Multilayer Laue lens for focusing X-ray into nanometer size

Jingtao Zhu (朱京涛), Qiushi Huang (黄秋实), Haochuan Li (李浩川),
Jing Xu (徐 敬), Xiaoqiang Wang (王晓强), Zhong Zhang (张 众),
Zhanshan Wang (王占山)*, and Lingyan Chen (陈玲燕)

Institute of Precision Optical Engineering, Physics Department, Tongji University, Shanghai 200092, China
**E-mail: wangzs@tongji.edu.cn*

Received November 25, 2009

A multilayer Laue lens with a 15-nm outermost zone width is designed for an incident X-ray beam with an energy of 8 keV. WSi_2/Si multilayer Laue lens with 324 layers and a total thickness of 7.9 μm is successfully fabricated using direct current magnetron sputtering method. After deposition, the multilayer is sliced and polished to achieve the ideal aspect ratio. Characterization results show that the multilayer structure is kept intact and the surface roughness is approximately 0.9 nm after slicing and repeated polishing.

OCIS codes: 340.0340, 310.1860, 340.7440, 3406720.
doi: 10.3788/COL201008Sl.0174.

Efficient focusing of X-ray beam to nanometer scale has always been a challenge, the accomplishment of which could bring great benefits to the development of material science, environmental science, and biomedicine. With the development of synchrotron radiation, high-brilliance and high-collimation X-ray sources have become available. The only limitation that remains is that of nanometer focusing optics, for which several methods have been proposed. Refractive lens can provide an X-ray spot with a lateral extension of 47 × 55 (nm)[1] while a K-B mirror can generate an X-ray nanobeam with a size of 85 × 95 (nm)[2]. For diffractive optics, a zone plate is a powerful tool used in the soft X-ray range because it provides a spatial resolution better than 15 nm[3]. However, for hard X-ray focusing, the aspect ratio, $z/d_{r_{\mathrm{out}}}$, where $d_{r_{\mathrm{out}}}$ is the outmost zone width and z is the depth of a zone plate, has to be very large, which cannot be achieved by etching methods. Recently, a new diffractive focusing optics named multilayer Laue lens (MLL) has attracted great attention. This zone plate-like optics method not only overcomes the limitation of aspect ratios[3], but also improves the accuracy of layer positions and decreases roughness relative to the results with sputter-sliced zone plates deposited on a column substrate[4]. One-dimensional (1D) MLL with an outermost layer width of 5 nm is capable of focusing X-ray to a line of 16-nm width with an efficiency of 31%[5]. Here, we present our initial work on the design, fabrication, and characterization of a new focusing optics, MLL.

MLL is constructed by a series of depth-graded multilayers. The multilayer is first deposited on a flat substrate, then sectioned and polished to an ideal depth. By itself, one multilayer is capable of focusing X-rays. To increase the aperture, two multilayer slices are assembled together to form a 1D MLL (Fig. 1). Each multilayer is tilted with an angle φ to the optical axis in order to improve focus efficiency. A detailed description of the MLL model can be seen in Ref. [6].

To characterize MLL with an X-ray diffractometer, the multilayer structure was initially designed for the incident light of Cu-Kα line (E=8 keV). The outermost zone width $d_{r_{\mathrm{out}}}$ and focal length f were chosen as 15 nm and 2 mm, respectively. According to zone plate law[7], the total number of layers should be N=342 and the total thickness of the multilayer should be r_{out}=10263 nm. However, the central part of the structure was not deposited in order to leave space for assembling; thus, the number of layers N_{exp} and total thickness t_{exp} deposited in experiment were 324 and 7908 nm, respectively. Parameters of the multilayer structure are listed in Table 1.

