Hard X-ray one dimensional nano-focusing at the SSRF using a WSi_2/Si multilayer Laue lens*

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Abstract: The multilayer Laue lens (MLL) is a novel diffraction optics which can realize nanometer focusing of hard X-rays with high efficiency. In this paper, a 7.9 μm-thick MLL with the outmost layer thickness of 15 nm is designed based on dynamical diffraction theory. The MLL is fabricated by first depositing the depth-graded multilayer using direct current (DC) magnetron sputtering technology. Then, the multilayer sample is sliced, and both cross-sections are thinned and polished to a depth of 35–41 μm. The focusing property of the MLL is measured at the Shanghai Synchrotron Facility (SSRF). One-dimensional (1D) focusing resolutions of 205 nm and 221 nm are obtained at E=14 keV and 18 keV, respectively. It demonstrates that the fabricated MLL can focus hard X-rays into nanometer scale.

Key words: nano-focusing, hard X-ray, multilayer Laue lens, synchrotron radiation, diffraction

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1 Introduction

The X-ray microscope is an attractive tool used to probe the micro- and nano-world. Compared with microscopes using visible-light or electrons, it shows higher resolution and has a larger penetration depth. Besides, the photon energies of X-rays cover the resonance lines of almost all elements, which provide an accurate mechanism for identifying elemental and chemical compositions. X-ray microscopes combined with different spectroscopy methods have brought new opportunities in materials, biology and environmental sciences [1, 2]. Spatial resolution has always been a difficult problem during the development of X-ray microscopes. Different optics are used to focus X-rays into nanometer scale. Zone plates have produced the highest resolution of 10–12 nm in the soft X-ray range [3–5]. A refractive lens has also generated a hard X-ray nanobeam with a size of 47 nm×55 nm [6]. Reflective mirrors have shown great progress recently with the advance of precision manufacturing techniques, it focuses X-rays in one dimension to a width of 7 nm [7].

The multilayer Laue lens is a linear zone plate structure which can realize nanometer focusing of hard X-rays in one dimension [8]. It is fabricated by depositing the depth-graded multilayer inversely on a flat substrate and then slicing and thinning the multilayer sample to an ideal cross-section depth. Through this process, MLL can reach a much larger aspect-ratio (cross-section depth to the outmost layer thickness) compared with zone plates which make it capable of focusing hard X-rays with high efficiency [9]. Based on this method, a line focus of 16 nm width with an efficiency of 31% has been obtained at E=19.5 keV [10]. Two dimensional focusing can be realized by assembling two MLLs in orthogonal directions. And a nanoscale 2D imaging with 25 nm×27 nm resolution was reported recently [11]. In this paper, we will report our design, fabrication and

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measurement results on developing this novel nano-focusing optics.

2 Design of MLL

MLL can be treated as a series of volume gratings with large aspect-ratio while the diffraction property has to be analyzed using the dynamical diffraction theory [12]. Due to the dynamical diffraction effect, both the diffraction efficiency and the focusing performance of MLL are sensitive to the Bragg condition [13]. Thus, there are basically three types of MLL as shown in Fig. 1. The flat MLL is similar to a linear zone plate structure while all layers are parallel to the optical axis (Fig. 1(a)). To approximate the Bragg condition, the MLL is tilted with a fixed angle as shown in Fig. 1(b). In the ideal case, each layer is tilted by a different angle so that the Bragg condition is satisfied in all areas, $\theta=\theta_{\rm B}\approx\lambda/(2\Lambda)$, Λ is the period of the local grating. This is the wedged structure (Fig. 1(c)).

The MLL used here was designed at E=14 keV using WSi_2 and Si as the material combination. The structural parameters are listed in Table 1. The one-dimensional coupled wave theory was used to calculate the diffraction efficiency of the three types of MLL [14].

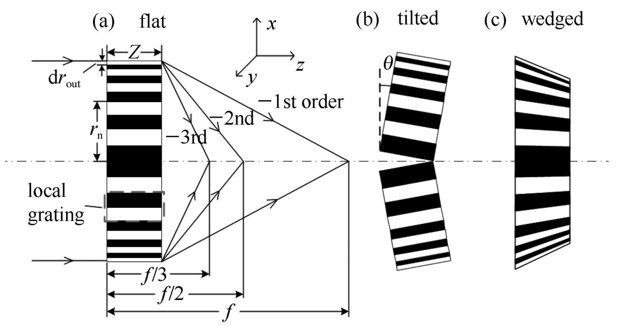

Fig. 1. A schematic of the MLL structure.

Table 1. Structural parameters of the designed MLL.

