Thinning and polishing of cross-section of depth-graded WSi_2/Si multilayer for linear zone plate application

Qiushi Huang (黄秋实)[1,2], Jingtao Zhu (朱京涛)[1,2*], Haochuan Li (李浩川)[1,2], Zhengxiang Shen (沈正祥)[1,2], Xiaoqiang Wang (王晓强)[1,2], Zhanshan Wang (王占山)[1,2], and Yongjian Tang (唐永建)[3]

[1]*Institute of Precision Optical Engineering, Physics Department, Tongji University, Shanghai 200092, China*

[2]*Shanghai Key Laboratory of Special Artificial Microstructure Materials and Technology, Tongji University, Shanghai 200092, China*

[3]*Research Center of Laser Fusion, China Academy of Engineering Physics, Mianyang 621900, China*

[*]*Corresponding author: jtzhu@tongji.edu.cn*

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A linear zone plate named multilayer laue lens (MLL) is fabricated using a depth-graded multilayer structure. The lens shows considerable potential in focusing an X-ray beam into a nanometer scale with high efficiency. In this letter, a depth-graded multilayer consisting of 324 alternating WSi_2 and Si layers with a total thickness of 7.9 μm is deposited based on the thickness sequence according to the demands of the zone plate law. Subsequently, the multilayer sample is sliced and thinned to an ideal depth along the cross-section direction using raw abrasives and diamond lapping. Finally, the cross-section is polished by a chemical mechanical polishing (CMP) technique to remove the damages and improve the surface smoothness. The final depth of the MLL is approximately 7 μm with an achieved aspect ratio greater than 400. Results of scanning electron microscopy (SEM) and atomic force microscopy (AFM) indicate that interfaces are sharp, and the multilayer structure remains undamaged after the thinning and polishing processes. The surface roughness achieved is 0.33 nm.

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High-brilliance synchrotron X-ray sources enhance the feasibility of nanobeam focusing, which is helpful to X-ray microscopy analysis under higher resolution. Several components have been developed to focus X-rays into a nanobeam, such as Kirkpatrick-Baez mirrors[1], compound refractive lens[2], Fresnel zone plate lens[3], and graded multilayer mirrors[4]. Fresnel zone plates have been demonstrated as powerful focusing lenses in X-ray microscopy applications. However, for hard X-rays, the aspect ratio of a zone plate (ratio of the depth of a zone plate to its outermost zone width) becomes exceedingly large that could not be achieved using traditional lithographic techniques. A novel linear zone plate, named multilayer laue lens (MLL), is developed by depositing the depth-graded multilayer reversely on flat substrates and subsequently slicing and polishing the lens to achieve a larger aspect ratio. As a result, MLL has shown capability to provide a significantly higher focusing efficiency and resolution especially in hard X-ray regions[5,6]. Furthermore, the outermost layers (i.e., the thinnest layers) of MLL, which determine the resolution, are firstly deposited on a substrate to control the thicknesses and quality of the critical layers better. A MLL structure with an outermost layer thickness of 5 nm has focused the 19.5-keV X-ray into a line focus of 16 nm, with an efficiency of 31%[7].

In this letter, a MLL is designed and a thickness-graded multilayer with 324 layers is successfully deposited using WSi_2 and Si alternately. The detailed slicing, thinning, and polishing processes of the multilayer sample are reported to form a nearly perfect linear zone plate with a high aspect ratio.

The MLL was designed at energy E=8 keV, with a focal length of 2.04 mm. According to the zone plate law, the depth-graded multilayer should consist of 324 alternating WSi_2 and Si layers with a total thickness of 7.9 μm. The outermost layer thickness of the zone plate is 15 nm. In the present study, the layer thickness increases from 15 to 65 nm as shown in Fig. 1.

The material combination of WSi_2/Si was chosen to fabricate the MLL because of its stable stress property[8]. To ensure better control of the position and quality of the outermost layers, which determine the focusing property, the multilayer was deposited reversely on a super polished Si(100) substrate using direct current magnetron sputtering technology[9,10]. After deposition, the cross-section of the multilayer was observed by scanning electron microscopy (SEM), as shown in Fig. 2. Figure 2(a) is an image of the entire cross-section, and Fig. 2(b)

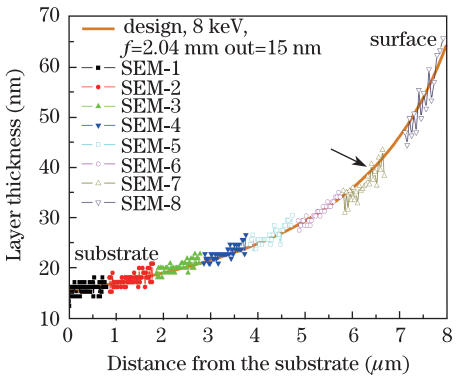

Fig. 1. Designed and deposited multilayer structure of MLL. The total number of layers is 324 with a total thickness of 7.9 μm.

