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Fabrication of customizable wedged multilayer Laue lenses by adding a stress layer

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ABSTRACT

Diffractive optics for hard X-rays feature superior properties in terms of resolution and efficiency, if volume diffraction effects are exploited all-over the aperture. For multilayer Laue lenses, preferably a wedged geometry is required to obtain this effect. We present an approach utilizing an additional stress layer to realize the necessary geometrical modifications where each lens can be customized to a selected photon energy independently of the given multilayer deposition. The quality of the deposition of the stress layer is evaluated using a laboratory X-ray microscope prior to its application at synchrotron radiation facilities with a special approach to measure the relative layer tilt at high spatial resolution.

1. Introduction

A large number of recent synchrotron radiation facilities is equipped with nanoprobe beamlines where various types of X-ray optics are being installed and developed to perform and to enhance X-ray nanoanalysis. Resolution and efficiency of the optics have direct impact to beam size and required time for data acquisition, and therefore, on the feasibility of the experiment itself. Diffractive optics consist of a set of alternating zones obeying the zone plate law. Volume diffraction has to be considered to obtain optimal performance [1]. The shape of the zones has to follow an elliptical path along the optical axis, and the ideal thickness in beam direction t is given by the Pendellösung of the dynamic theory of diffraction. It can be calculated for each wavelength λ if the susceptibility χ is known for both materials A and B:

$$t = \frac{\pi\lambda}{2|\chi_A - \chi_B|}.$$

Fig. 1 shows those thicknesses for the WSi_2/Si system. The desired values exceed the capabilities of Fresnel zone plate fabrication by electron beam lithography and subsequent etching in particular, if zone widths in the single nanometer range have to be fabricated to provide sub-10 nm resolution.

Multilayer Laue lenses (MLL) are a promising approach to circumvent these limitations [2]. The individual zones are fabricated by thin film deposition methods on planar or cylindrical substrates. The actual lens element is subsequently cut out of the coating. In the case of a planar MLL, a line focus is obtained, and two lenses have to be arranged perpendicularly in series to get a point focus. The focal length of real optics is long against their thickness. Therefore, the elliptical path can be linearized leading to a wedged geometry of the multilayer stack [3]. The necessary tilt of a zone which has a distance h' from the optical axis results from the Bragg equation and thus

$$\theta(h') = \frac{h'}{2f}.$$

Such a linear incline can be realized, if a strong thickness gradient is incorporated during layer deposition [3]. However, MLLs that are obtained from such depositions are fixed for operation at a certain photon energy chosen by the geometric design, and requirements regarding precise thickness control might conflict with measures to realize the gradient.

This approach is based on a generic MLL deposition without a strong gradient in layer thickness. The raw MLL is prepared in a way that it stands freely onto the substrate without any connection to the remaining multilayer stack. Subsequently, a stress layer is deposited onto the front side, leading to an elastic deformation of the lens. This preparation results in a relative tilting of the zones along the height of the MLL according to the requirements of a wedged MLL. The final shape can be controlled with the composition and the thickness of the additional

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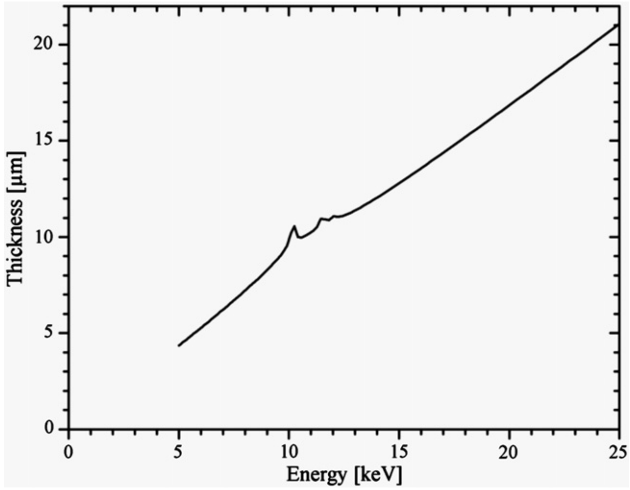

Fig. 1. Thickness of an MLL to obtain optimal diffraction efficiency as a function of the photon energy; index of refraction taken from [7].

layer. In this study, silicon dioxide grown by reactive ion beam sputter deposition yielding a compressive residual stress of −1 GPa has been chosen. The relative layer tilt is subsequently analyzed with laboratory X-ray microscopy (XRM) which provides the necessary input for an iterative process to find suitable parameters.