E=14 keV	$r_{\rm out}$/μm	f/mm	$dr_{\rm out}$/nm	$t_{\rm dep.}$/μm	$N_{\rm dep.}$
WSi_2/Si	10.3	3.53	15	7.9	324

The focusing property was calculated using the Fresnel-Kirchhoff diffraction formula [15]. A full MLL structure consists of two depth-graded multilayers as shown in Fig. 1. However, to make the two halves focus on the same spot, the layer position errors of the two multilayers have to be limited within a few nanometers. This requires a very high precision of fabrication [16]. A half MLL structure can still realize 1D focusing of X-rays at the sacrifice of partial resolution and photon flux. Moreover, the central layers of each half MLL are very thick, which contribute little to the focusing, they can also be omitted during deposition. Thus, the total thickness of the actual deposited multilayer is 7.9 μm, which only corresponds to 38% of the full structure. To compare the focusing property, the intensity profiles in the focal plane of the full MLL, half MLL and the 7.9 μm-MLL are shown in Fig. 2. For the full MLL with an outmost layer thickness of 15 nm, the full width half maximum (FWHM) of the focal line is 12.4nm. For the half MLL, the focal line broadens to 27.4 nm as the numerical aperture is half decreased. After the central layers are omitted, the width of the focal line is 36.4 nm. It can be seen that the 7.9 μm-MLL can still achieve high spatial resolution while the fabrication is much easier.

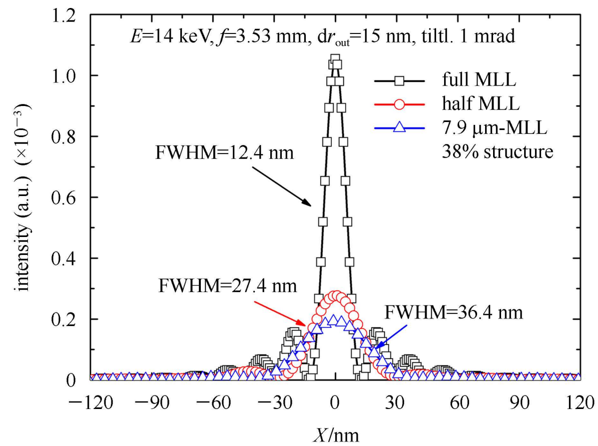

Fig. 2. Intensity profiles in the focal plane of the full and half MLL structures.

3 Fabrication of MLL

The fabrication of the MLL can be divided into two parts. The first one is the deposition of the depth-graded multilayer, and then the multilayer sample is sliced and thinned to a cross-section depth of tens of microns.

The material combination of WSi_2/Si is selected to fabricate the MLL due to its stable stress property and sharp interfaces [17, 18]. The multilayer is deposited on super polished silicon substrate using direct current (DC) magnetron sputtering technology. The base pressure before deposition is 2.0E-4 Pa and Ar is used as the working gas with a pressure of 0.2 Pa. The layer thicknesses range of the designed MLL is 15–64 nm and the deposition rates of WSi_2 and Si are 0.4 nm/s and 0.3 nm/s, respectively.

After deposition, the multilayer was sliced into pieces along the direction perpendicular to the surface. Then, both cross-sections of the multilayer sample were ground using SiC and diamond abrasives with different particle sizes to thin the depth. Chemical mechanical polishing (CMP) was finally used to remove damage and improve the smoothness. Details of the sampling process has been reported elsewhere [19]. The cross-section of the multilayer was observed by a scanning electron microscope

(SEM) after the sampling process as shown in Fig. 3. Fig. 3(a) shows the entire multilayer structure. Fig. 3(b) and (c) are the enlarged images of the areas close to the surface and substrate, respectively. It can be seen that the multilayer structure is kept undamaged after the repeated grinding and polishing. All the layer interfaces are still flat and sharp. Although the optimum depth of the 7.9 μm-MLL is 14 μm, it is very difficult to fabricate this ultra-thin sample. In this paper, we set the depth of the MLL as $z=\sim 40$ μm which is the second maximum position of the diffraction efficiency. The fabricated depth of the 7.9 μm-MLL is 35–41 μm, which indicates an aspect-ratio of more than 1000.

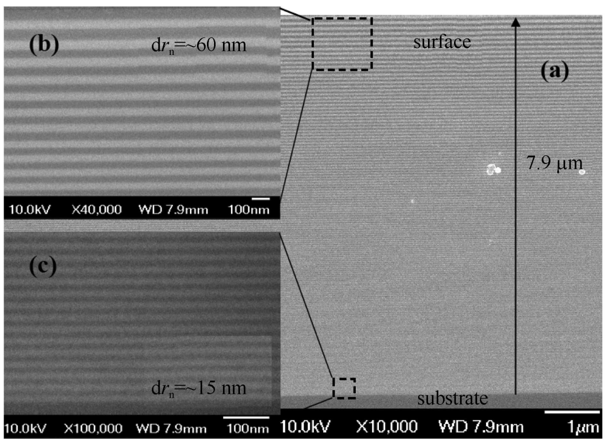

Fig. 3. SEM images of the cross-section of 7.9 μm-MLL after the sampling process.