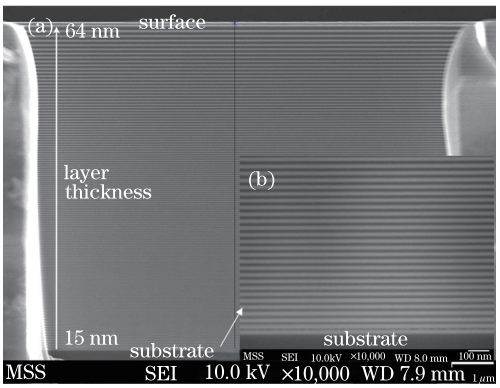

Fig. 2. SEM images of the cross-section of WSi_2/Si thickness-graded multilayer showing (a) the entire cross section and (b) an enlarged image of the area of the layers close to the substrate.

is an enlarged image of the area of the layers near the substrate. The bright layers are WSi_2 and the dark layers are Si. All interfaces are evidently sharp and flat, which indicates the good quality of the multilayer.

To measure the deposited multilayer structure, the 7.9-μm cross-section was divided into eight parts and observed with larger magnification separately. The measured multilayer structure is also shown in Fig. 1 for comparison. The deposited multilayer structure is mainly consistent with the design from the substrate to the surface. The oscillation of the measured layer thicknesses is caused by the inaccuracy of the SEM measurement. Moreover, the small deviation of the seventh part, which is indicated by an arrow, shown in Fig. 1, is attributed to the layer position errors induced by the stitching of the eight images together.

MLL can achieve a larger depth through the slicing and thinning processes. Large depth makes it superior to traditional zone plates fabricated using e-beam lithography techniques. Thus, the thinning process used to obtain the ideal aspect ratio is crucial in the fabrication of a MLL. According to the calculated results, the ideal depth of the linear zone plate is 7 μm. Thus, the aspect ratio is 466 for an outermost layer having a thickness of 15 nm. To produce this ultra-thin cross-section with an intact multilayer structure, the sample should be sliced and extremely thinned to the desired thickness.

A fabrication process which combines the methods of sample preparation for transmission electron microscopy (TEM) and ultra-smooth surface machining techniques was adopted[11−13]. Figure 3 is the schematic of the process.

The depth-graded multilayer sample shown in Fig. 3(a) was sliced into pieces using a diamond dicing cutter. The dimensions of each piece are 5 mm (length) and 2 mm (width), as shown in Fig. 3(b). This leaves a large area for the removal of the damage caused by dicing[14]. To protect the multilayer during the thinning and polishing, a sandwich structure was formed by gluing a clean piece of Si wafer of the same size onto the surface of the multilayer (Fig. 3(c)). Subsequently, the sandwich sample was mounted on a holder using bonding wax, which provides high bonding strength for the sample. Both of the two cross-sections of the sample required thinning and polishing to obtain the desired 7-μm depth (Figs. 3(d)–(g)). The details of the process are reported below.

Initially, the cross-section was grinded using raw abrasives to quickly reduce the depth and to obtain a rough plane. Subsequently, the cross-section was grinded and lapped sequentially using a fine diamond lapping film with particle sizes of 15, 3, and 1 μm. The sequential reduction in grit size reduces the depth and gradually decreases the damages caused by the pre-procedure.

To remove the surface damages further, a 0.5-μm diamond suspension applied with polyurethane pads was used to polish the cross-section. The polished sample was characterized by SEM. Although the multilayer structure was visible, as shown in Fig. 4, the agglomeration of diamond particles induced damages and scratches on the surface, as highlighted by the arrows. In spite of the additional polishing done on the sample by a 0.1-μm diamond suspension, several small scratches caused by the pointed shape and high degree of hardness of the diamond particles have remained. These imperfections can reduce the performance of MLL and should therefore be improved.

To remove all the damages and scratches and improve the smoothness of surface, chemical mechanical polishing

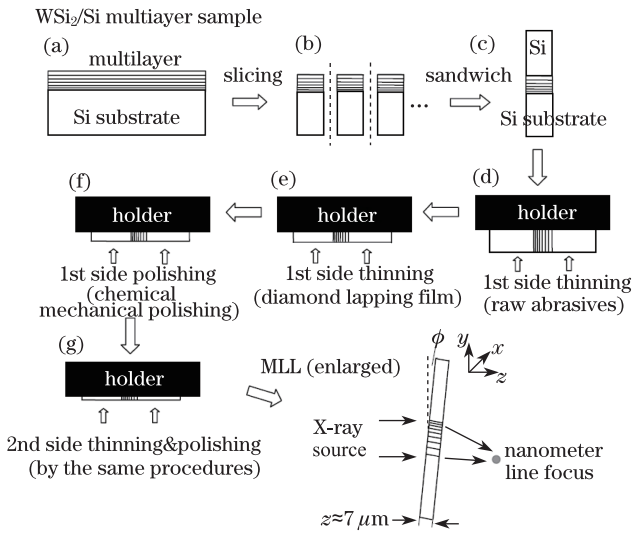

Fig. 3. Schematic illustrations of the thinning and polishing processes of the multilayer sample.