For efficient focusing of X-rays, the aspect ratio of MLL was made very large. The diffraction kinematics model was found unsuitable for this purpose; therefore, we used the coupled-wave theory (CWT) to analyze the diffraction properties of MLL[8,9]. The whole structure

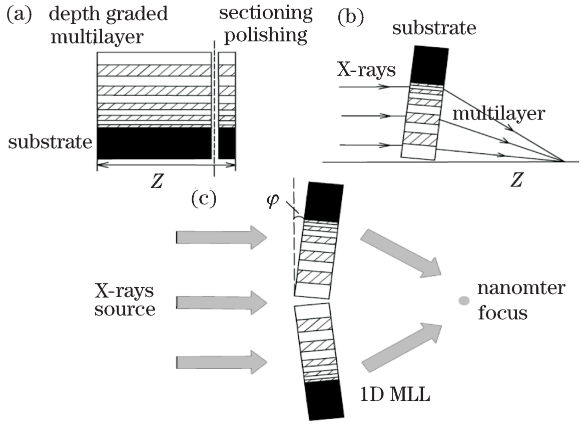

Fig. 1. Schematics of an MLL: a) and b) a depth-graded multilayer is deposited on a flat substrate, then sectioned and polished to an ideal depth; c) two multilayers are assembled together to form a 1D MLL.

Table 1. Parameters of the Zone Plate Multilayer Structure

Λ (nm)	f (mm)	$d_{r_{\mathrm{out}}}$(nm)	N	r_{out}(nm)	Nexp	t_{exp}(nm)
0.154	2	15	342	10263	324	7908

1671-7694/2010/S10174-03

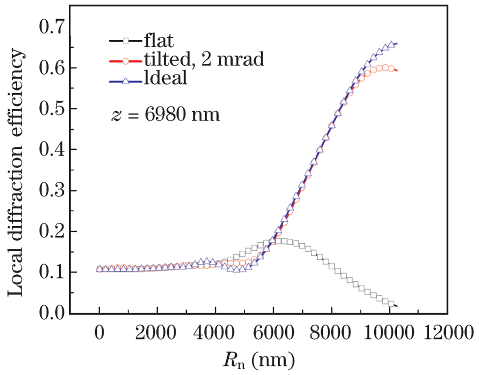

Fig. 2. Local diffraction efficiency as a function of the positions Rn of MLL. In the "flat" case (square), all layers were parallel to the optical axis with no tilted angle. In the "tilted" case (circle), the whole structure was tilted 2 mrad to the axis. In the "ideal" case (triangle), each layer was tilted with an optimum angle to satisfy the Bragg condition.

Fig. 3. SEM image of the cross-section of the zone plate multilayer structure.

can be considered as a series of local volume gratings. From the central to outer area, the period of gratings gradually decreased and the diffraction behavior transited from the Raman-Nath diffraction to the Bragg diffraction. In the Bragg regime, diffraction efficiency depends greatly on the Bragg condition; thus, to obtain higher efficiency, the multilayer structure has to be tilted with an optimum angle φ. According to 1D CWT, the complex amplitude of different diffraction orders on the rear surface $A_l(z)$ can be calculated using a numerical method. The local diffraction efficiency, $\eta = A_{-1}(z)A^*_{-1}(z)$, and the intensity distribution in the image plane can be obtained by superimposing all the wave fields propagated to the image plane.

Diffraction efficiency of local gratings in three different cases is shown in Fig. 2. The depth of MLL, z, was optimized at 6980 nm. It can be seen that in the "flat" case, local diffraction efficiency dropped quickly at the outer area where layer thicknesses have become smaller. In the "tilted" case, the efficiency curve was very close to the "ideal" case throughout the whole structure. Diffraction efficiency of the tilted MLL reached a maximum of 60% at the outer area. Thus, the multilayer structure of MLL has to be tilted with an optimum angle in practical use to improve the diffraction efficiency. According to calculations of the intensity distribution at the image plane, the resolution of the tilted case was 10.5 nm.

To fabricate the zone plate of MLL, which has a great number of layers, a material combination with very stable stress state and sharp interfaces is crucial. Combinations of Mo/Si, $MoSi_2$/Si, W/Si, and WSi_2/Si were studied. The combinations of Si and silicide showed more stable stress state and smaller interface diffusion, which is consistent with the results reported in Refs. [10] and [11]. Here, the combination of WSi_2/Si was also used to fabricate MLL.