4 Nano-focusing measurement at SSRF

The nano-focusing experiment of the MLL was performed at Beam-line 15U, Shanghai Synchrotron Radiation Facility (SSRF). According to the optics layout in the beam-line, the emitted radiation of the undulator is deflected by a toroidal mirror which collimates the X-ray beam in the vertical direction and focuses it in the horizontal direction. A double crystal monochromator equipped with Si(111) is used to do the spectral filtering of the radiation which produces an energy resolution of ∼1.5E-4 at E=14 keV. Precise slits in front of the MLL allow illumination of only the multilayer structure which avoids the background of the transmitted X-rays.

The nano-focusing experiment system in BL15U is shown in Fig. 4. The incident beam size was shaped by slit4 to a size of ∼8 μm×100 μm (V×H). The MLL was mounted in the head of the extended stage to produce the millimeter focal length. As the incident beam shows better spatial coherence in the vertical direction [20], the MLL was aligned to focus in this direction. The focal line was measured by using a Cu thin film with the cross-section of 8nm in width and 15 μm in depth. The Cu film was carefully aligned parallel to the focal line and then scanned across the focus using a nanometer translation-stage. The intensity of Cu Kα lines was monitored by the fluorescence detector which finally shows the intensity distribution of the focus line.

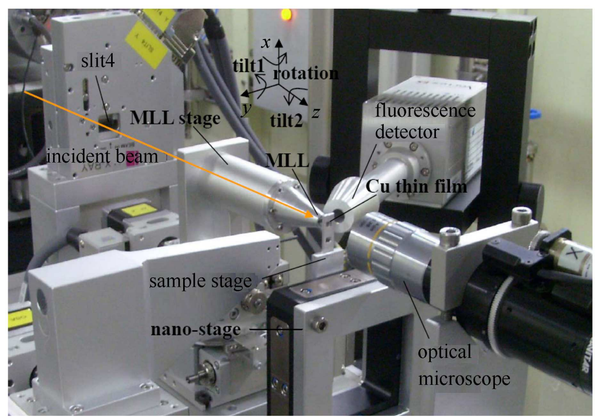

Fig. 4. The nano-focusing experiment system in beam-line 15U.

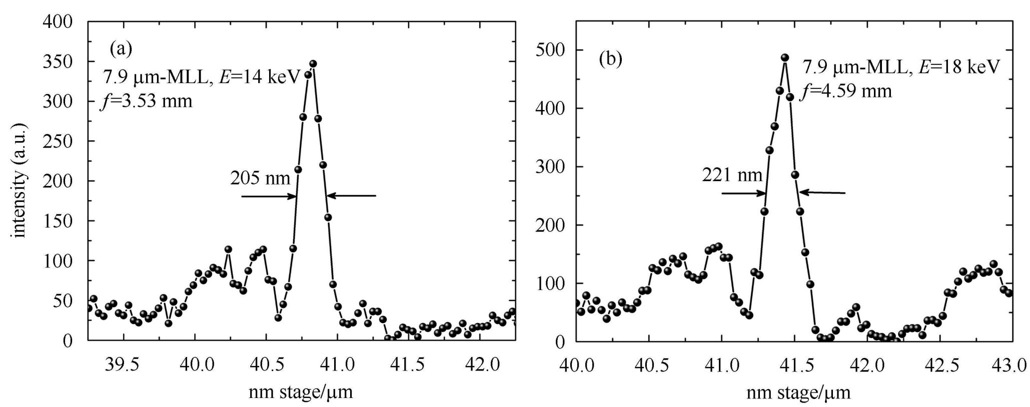

Fig. 5. The measured focal lines of the 7.9 μm-MLL at E=14 keV (a) and 18 keV (b).

The measured focusing results of the 7.9 μm-MLL are shown in Fig. 5. The width of the focal line at E=14 keV is 205 nm (FWHM). There is little side-lobe around the focus. This measurement has been performed several times to ensure repeatability. To characterize the focusing property of the MLL at higher energy, the MLL was also measured at 18 keV and the focus was 221 nm in width.

The discrepancy between the measured width and the theoretical value can be attributed to three reasons. First, the layer position errors of the depth-graded multilayer and the structural damage induced by the sampling process will broaden the focal line. Second, the one dimensional focusing measurement using the Cu film requires a high alignment precision; the unparallel between the focal line and the Cu film will enlarge the measured result. Third, the wave front aberration of the incident beam and the vibration of the stages will also broaden the focus. Further research will be made to improve the MLL preparation and the measurement system at the SSRF.

5 Conclusion

A 7.9 μm-thick multilayer Laue lens with an outermost layer thickness of 15 nm is designed and fabricated in this paper. Based on the dynamical diffraction model, the 7.9 μm-MLL shows a high focusing resolution of 36.4 nm in theory. After fabrication, the focusing property of the MLL was measured at the SSRF. The measured results show that a focusing resolution of 205–221 nm was achieved at E=14 keV and 18 keV. To the best of our knowledge, it is the first report of nano-focusing optics in the hard X-ray region in China. More efforts will be made to improve the fabrication process and the nano-focusing experiment system to further decrease the resolution of the MLL.

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