2. Calculation

A finite element analysis (FEA) is performed to determine a suitable thickness of the stress layer and to understand disturbing elastic deformations perpendicular to the desired bending. A simple model is used that consists of a block of silicon substrate, the MLL material and a SiO_2 stress layer at the front side, see Fig. 2a. For simplification, all materials are modeled with isotropic elastic properties, although single crystalline silicon is anisotropic and the multilayer stack shows transverse isotropy. The dimensions are set to values of real lenses for future application at $E = 20.0$ keV that have a width of 100 µm, a height of 54 µm and a thickness of 16.8 µm. Simple stiffening elements are added to the sides of the lens to reduce unwanted deformations. As boundary conditions, the displacement of all bottom nodes is fixed and a symmetry boundary condition is applied to the center plane of the lens. Therefore, only one half of the lens is modeled. The effect of residual stress is simulated by a mismatch of thermal expansion during temperature change between the stress layer with a certain coefficient of thermal expansion (CTE) and both other materials whose CTE is set

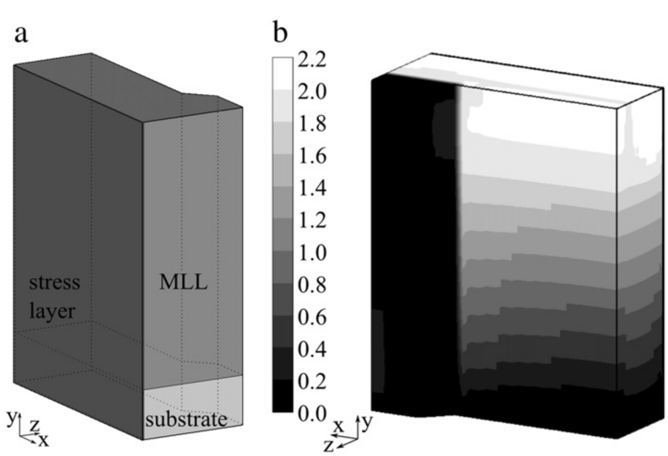

Fig. 2. Finite element analysis: (a) model showing the assignment of all materials, (b) contour plot of the relative layer tilt in mrad. A coarse grayscale map was chosen to emphasize the deviations along the width of the MLL.

to zero. Subsequently, the temperature change is modified iteratively until the compressive stress inside the stress layer equals −1 GPa.

For X-ray applications, two properties are of interest: first, the difference in vertical displacement Δu along the width of the aperture has to be small compared to the corresponding zone width. This information can be directly extracted from the FEA. In general, Δu increases with increasing distance from the substrate, if no stiffening elements are used. This can be explained with the biaxial stress resulting from the SiO_2 layer. It is a favorable situation that the thinnest layers are deposited onto the substrate. Second, the relative layer tilts are calculated from the output of the FEA according to the following algorithm that is implemented in a program. For each element, the corresponding nodes are ordered and their positions and displacements are determined. Subsequently, two nodes with minimum and maximum positions along the optical axis are identified and the tilt angle can be calculated from their vertical displacements and projected distance in beam direction. The calculated tilt angle is assigned to all nodes of the element and the result is added as an additional dataset to the FEA output. Fig. 2b shows a contour plot of a result, indicating that a linear increase can be expected along the height of the MLL. However, a slight variation along the bottom of the MLL is noticed.