The multilayer structure was deposited on flat Si substrates using direct current (DC) magnetron sputtering technology. To better control the positions and quality of the outmost layers that determine the resolution of MLL[12], the multilayer was deposited in inverse order, from layer 342 to 19. The whole sputtering process lasted for 16 h. To calibrate the drifts of growth rate during the whole process[13], periodic multilayer samples were deposited at the beginning, middle, and final stages. A thick periodic multilayer with 361 periods was also deposited during the whole process, whose thickness was measured to be 14.32 nm. It was determined that during the 16 hours of sputtering, the growth rates of WSi_2 and Si drifted no more than 2%, which indicates a relatively stable sputtering process.

After deposition, the zone plate multilayer was sliced, and the cross-section was observed using scanning electron microscopy (SEM). As illustrated in Fig. 3, the interfaces are sharp and flat. No buckling or peeling is observed, which indicates a very stable stress state of the multilayer. To characterize the multilayer structure precisely, the whole cross-section was divided into eight parts and scanned by SEM. Analyzing the results show that the fabricated multilayer structure is consistent with the designed structure.

To obtain the ideal aspect ratio, the multilayer structure had to be sliced and polished to the desired depth. The zone plate multilayer was prepared into plane-shaped samples and wedge-shaped samples, which were used to investigate the dependence of diffraction property on depth.

A series of thinning processes similar to the TEM specimen preparation[14] were performed. First, the silicon substrate with a deposited multilayer was sliced by a diamond cutter into 2 ×5 (mm) pieces. Two pieces of samples were then bonded face-to-face to protect the multilayer from damage during polishing.

The process of the cross-section polishing included several procedures. Raw abrasive papers and diamond lapping films with particle sizes of 15, 3, and 1 μm were used alternately for manual polishing to decrease the depth and remove the damage caused by the last procedure. However, the multilayer could not be observed by SEM after 1-μm lapping film polishing. Thus, a 0.5-μm diamond suspension was used to remove the damage on the cross-section of the multilayer and to improve the smoothness. After polishing, the depth of the tip area of the wedge sample was 10 μm, with an aspect ratio of 666. The depth of the plane sample was approximately 10 μm.

To characterize the multilayer structure and diffraction property of MLL after polishing, SEM and atomic force microscopy (AFM) measurements were performed. As shown in the SEM image (Fig. 4), the multilayer struc-

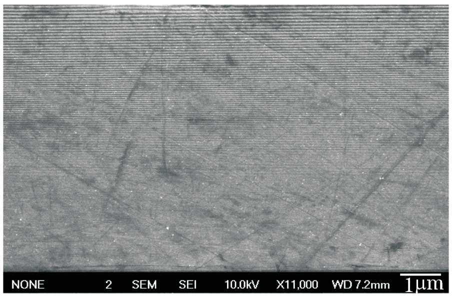

Fig. 4. SEM image of the polished surface of the multilayer sample.

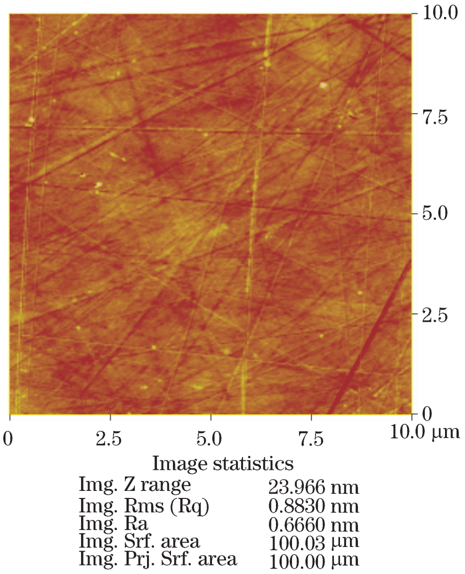

Fig. 5. AFM image of the polished surface of the multilayer sample.

ture was kept intact, and interfaces were sharp and flat after sectioning and repeated polishing.

