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Nanosurveyor 2: A Compact Instrument for Nano-Tomography at the Advanced Light Source

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NANOSURVEYOR 2: A COMPACT INSTRUMENT FOR NANO-TOMOGRAPHY 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 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 point to point (25 nm steps) scan rates of greater than 120 Hz with a positioning accuracy of better than 1 nm RMS. The instrument will enable the use of commercially available sample holders compatible with FEI transmission electron microscopes thus also allowing *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 10 nm in two dimensions using 750 eV x-rays. Once moved to the new Coherent Scattering and Microscopy beamline it will enable spectromicroscopy and tomography of nano-materials with wavelength limited spatial resolution.

1. 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 a prototype design for an ultra-stable, high speed scanning x-ray microscope which is fully compatible with many electron microscopy sample holders. The microscope, called Nanosurveyor 2, will enable wavelength limited x-ray microscopy and facilitate correlative imaging using x-rays and electrons.

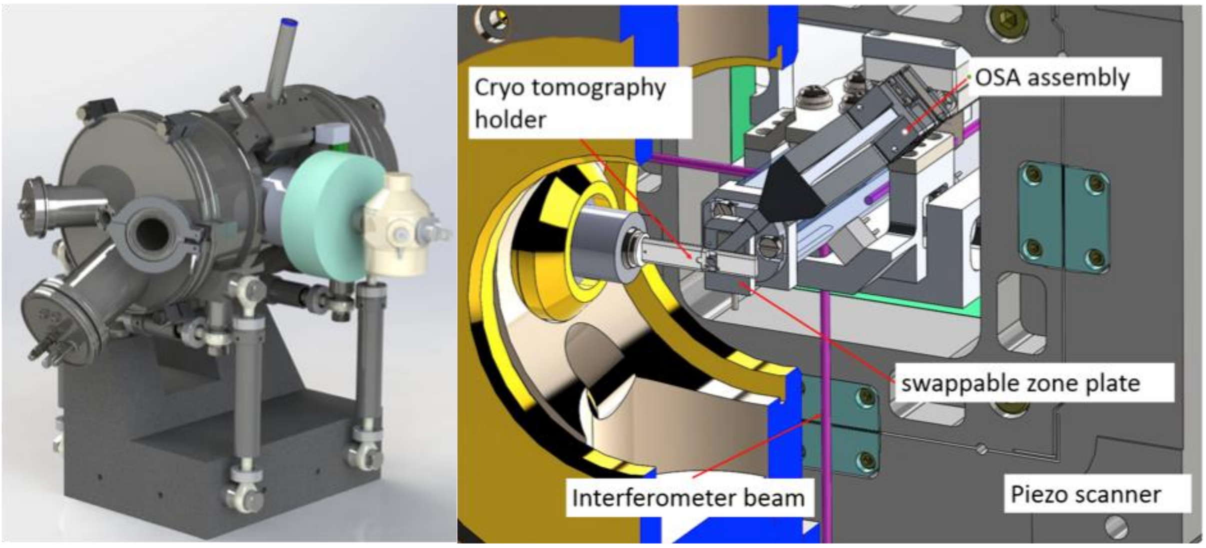

Figure 1 CAD models of the Nanosurveyor 2 instrument showing Gatan cryo tomography sample holder and internal zone plate optic scanning hardware with reference to TEM sample holder.

2. Mechanical and Optical Design

The prototype nanosurveyor 2 instrument is based on the sample area hardware of an FEI CM200 series TEM. We have modified the octagon sample area hardware removed from an FEI TEM 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 which 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 rigidly 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). This enables us to utilize the entire 120 μm range of our XY scanning system rather than being limited to a fraction of the OSA diameter (typically 50 μm).

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 which 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 which 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

