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Development and characterization of monolithic Multilayer Laue Lens nanofocusing optics

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Abstract

We have developed an experimental approach to bond two independent linear Multilayer Laue Lenses (MLL) together. A monolithic MLL structure was characterized using ptychography at 12 keV photon energy, and we demonstrated 12 nm and 24 nm focusing in horizontal and vertical directions respectively. Fabrication of 2D MLL optics allows installation of these focusing elements in more conventional microscopes suitable for x-ray imaging using zone plates (ZP), and opens easier access to 2D imaging with high spatial resolution in the hard x-ray regime.

X-ray microscopy is a powerful characterization tool routinely applied to address emerging questions in many fields of science and technology. [1–3] In most cases, imaging resolution is determined by the size of the focused beam delivered by an x-ray microscope. [4] Different types of x-ray focusing optics can be used to achieve nanometer scale spot size. [5–10] Multi-layer Laue Lenses (MLL) were proposed as an alternative approach to achieve nanofocusing and overcome the well-known ‘aspect ratio problem’ of the zone plates for the hard x-ray regime. [11] Recently, 11 nm focusing in one dimension using tilted MLL and 8 nm focusing utilizing wedged MLL have been reported.[12, 13] MLLs are diffractive optics comprised of layers of alternating materials with different refractive indices. An individual MLL can be considered as a special 1D zone plate with an additional requirement to satisfy a Bragg condition in order to achieve optimal performance. A pair of MLLs needs to be aligned orthogonally with respect to each other to achieve point focusing. This requirement poses technical challenges on a microscope capable of accommodating and aligning of two 1D optics. A number of MLL-based microscopes have been built and commissioned in the recent years. [14–17] A total of eight degrees of rotational and translational motions are required to perform alignment of two crossed MLL optics and satisfy Bragg conditions to achieve point focusing (see Fig. 1a). A typical working distance for imaging with 10 nm spatial resolution at 12 keV photon energy does not exceed one millimeter between Order Sorting Aperture (OSA) and the focal plane. [18] The distances between MLLs and an OSA are less than a few millimeters. In practice, this requires an extremely complex and requires a stable instrument capable of delivering over twenty degrees of motion in a very small envelope, as is the case for the MLL microscope developed for the Hard X-ray Nanoprobe Beamline at the NSLS-II. [16] Moreover, the positions of both MLL optics and the sample have to remain constant (nm-level accuracy) throughout the experiment to maintain a stable image. A pair of MLLs bonded monolithically can greatly reduce the instrumental complexity. In the past few years, there has been an increasing effort to develop 2D MLL optics through bonding [19] and the use of bonded structures for full field x-ray imaging. [20]. During these experiments sub-100 nm resolution imaging has been demonstrated. In this work, we present an experimental method specifically developed to bond two individual 1D MLL optics together, control the gap between two lenses, and demonstrate 12 nm and 24 nm focusing in horizontal and vertical directions respectively.

When aligning two linear MLL optics, the challenging tasks are to maintain orthogonality (X in Fig 1a) and to control the separation distance between the two lenses since they require two independent stacks of stages as part of the x-ray microscope. [16] A loss of orthogonality introduces additional undesirable cross phase term in the wavefront which causes blurring effects on the focus. A criterion for adequate orthogonality has been developed, $\gamma < \lambda f / 2D^2$, where λ is the wavelength, f is the focal distance and D is the aperture of the lens. [21] To achieve 10 nm point focus, the misalignment γ should remain below 0.01° for a typical MLL with a focal length of 4 mm at 12 keV photon energy and aperture size D of 40 μm . Angular alignment must be maintained throughout the experiment. The misalignment tolerance to the latter is determined by the depth of focus at a given operating energy. For the very same lens the error in separation distance should be no larger than 4 μm . [21] During bonding process, we optimized X alignment. The remaining two angles to satisfy the Bragg condition, Φ and Θ , can be adjusted using conventional tip-tilt stage when performing final adjustments in the x-ray microscope. The fact that X angle is well defined allows installation of bonded MLL optics into a much simpler x-ray microscope, and makes it compatible with conventional ZP-based systems equipped with tip-tilt angular adjustment of the nanofocusing optics.

MLL optics were fabricated via magnetron sputtering deposition and sectioned by reactive ion etching followed by focused ion beam (FIB) milling to refine the surface. [22] Non-wedged MLLs having an aperture of 43 μm , comprised of 6510-layers with layer thicknesses varying between 4 and 20.8 nm were bonded. The overall linear dimensions of each individual lens, including substrate, equals $\sim 3\text{x}2\text{x}0.5\text{ mm}^3$. The major challenge in preserving X alignment during bonding process pertains to a small size of the lens itself. For a given length in X or Y of the MLL optic, 0.01° of angular tolerance X translates into 175 nm height variation for every 1 mm of optic length. This is beyond resolution of any optical microscope, therefore we employed the Zygo NewView 6000 3D optical surface profiler to characterize and correct misalignment errors during the bonding process. The profiler is capable of resolving 1 nm height steps which correspond to 0.00006° angular error per millimeter length. Fig. 1b - 1c show the CAD model and a photograph of the developed MLL bonding system. Component 'A' in Fig. 1b has three linear and two angular degrees of motion and accommodates one MLL optic. Component 'B' in Fig. 1b provides two angular motions for another MLL. The entire setup resides on top of an X - Y slider and allows positioning of MLLs under

FIG. 1. a) Schematic of the MLL microscope, all degrees of motion required to perform full MLL alignment are indicated. b) Computer Aided Design (CAD) model of the alignment system developed for MLL bonding. 'A' represents manipulation system for vertical MLL and 'B' for horizontal MLL respectively. 'C' schematically shows the head of a white light interferometer. The inset shows two bonded lenses with an overlapping MLLs region marked with a circle. Note, there is a redundant Φ motion in parts 'A' and 'B' used to optimize the view angle when aligning with respect to the interferometer head c) photograph of the actual bonding setup.

the profiler head 'C' during bonding process. The inset in Fig. 1b shows schematic of two bonded MLLs with an overlapping area marked with a circle.