To quantitatively evaluate the surface roughness of the polished surface, AFM measurement was used. Different areas of the surface were measured and the roughness was approximately 0.9 nm (root mean square) (Fig. 5). Additional polishing processes for smoother surfaces are currently being studied in our laboratory.

In conclusion, MLL used at the incident energy of 8 keV and with an outermost layer width of 15 nm is designed. The local diffraction efficiency at the outer area of the MLL reaches 60%, with a resolution of 10.5 nm. A WSi_2/Si zone plate multilayer with 324 layers and a total thickness of 7.9 μm is fabricated using DC magnetron sputtering. To acquire the ideal aspect ratio, the multilayer structure is sectioned and polished to the desired depth. Depth of the tip area of the wedge-shaped sample is 10 μm, while the aspect ratio is 666. Depth of the plane-shaped sample is approximately 10 μm. SEM and AFM measurements show that the multilayer structure is kept intact after slicing and repeated polishing, while the roughness of the polished surface is approximately 0.9 nm. The focusing property of the multilayer Laue lens will be researched in future experiments.

This work was supported by the National Natural Science Foundation of China (Nos. 10675092 and 10975139), Shanghai Education Development Foundation (No. 2008CG25), Shanghai Natural Science Foundation (No. 09ZR1434300), and Young Talents Foundation of Tongji University. The authors would like to thank Prof. X. Wu and Mr. X. Zhang of Fudan University for their help in SEM measurement.

References

1. C. G. Schroer, O. Kurapova, J. Patommel, P. Boye, J. Feldkamp, B. Lengeler, M. Burghammer, C. Riekel, L. Vincze, A. van der Hart, and M. Küchler, Appl. Phys. Lett. **87,** 124103 (2005).
2. W. Liu, G. E. Ice, J. Z. Tischler, A. Khounsary, C. Liu, L. Assoufid, and A. T. Macrander, Rev. Sci. Instrum. **76,** 113701 (2005).
3. W. Chao, B. D. Harteneck, J. A. Liddle, E. H. Anderson, and D. T. Attwood, Nature **435,** 1210 (2005).
4. C. Liu, R. Conley, A. T. Macrander, J. Maser, H. C. Kang, M. A. Zurbuchen, and G. B. Stephenson, J. Appl. Phys. **98,** 113519 (2005).
5. H. C. Kang, H. Yan, R. P. Winarski, M. V. Holt, J. Maser, C. Liu, R. Conley, S. Vogt, A. T. Macrander, and G. B. Stephenson, Appl. Phys. Lett. **92,** 221114 (2008).
6. J. Maser, G. B. Stephenson, S. Vogt, W. Yun, A. Macrander, H. C. Kang, C. Liu, and R. Conley, Proc. SPIE **5539,** 185 (2004).
7. D. T. Attwood, *Soft X-Rays and Extreme Ultraviolet Radiation: Principles and Applications* (Cambridge, London, 1999).
8. Q. Huang, H. Li, J. Zhu, T. Sang, Z. Wang, and L. Chen, Acta Photon. Sin. (in Chinese) **38,** 2299 (2009).
9. J. Maser and G. Schmahl, Opt. Commun. **89,** 355 (1992).
10. A. I. Fedorenko, V. V. Kondratenko, L. S. Palatnik, S. A. Yulin, and E. N. Zubarev, Proc. SPIE **2453,** 11 (1995).
11. C. Liu, R. Conley, and A. T. Macrander, Proc. SPIE **6317,** 63170J (2006).
12. M. Born and E. Wolf, *Principles of Optics* (Cambridge, London, 1999).
13. C. Liu, R. Conley, A. T. Macrander, J. Maser, H. C. Kang, and G. B. Stephenson, Thin Solid Films **515,** 654 (2005).
14. L. D. Madsen, L. Weaver, and S. N. Jacobsen, Microsc. Res. Tech. **36,** 354 (1997).