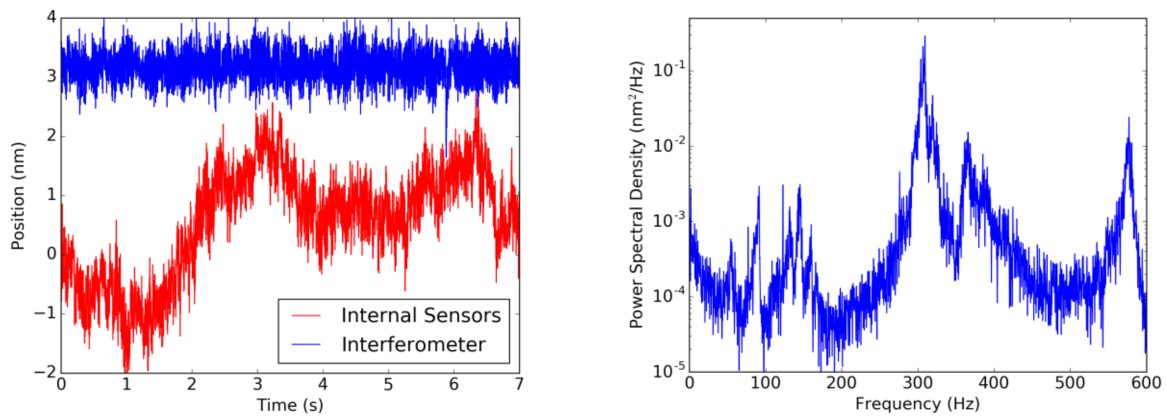

Figure 2 Left: position of the zone plate as measured by the interferometer when the zone plate is in closed loop control of the internal capacitive sensors (red line) and the interferometer (blue line). The position is measured at 100 kHz but the data is displayed at 1 kHz. Right: power spectrum of the zone plate vibrations when in closed loop control of the interferometer. The first resonance is at 300 Hz and over the full 7 second measurement has an RMS of 1 nm.

(200 frames per second) which resides in a light tight vacuum section immediately downstream of the sample.

3. Stability Analysis

Two mechanisms are available for closing the loop in the zone-plate XY stage: feedback from internal capacitive sensors of the stage, or an external interferometer measurement. Although the interferometer heads have the advantage of directly measuring the position of the final moving part (as opposed to the capacitive sensors, which measure the motor movement,) their accuracy depends on the stability/stiffness of the structure in between the optical head mounts and the moving stage. Figure 2 shows interferometer measurements of the zone plate when under closed loop control. The capacitive sensors stabilize the stage to high precision but there is still some residual relative motion of the zone plate at the few nanometer scale. The first resonant frequency for these vibrations is at 300 Hz and over the full 7 second measurement has a Root Mean Square (RMS) amplitude of 1 nm.

The interferometer can output position information either in incremental form (quadrature) or absolute (High Speed Serial Link - HSSL). While quadrature benefits from faster update rates, limited by a 40ns clock, it has the disadvantage of suffering from a tradeoff between communicated precision and maximum speed with which the stage can move. This means that when the feedback parameters and the stage tuning is configured for optimal scanning performance, the quadrature precision cannot be set below 1 nm. This is especially critical for a new beamline called COSMIC at the ALS, dedicated to coherent imaging and scattering. Due to the high coherent flux, motor moves will be the speed bottleneck. An alternative is to use HSSL, which is immune to lossy communication, and has virtually no limit on communicated precision. The downside of HSSL is a longer clock cycle of 80 ns, and the need for 48 cycles to communicate a single position. This still amounts to only a few milliseconds per position update, which is sufficient, given motor moves take milliseconds.

4. Conclusions

We present the conceptual design and preliminary performance analysis 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] Thibault P, Dierolf M, Menzel A, Bunk O, David C and Pfeiffer F 2008 Science 321 379–382
[2] Shapiro D A, Yu Y S, Tyliszczak T, Cabana J, Celestre R, Chao W, Kaznatcheev K, Kilcoyne A L D, Maia F, Marchesini S, Meng Y S, Warwick T, Yang L L and Padmore H A 2014 Nature Photonics 8 765–769 ISSN 1749-4885 URL http://www.nature.com/nphoton/journal/v8/n10/full/nphoton.2014.207.html
[3] Denes P, Doering D, Padmore H A, Walder J P and Weizeorick J 2009 Re- view of Scientific Instruments 80 083302 ISSN 0034-6748, 1089-7623 URL http://scitation.aip.org/content/aip/journal/rsi/80/8/10.1063/1.3187222
[4] Kilcoyne A L D, Tyliszczak T, Steele W F, Fakra S, Hitchcock P, Franck K, Anderson E, Harteneck B, Rightor E G, Mitchell G E, Hitchcock A P, Yang L, Warwick T and Ade H 2003 Journal of Synchrotron Radiation 10 125–136 URL http://dx.doi.org/10.1107/S0909049502017739
[5] Lim J, Li Y, Alsem D H, So H, Lee S C, Bai P, Cogswell D A, Liu X, Jin N, Yu Ys, Salmon N J, Shapiro D A, Bazant M Z, Tyliszczak T and Chueh W C 2016 Science 353 566–571 ISSN 0036-8075, 1095-9203 URL http://science.sciencemag.org/content/353/6299/566