Recent advances in nano-scale spatial resolution x-ray microscopy instrumentation at NSLS-II

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Recent advances in nano-scale spatial resolution x-ray microscopy instrumentation at NSLS-II

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Abstract. X-ray microscopy is an invaluable and powerful characterization tool applied in many scientific fields, such as materials science, biology, environmental science, and energy research. In recent years it has been driven by rapid developments of novel technologies and systems resulting in imaging experiments elucidating structural inhomogeneities and chemical reactions at the nanometer scale. To obtain high spatial resolution comprehensive chemical and structural information, an X-ray microscope must be equipped with adequate capabilities and allow for simultaneous acquisition of multiple datasets. In recent years, a number of X-ray microscopes have been designed, constructed, and commissioned at NSLS-II. Here we provide an overview of the microscopy instrumentation development program at NSLS-II and specifically focus on the multilayer Laue lens–based hard X-ray nanoprobe optimized for ~10 nm spatial resolution imaging, its current status, and future upgrades along with recently constructed Kirkpatrick-Baez based scanning microscope designed for ~100 nm spatial resolution experiments.

INTRODUCTION

The rapid emergence of ultra-brilliant synchrotron facilities and recent advances in the fabrication of efficient nano-focusing optics stimulated the development and applications of focused beams to study various phenomena in science, engineering, and technology. [1-5] When the focal spot of an X-ray beam is reduced to 100 nm and below, there is a need to develop a dedicated instrument suitable for a given application. This involves the thorough design, characterization, and performance evaluations in both lab- and synchrotron environments prior to science commissioning and user operation. A number of scanning and full-field microscopes have been designed, constructed, and commissioned within the Imaging and Microscopy program at the National Synchrotron Light Source II during the last five years. [6-8] All of these systems were developed in-house through a cohesive effort of the beamlines staff, engineering group and the nano-positioning team aimed to assist in the design and construction of the cutting-edge microscopy instrumentation. The developed systems utilize the most recent advances in nano-positioning solutions and associated controls, with some of the approaches being adopted beyond the Imaging and Microscopy program at NSLS-II [9]. A few examples presented below reflect on some of the microscopy systems being developed at NSLS-II in recent years.

HARD X-RAY MICROSCOPY AT THE NANOPROBE BEAMLINE

Hard X-ray nanoprobe beamline (HXN) at NSLS-II is a multimodal microscopy tool used to provide hard X-ray imaging capabilities with spatial resolution approaching 10 nm. [10] It allows simultaneous acquisition of the fluorescence, differential phase contrast, and ptychography datasets in both 2D and 3D. HXN microscope is a unique apparatus that utilizes multilayer Laue lenses (MLL) for nano-focusing and is offered to the general user community. Prior to the construction and commissioning of the HXN endstation, extensive instrumentation R&D work has been carried out [11-17], and it incorporated concepts and approaches used in the scanning probe microscopy community. [18] At present, complimentary to MLLs used for nano-focusing above 12 keV photon energy, HXN microscope is also equipped with the Zone Plate (ZP) optic for imaging experiments at 6-18 keV and spatial resolution above 30 nm. MLLs were introduced as an efficient alternative to performing nano-focusing in the hard X-ray regime [19] and so far have demonstrated 2D resolution of 8.4x6.8 nm^2. [20] An individual MLL optic can be considered as a liner

ZP, which requires angular adjustment to satisfy Bragg condition and provide line focusing. The two independent MLLs must be positioned orthogonally with respect to each other in order to achieve point focus. In total, eight degrees of motion (five translations and three rotations) are required to perform the full alignment. The typical focal distance of an MLL optic with a 10 nm focus and an aperture of 50 μm does not exceed 5 mm at 12 keV. The resulting working distance between the sample and the Order Sorting Aperture (OSA) is no greater than 1 mm. For a 10 nm point focus, angular misalignment between two lenses must be less than 0.01°, and the accuracy of placing both MLLs along the X-ray beam direction must be better than a depth of focus ~ 3 μm. [21] Such stringent technical requirements call for novel solutions to enable user-available point focusing below 10 nm.

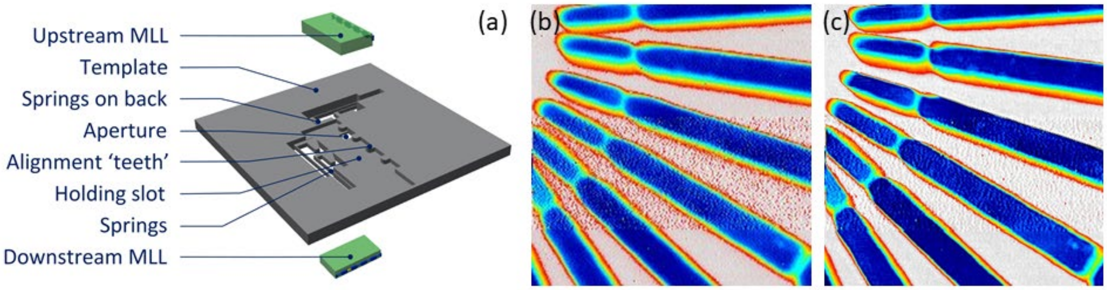

FIGURE 1. A monolithic MEMS-based 2D MLLs nanofocusing optic. **a)** an overview of a template with all key features being labeled. **b)** Scanning Transmission x-ray Microscopy (STXM) image formed by plotting the total transmission as a function of sample position. **c)** ptychography image for the STXM measurement (amplitude). Smallest features of 20 nm (gaps between inner and outer spokes) can be resolved. Scan parameters: 2 μm × 2 μm scan area with a step size of 10 nm and a dwell time of 0.1s, photon energy 13.6 keV.