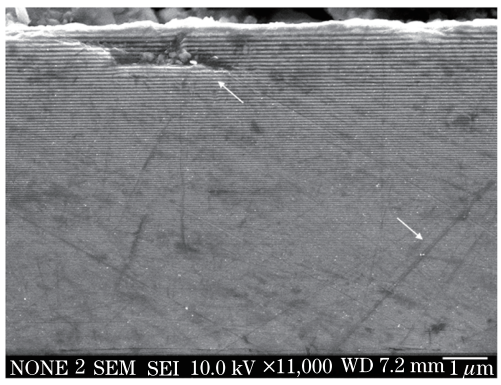

Fig. 4. SEM image of the multilayer cross-section obtained after the 0.5-μm diamond suspension polishing process.

(CMP) was used. CMP is a unique technique used in the semiconductor industry, which combines the advantages of both mechanical and chemical polishing. The working process of CMP consists of two stages. Firstly, the chemical slurry reacts with and weakens the surface materials; then, the mechanical forces produced by the nanometer abrasives and polishing cloth wipe the softened materials away, resulting in a precise removal at the atomic scale[15]. In the present experiment, the colloidal silica slurry, in conjunction with a velvet polishing cloth, was used.

The cross-section of the multilayer sample was roughly polished using slurry with a concentration of approximately 3% to remove the damage caused by diamond lapping quickly and to improve the flatness. A final polishing step was performed using slurry with a concentration of approximately 1% to improve the smoothness.

Both sides of the cross-section were thinned and polished using the same methods. However, when the depth of multilayer is less than 100 μm, extreme caution should be exercised to avoid breaking the ultra-thin flake. The depth was monitored during the thinning process using an optical microscope, which facilitated achievement of the desired value.

After thinning and polishing the cross-section of the depth-graded multilayer, the structure was observed by SEM. The SEM images of the cross-section after CMP are shown in Fig. 5. Figure 5(a) shows the entire cross-section of the multilayer, and Fig. 5(b) is an enlarged image of the area of the layers close to the substrate. The fine multilayer structure, including the 15-nm-thick layers near the substrate, is evidently undamaged, and all the interfaces remain flat and sharp after the entire process. No deformation or peeling of layers is observed. The spots indicated by the arrows in Fig. 5(a) are contaminants.

To evaluate the smoothness of the surface of the polished cross-section, atomic force microscopy (AFM) was used, and the result is shown in Fig. 6. Although contaminants are present, the root mean square (RMS) roughness across the entire surface is 0.33 nm. The depth of the MLL was measured using a stylus profiler, and the result shows a depth of approximately 6–7 μm. Thus, a MLL with a high aspect ratio of 400–466 has been fabricated. The deviation of the cross-section depth was mainly

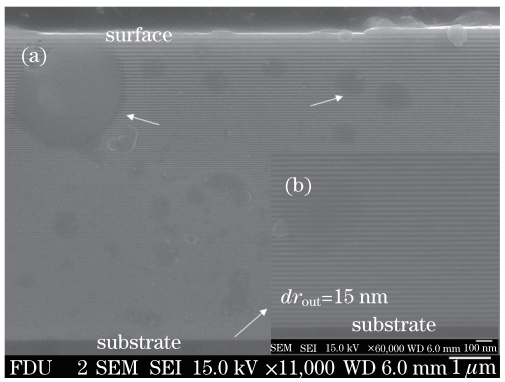

Fig. 5. SEM images of the multilayer cross-section after CMP showing (a) an image of the entire cross-section of multilayer and (b) an enlarged area of the layers close to the substrate. The spots indicated by arrows are contaminants.

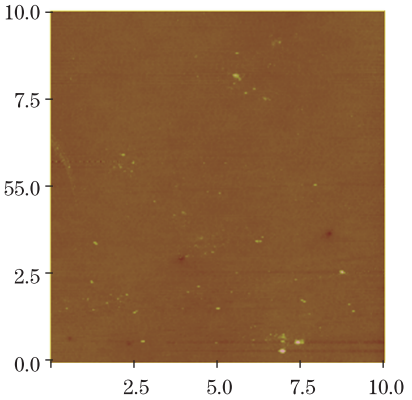

Fig. 6. AFM image of the polished cross-section. The measured dimension is 10×10 (μm), z range of the image is 25.85 nm, and the RMS roughness is 0.33 nm.

caused by the thinning process, which can be improved in future experiments.

A depth-graded WSi_2/Si multilayer is deposited, and the slicing, thinning, and polishing processes for the multilayer are explored to obtain a linear zone plate, MLL, for X-ray focusing applications. To achieve the ideal aspect ratio with minimal damage, the multilayer sample is sliced and thinned on both sides of the cross-section. To remove all the damages and improve the smoothness of the surface, chemical mechanical polishing is used as a final step. After all the repeated fabrication processes, the cross-section is measured using SEM and AFM. Results show that the multilayer structure has remained undamaged and that the surface roughness is only 0.33 nm.

The aspect ratio of the fabricated MLL is greater than 400, which is significantly larger than that of the zone plates fabricated using the traditional e-beam lithography method. The thinning and polishing processes, which include CMP, are evidently suitable for the fabrication of a MLL for hard X-ray focusing applications. The optical properties of the MLL will be studied in the succeeding experiments.

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