The relative layer tilt can be determined experimentally with laboratory XRM if a tilt series of the lens is analyzed. In this case, the MLL is imaged with a Fresnel zone plate and the corresponding radiographs at different angles are acquired. At first glance, a diffraction pattern is observed that changes with the tilt angle. It can be assumed that the recorded intensity I at a certain position is defined by illumination, thickness of the MLL, local zone width and tilt of the zone with respect to the optical axis. The first two parameters are fixed and the zone width just depends on the vertical position. Therefore, $I(\theta)$ is assumed to have a fixed dependency for each position. Considering an MLL A whose layers are parallel to the optical axis at $\theta = 0$ and a second MLL B that is rotated by $\Delta\theta$, it will be $I_A(\theta) = I_B(\theta - \Delta\theta)$. $\Delta\theta$ can be determined by cross correlation, e.g. if the center of gravity of $I_A(\theta) \star I_B(\theta)$ is found. This method will be applied to tilt series that are acquired before and after stress layer deposition. Here, for each pixel of the radiograph, $\Delta\theta$ is calculated. The initial state is assumed to have all layers in parallel to the optical axis. The distribution of $\Delta\theta$, that can be shown as a map over the full aperture of the MLL, then reveals relative tilts at high spatial resolution that are caused by the stress layer.

3. Experimental details

An MLL deposition is used that has a slight radial decrease in layer thickness to achieve focal length matching of crossed lenses in the future [4]. It consists of 6488 WSi_2/Si layers with a thickness of 5.3 nm to 20 nm, a total height of 54 µm and a focal length of 10.6 mm at $E =$ 17 keV. The requested relative tilt between the first and the last layer for this photon energy is 2.55 mrad. 50 µm thick stripes are cut out of the wafer, and the actual MLL is subsequently milled with focused ion beam (FIB) to the requested thickness of 14.5 µm, including some stiffening elements at both sides. In addition, the lens element is detached by FIB from the remaining multilayer stack at both sides and a 300 nm thick SiO_2 stress layer is deposited onto its front side by reactive ion beam sputter deposition. This thickness was chosen based on former FEA calculations and preliminary experiments with stress layers on other MLLs. An energy of 1 keV for sputtering Ar^+ ions is used which results in a relatively high compressive residual stress within the deposited layer of about −1.0 GPa. Fig. 3a shows a micrograph of the resulting MLL.

The XRM measurements are performed in an Xradia NanoXCT-100 laboratory X-ray microscope using the Fresnel zone plate with a field of view of 66.5 µm. Tilt series are recorded within an angular range of 3.0° in 75 steps. To image the entire lens, two radiographs are stitched at each angle to increase the field of view, see Fig. 3b. The radiographs are aligned using ImageJ [5] and the TurboReg plugin [6]. An ImageJ

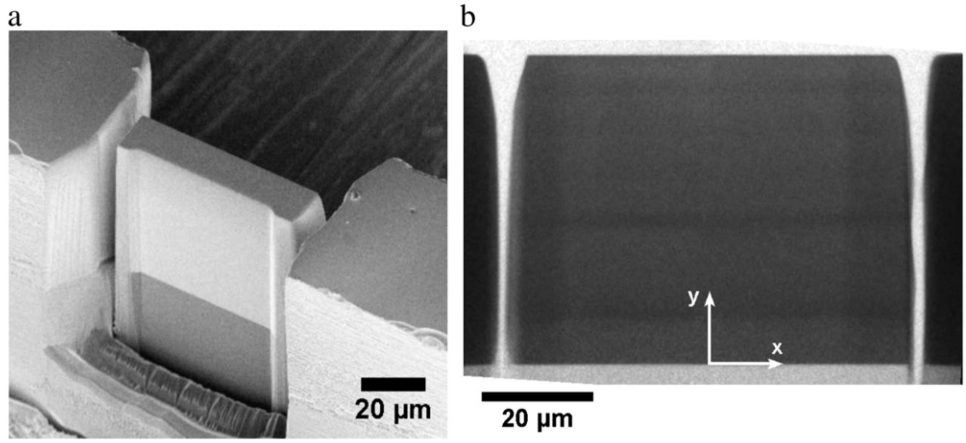

Fig. 3. (a) SEM micrographs of the MLL before stress layer deposition, (b) XRM radiograph of the entire lens with diffraction pattern.

plugin implementing the algorithm described above calculates a map of $\Delta\theta$ for the entire field of view.

