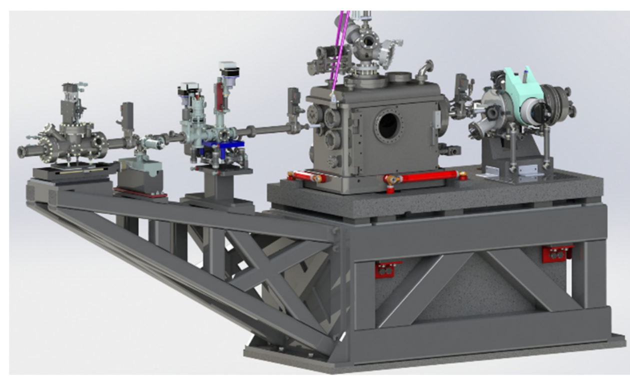

Figure 2 : COSMIC Imaging endstation with Nanosurveyor 1 and 2 instruments mounted in-line.

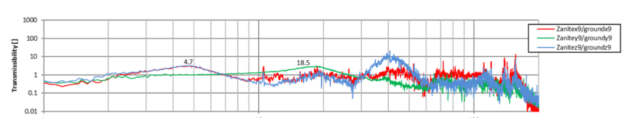

Figure 3: Vibrometer measurements showing the spectra of isolation in X,Y,Z between the experimental floor and the top of the passively isolated instrument structure.

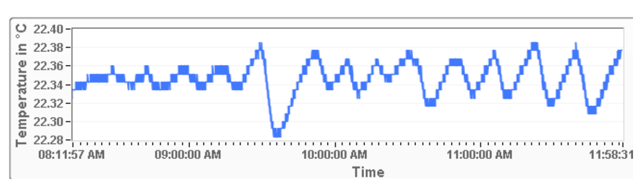

Figure 4: Plot showing typical stability of temperature in the environmental enclosure over a period of 4 hours.

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COMMISSIONING RESULTS

First ptychography results from Nanosurveyor 2 at the COSMIC beamline during commissioning. The sample, provided by a Beam Technologies, is a WSi2/Si multilayer, with fundamental linewidth of 2.8nm (all other lines are integer multiples), and thickness of 100nm. Data was collected at beam energy of 1000eV using a 45nm zone plate supplied by the Center for X-Ray Optics (CXRO).

The zone plate was scanned with a step size of 30nm and the sample was exposed for 20msec at each point. The CCD was placed further downstream than the standard operating position to increase dynamic range for commissioning, thus decreasing the Numerical Aperture and limiting the resolution to 10nm (Fig. 5).

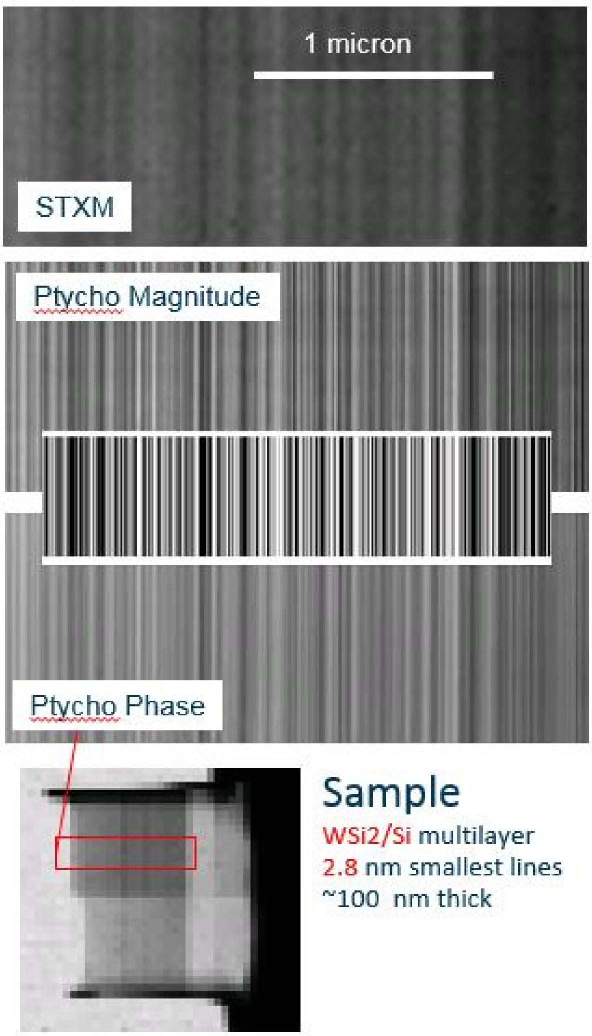

Figure 5: First ptychography results from COSMIC Imaging endstation at Advanced Light Source.

CONCLUSION

We present the design and preliminary performance results of a new compact scanning transmission x-ray microscope dedicated to ptychographic imaging at the ALS. The microscope is based upon the sample goniometer and transfer system of a commercial TEM which has been customized to also house a high performance zone plate scanning mechanism. This mechanism can perform point to point scans at well over 100 Hz which is fast enough to keep pace with the short exposure times possible at new high brightness beamlines and the short readout time of a custom fast frame rate CCD. We have also demonstrated that the stability of the instrument is suitable to reach the microscope design goal of soft x-ray wavelength limited imaging.

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REFERENCES

[1] P. Thibault, M. Dierolf, A. Menzel, O. Bunk, C. David, F. Pfeiffer, "High-Resolution Scanning X-ray Diffration Microscopy", in *Science*, 321, 2008, pp 379–382

[2] D. A. Shapiro, Y. S. Yu, T. Tyliszczak, J. Cabana, R. Celestre, W. Chao, K. Kaznatcheev, A. L. D. Kilcoyne, F. Maia, S. Marchesini, Y. S. Meng, T. Warwick, L. L. Yang, H. A. Padmore, "Chemical composition mapping with nanometre resolution by soft X-ray microscopy", in *Nature Photonics* 8, 2014, pp 765–769, ISSN 1749-4885, http://www.nature.com/nphoton/journal/v8/n10/full/nphoton.2014.207.html

[3] P. Denes, D. Doering, H. A. Padmore, J. P. Walder, J. Weizeorick, "A fast, direct x-ray detection charge-coupled device", in *Review of Scientific Instruments* 80, 083302, 2009, pp 1089-7623, ISSN 0034-6748 http://scitation.aip.org/content/aip/journal/rsi/80/8/10.1063/1.3187222

[4] 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, H. Ade, "Interferometer-controlled scanning transmission X-ray microscopes at the Advanced Light Source", in *Journal of Synchrotron Radiation* 10, 2003, 125–136, http://dx.doi.org/10.1107/S0909049502017739

[5] J. Lim, Y. Li, D. H. Alsem, H. So, S. C. Lee, P. Bai, D. A. Cogswell, X. Liu, N. Jin, Ys. Yu, N. J. Salmon, D. A. Shapiro, M. Z. Bazant, T. Tyliszczak, W. C. Chueh, "Origin and hysteresis of lithium compositional spatiodynamics within battery primary particles", in *Science* 353, 2004, pp 566–571, http://science.sciencemag.org/content/353/6299/566