The development of integrated 2D MLL nanofocusing optics could greatly reduce the complexity of an instrument and minimize the degrees of nano-scale motion required for MLL alignment. There has been a number of attempts in the past few years targeting the development of integrated 2D MLL optics. [22-25] Typically, FIB welding or UV-adhesives have been used for direct bonding. However, UV-adhesives may cause uncontrollable stress and potential contamination of the MLL optics itself resulting in worse-than-expected focusing performance. It is especially true in the case of wedged MLLs, when parameters of individual lenses are tailored to specific photon energy and astigmatism could not be compensated by varying x-ray energy without compromising efficiency. In order to overcome the limitations of the direct bonding method, we have developed a more flexible way to combine two MLLs together using technologies applied in the fabrication of microelectromechanical systems (MEMS). The MEMS template developed for direct MLLs bonding includes a silicon holder with pre-aligned slots tailored to individual MLLs, and a set of silicon springs to secure MLLs at correct locations, see Fig. 1a. Microfabrication is performed on both sides of a thick silicon chip. The structures on both sides of a silicon chip were patterned orthogonally with respect to each other through a backside aligning lithography process. Both MLL optics are secured on both sides of a chip by the silicon springs. The overall dimensions of a device are ~10 × 10 mm^2. Fig. 1b and 1c demonstrate preliminary results obtained during nano-focusing experiments at HXN, astigmatism-free point focus below 15 nm in both directions has been demonstrated, it is close to the expected focusing performance of the used MLL lenses. [26,27] We believe, 2D integrated MLL optic is a path forward which enables broader applications of MLLs in the X-ray microscopy community.

KB-BASED SUB-100 NANOMETER SCANNING MICROSCOPE FOR SUB-MICRON RESOLUTION SPECTROSCOPY BEAMLINE

The Submicron Resolution X-ray Spectroscopy beamline (SRX) has been designed and constructed to enable x-ray fluorescence microscopy studies with sub-micrometer spatial resolution in the photon energy range between 4.5 and

20 keV. The spatially resolved X-ray Absorption Near Edge Structure (XANES) spectroscopy also can be performed in both fluorescence and transmission modes. At present, a new nano-focusing endstation has been constructed and installed at the beamline. It is equipped with a pair of KB focusing mirrors and a sample stage capable of providing raster scanning in both 2D and 3D, shown in Fig. 2 (left panel). A set of fixed curve elliptical KB optics has been installed in a separate vacuum enclosure. For the optics, the tangential slope errors were determined to be less than 60 nrad RMS and the shape errors < 0.8 nm RMS. The optics system utilizes piezo-driven flexures for enhanced stability and resolution. Fig. 2 (right panel) demonstrates 25 nanoradian angular steps performed by the vertical focusing mirror actuators. There are two options for the feedback signal used by the KB motors. First, the built-in motor encoders can be used for initial alignment, and when in position global interferometric feedback can be turned on to ensure long-term stability of the system. The sample stage is equipped with a slip ring and enables infinite rotation. Moreover, line-focusing interferometry has been used to track the position of a sample with respect to the global invar reference frame. At present, the KB-microscope is undergoing commissioning of its technical capabilities, and is available for general users.

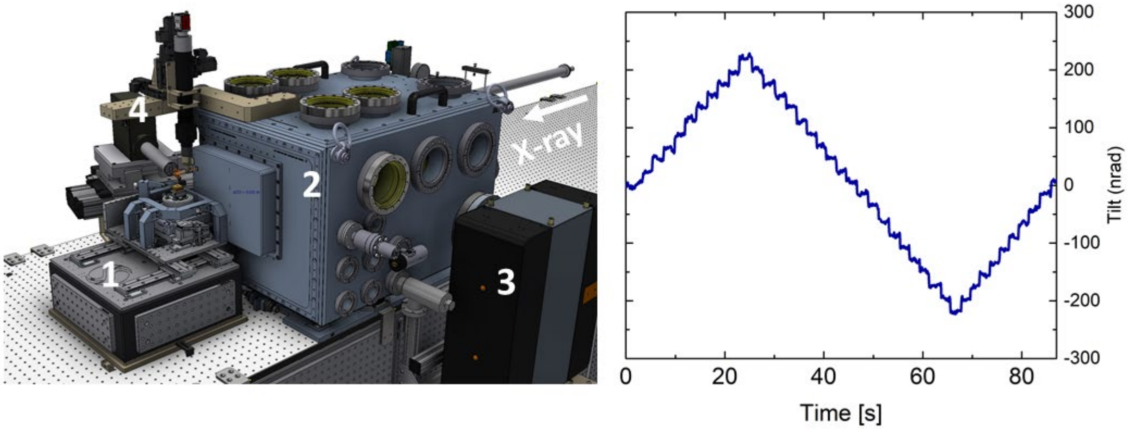

FIGURE 2. Left panel: CAD rendering of a KB microscope. 1 – sample stage, 2 – KB manipulation system and vacuum enclosure, 3 – pumping system, 4 – fluorescent detector and visible light microscope assembly. Right panel: 25 nanoradian angular steps performed by a vertical focusing mirror and independently verified through interferometric measurements.

CONCLUSIONS

In summary, we have illustrated recent X-ray microscopy instrumentation developments that took place at NSLS-II during the past five years. In the future, a much stronger synergy between instrumentation and methods available in the X-ray, electron, and scanning probe microscopy communities is envisioned in order to strengthen and benefit each of these scientific fields.

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