4. Results and discussion

Fig. 4 shows representations from the tilt series. The intensity is plotted as a map in (y, θ) space before and after layer deposition, x is fixed to be in the center of the lens. The expected differences are visible comparing both plots. The pattern is almost horizontally symmetric before layer deposition. With the stress layer, the pattern is significantly shifted to larger angles at increasing y values.

Fig. 5a shows the calculated map of the relative layer tilt for this MLL. As expected, an increasing relative layer tilt along the height of the MLL is observed. Line plots of $\Delta\theta$ can be analyzed along the width and height of the MLL for a detailed evaluation, see Fig. 5b. An almost linear increase of the tilting angle along the height of the lens is observed. The slope is fitted by linear regression to 0.031 mrad/μm, which is less than the requested value of 0.047 mrad/μm necessary for a photon energy of $E = 17$ keV. The optimum performance of this lens should be expected at $E = 25$ keV. $\Delta\theta$ is almost constant along the width of the MLL at the top-most position while a relaxation is noticed at its bottom. The latter fact is in good agreement with the results from FEA. The observed deviations are related to the experimental conditions of this initial experiment. In particular, the deposition of the stress layer is not yet optimized for this kind of samples, and e.g. shadowing effects due to the topography of the lens have to be expected.

To test the robustness of the presented method to obtain the $\Delta\theta$ distribution over the aperture of the MLL, typical misalignments were enforced and the acquired datasets were evaluated against the initial state. First, the entire MLL specimen was rotated by 5° inside the object plane. The resulting radiographs were aligned with the initial dataset by translation and rotation. Second, the object was defocused by 200 μm what is larger than the depth of focus. In both cases, the $\Delta\theta$ evaluation showed no significant difference to zero.

According to these initial results, the approach described in this paper seems to be feasible to manufacture wedged multilayer Laue lenses. The presented analysis to measure the relative layer tilt that is based on the evaluation of tilt series of the MLL with a laboratory X-ray microscope is expected to be a robust approach to analyze the desired elastic deformation of the lens. Along the height of the MLL, a linear increase of the relative layer tilt was observed, which can be matched to a requested value if the thickness of the stress layer is changed. There is a significant bending of the MLL noticeable along its width that is assumed to have negative impact on X-ray focusing properties. To reduce this effect below critical levels, optimal parameters for stiffening elements at the sides of the MLL have to be determined based on FEA, and the width of the MLL has to be enlarged compared to the size of the aperture to keep those boundary effects away from the

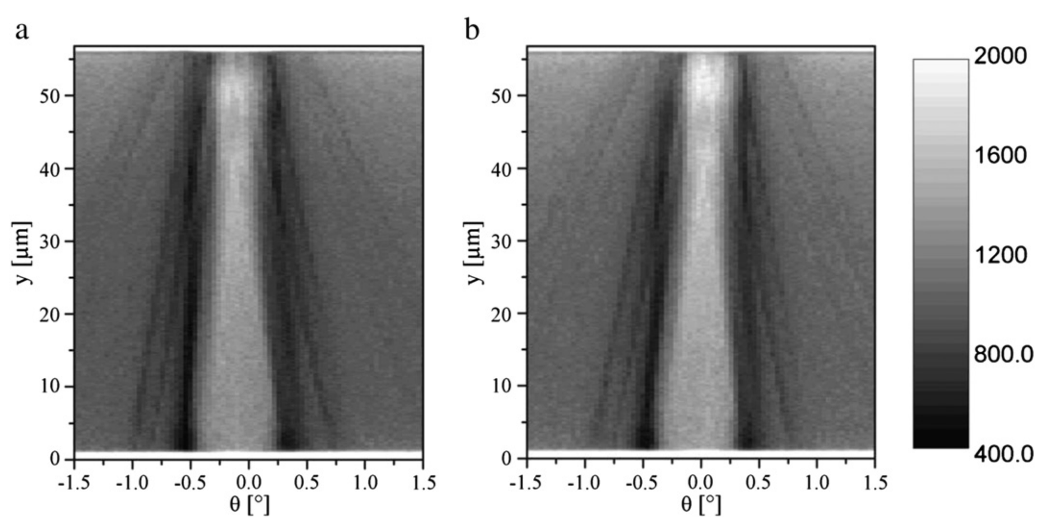

Fig. 4. Intensity map in y-θ space (a) before and (b) after stress layer deposition at the center of the MLL.