FIG. 2. a) optical microscope image of two bonded MLL's. The overlapping area is marked with a circle. b) SEM image of two bonded lenses, c) optical surface profiler image of two bonded MLL's, the height difference between planes of two lenses equals $221 \mu\text{m}$.

The bonding process was performed in a few steps. First, one MLL optic was temporarily adhered to one side of a reference object mounted on part 'B' in Fig. 1b. Our reference is a precisely manufactured, optical grade structure which has two surfaces orthogonal with respect to each other; orthogonality error is less than $\pm 0.004^\circ$. During this step, top surface

(side opposite to the substrate) of the MLL optic was roughly aligned with respect to one surface of the reference. During the second step, profiles of both MLL and reference were mapped using profiler, the misalignment error between those two was recorded. Next, the second MLL optic was brought close to the reference/MLL assembly utilizing manipulator 'A' shown in Fig. 1b. The orientation of the second MLL with respect to the reference was measured by profiler and adjusted taking into account the misalignment error of the first MLL with respect to the reference. All linear and angular degrees of motion of part 'A' (see Fig. 1b)) were used to adjust the planes of the MLLs to be parallel along Z while preserving orthogonality of the normals to the planes. When fully aligned, low thermal expansion UV curing adhesive was applied to hold both lenses together. We measured orthogonality between two lenses right after bonding and the following day. It remained within $\pm 0.001^\circ$ from its original value indicating no time-dependent drifts.

Figure 2 demonstrates the optical microscope image of two bonded lenses (panel a), SEM image of crossed MLLs (panel b) and surface profiles of two lenses measured by the white light interferometer (panel c). Planes of two lenses were measured to be separated by $221\ \mu\text{m}$ which consists of thickness of the UV glue between the two lenses and the recess of the FIB'd lens with respect to the Si substrate.

In order to verify focusing performance of the bonded MLLs we mounted monolithic lenses in the microscope specifically developed for optics characterization. [23] Ptychography measurements have been taken at 3-ID beamline of the NSLS-II. Bonded MLL structures were angularly aligned to deliver an optimized focus at 12 keV photon energy. A star test pattern fabricated with $1\ \mu\text{m}$ thick gold was scanned laterally across the beam at a defocal plane $\sim 70\ \mu\text{m}$ away from the horizontal focal plane. The scan trajectory followed a Fermat spiral pattern [24] with an incremental step size of 100 nm and covered a $4\ \mu\text{m} \times 4\ \mu\text{m}$ region with 512 points. The far-field diffraction signal was recorded using a Timepix pixel-area detector placed 0.7 m downstream from the sample. The Bragg angle has been optimized to provide maximum diffraction intensity and cover the entire numerical aperture. Diffraction intensity peak was located approximately $15\ \mu\text{m}$ away from the outermost zone. 100-iteration of difference map algorithm [25] was used to reconstruct transmission function of the test pattern and the wavefront of the focused beam. The horizontal and vertical focal planes were located by numerically propagating the obtained wavefront, as shown in Fig. 3. The field-of-view of the reconstructed probe ($\sim 1.2\ \mu\text{m}$) is determined by the experimental setup.

FIG. 3. Wavefront reconstructions at a) - b) horizontal and d) - e) vertical focal planes. Horizontal and vertical planes are separated by $221 \mu\text{m}$ and yield the focusing of 12 nm and 24 nm respectively. c) and f) show the reconstructed probe and object at the measurement plane with brightness being the amplitude and color being the phase respectively

During the numerical propagation process the wavefront diverges significantly with its dimensions exceeding the field of view, it causes aliasing effect and introduces artificial fringes in the propagated wavefront at the vertical focal plane, as shown in Fig. 3(d). To mitigate the aliasing effect, the propagated wavefronts were integrated along both horizontal and vertical directions in order to generate line profiles and estimate focusing. The focal sizes were determined to be 12 nm and 24 nm in horizontal and vertical directions, respectively. The horizontal focusing results are consistent with our previous measurements [12]. Vertical focusing is believed to be affected by the bonding procedure, i.e. excessive amount of UV adhesive partially blocking the lens aperture and therefore worsening focusing performance by approximately a factor of two. The propagation was conducted with $1 \mu\text{m}$ step size, and revealed the separation between two focal planes is $\sim 221 \mu\text{m}$, which very well agrees with profilometry measurements. Precise control of the distance between two linear MLL optics during bonding process allows to fabricate lenses with different focal distances, therefore

both horizontal and vertical focal planes can be overlayed yielding a possibility to perform direct imaging along with ptychography.

In summary, we have developed a reliable method to bond two linear MLL optics together. By performing ptychography measurements we demonstrated focusing of 12 nm and 24 nm in horizontal and vertical directions respectively. Although point focusing in the common focal plane was not demonstrated, by bonding lenses with different focal lengths and choosing photon energy matching the gap, astigmatism can be eliminated. Development of monolithic MLL optics allows to implement such optics into microscopy systems suitable for operation using zone plates, and perform multimodality imaging with high spatial resolution and focusing efficiency in the hard x-ray regime.

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