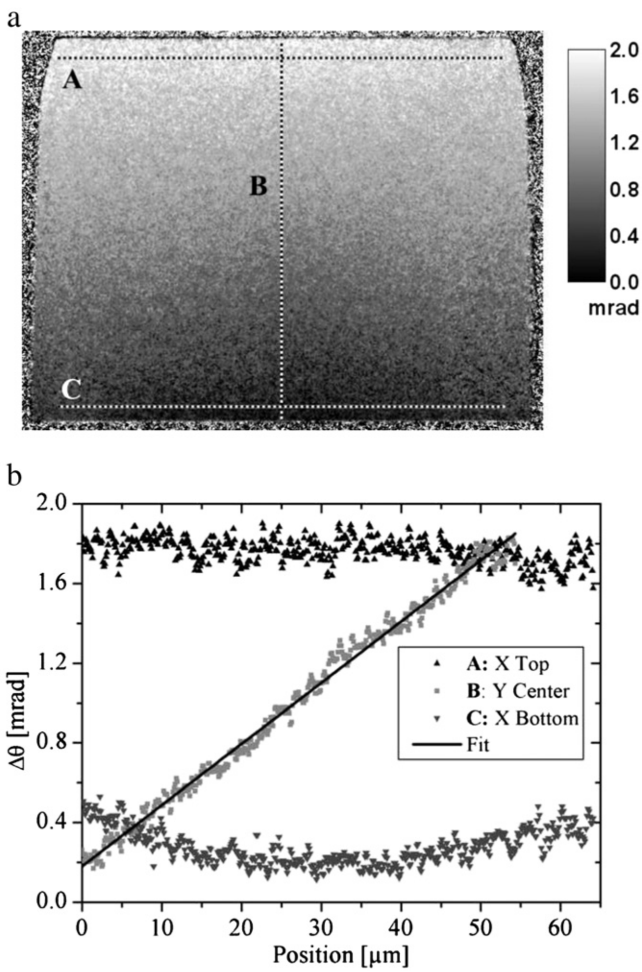

Fig. 5. (a) Map of $\Delta\theta$, (b) line plots of $\Delta\theta$ along A, B and C.

final aperture. The deposition of the additional SiO_2 stress layer has to be improved as well to avoid shadowing effects. The layer has a thickness of about 300 nm, and has a negligible absorption of 1% at $E = 5$ keV and less than 0.02% at $E = 20$ keV.

Reflecting on the measured intensity vs. tilt angle at XRM investigations, the shape of this function is mainly influenced by two factors: An offset in layer tilt shifts this function along θ, and the local zone width influences the distance of the two observed dips that are assumed to be caused by Bragg diffraction of the incident hollow cone illumination with the multilayer grating. Therefore, it should be possible to develop a fit function to find both parameters without any need of a reference. Then, this approach can also be used to analyze other effects that influence the optical properties as well and to get valuable feedback for fabrication of MLLs.

5. Conclusions

To conclude the results, we present an approach to fabricate wedged MLLs that are promising optics for single nanometer focusing of hard X-rays. In contrast to the existing method that is based on the employment of a strong thickness gradient of the multilayer deposition, the use of an additional stress layer uncouples multilayer deposition from the adjustment of the lens to a certain photon energy. This approach reduces the demands on magnetron sputter deposition, and an existing multilayer can be used to manufacture lenses for operation at several energies. The achieved tilting of the layers can be analyzed with sub-micron resolution along the full aperture of the MLL with laboratory X-ray microscopy. This analysis provides an effective way for the control of the MLL fabrication process prior to experiments with synchrotron radiation, where the optical properties are finally determined.

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