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Fabrication of high aspect ratio gratings for X-ray imaging*

HOU Shuang-yue (侯双月)[1], **XIONG Ying** (熊瑛)[1], **CHEN Shan** (陈珊)[1], **XIONG Peng-hui** (熊鹏辉)[1,2], **CHEN Xiang-yu** (陈翔宇)[2], **ZHANG Xiao-bo** (张晓波)[1], **TIAN Yang-chao** (田扬超)[1], **and LIU Gang** (刘刚)[1]**

1. National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230029, China

2. Department of Precision Machinery and Precision Instrumentation, University of Science and Technology of China, Hefei 230026, China

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Grating-based X-ray imaging system is an important tool to investigate the inner structure of thick samples. The key components of the system consist of three golden gratings. The high aspect ratio gratings are fabricated using the SU-8 material. Considering the grating linewidth broadening varies with exposure dose, the relationship between linewidth broadening and exposure dose is studied experimentally. A series of gratings with different periods and different duty cycles are designed by optimizing the linewidth and exposure dose. Finally, the gratings are successfully fabricated by combining UV lithography and electroplating.

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Since X-ray was discovered by Roentgen in 1895, X-ray-based image measurement has become one of the essential technologies, which is widely applied in medical diagnostics and nondestructive inspections. Conventional X-ray imaging based on the differences in absorption of sample constituents is commonly used to detect metal and other materials made of heavy elements. However, for some weak absorbers, such as soft biological tissues or polymers, the technique is too insensitive to obtain high-contrast absorption images. In order to overcome this, the X-ray phase contrast imaging is proposed[1]. Unlike conventional X-ray imaging based on attenuation property, the X-ray phase contrast imaging utilizes the characteristic of wave-particle dualism of X-ray. The absorption factors for materials consisting of low elements are three orders of magnitude smaller than their phase factors[2]. Therefore, the image contrast can be enhanced substantially by recording the phase shift of X-rays passing through the sample rather than only the absorption[3-9].

In 2006, Pfeiffer et al introduced an absorption grating near the X-ray tube[10]. The introduction of this grating makes the grating-based phase contrast imaging method be implemented in ordinary laboratories. The setup[11] with three gratings is shown in Fig.1. As the important optical device of the setup, the three gratings are essential to be fabricated. The fabrication of the three gratings is very difficult at present. A number of techniques have been developed to fabricate high aspect ratio gratings, including LIGA[12] and anisotropic wet etching[13]. In this letter, particular focus is put on the fabrication of high aspect ratio gratings with standard lithography and electroplating. The dimensions of gratings are controlled precisely by optimizing the process parameters.

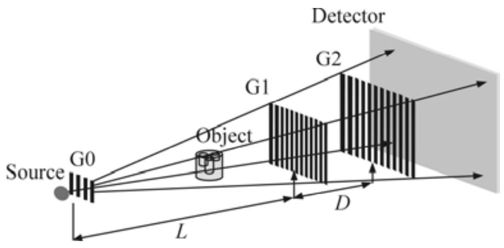

Fig.1 Schematic view of the grating-based X-ray imaging system with conventional X-ray tube

The grating-based methods mentioned above use three absorption gratings as the key optical elements. As the X-ray photon energy can be up to 50 keV during the experiment process, it is essential to fabricate large-area absorption gratings with high aspect ratio to provide sufficient image contrast. Since state-of-the-art absorption materials used for absorption gratings rely on gold, the main requirement of the three gratings is a sufficient height of the gold absorber to absorb the X-ray. It can be calculated from the X-ray optical constants of gold that a thickness of over 200 μm is needed to acquire a high

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** E-mail:liugang@ustc.edu.cn

X-ray absorptivity. At present, the fabricating process for the high aspect ratio gratings is a great challenge because of the surface tension of the drying liquid which can bring about pattern collapse during the experiment[14-16]. Fig.2 shows the optical microscopy image of gratings which are supported by the top-plate structure partly. It can be seen that high aspect ratio gratings without a top-plate structure would stick together while the gratings with a top-plate structure stand still. As a consequence, it is necessary to manufacture a top-plate structure to stiffen the overall structure and prevent stiction in HARS. This top-plate structure can be easily realized through a low-dose UV exposure.

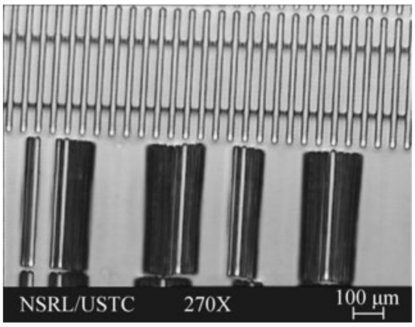


Fig.2 The upper half of the optical microscopy image shows gratings with a top-plate structure while the lower half shows gratings without a top-plate structure

The individual fabrication process is illustrated in Fig.3. The standard photo lithography process of a negative photoresist for the pattern definition is employed. The grating with a period of 60 μm and a duty cycle of 0.5 is used as an example to explain the fabrication process. An SU-8 structure is fabricated using a silicon wafer polished as the substrate. About 5 nm of titanium and 30 nm of gold are sputtered as the conducting layer. A 250 μm layer of SU-8 photoresist is spin-coated on silicon substrate. After heat treatment and sufficient cooling, the photoresist is exposed with suitable UV dose about 300 mJ/cm^2 under a mask with appropriate grating dimension first. Subsequently, a low-dose UV exposure of 30 mJ/cm^2 is used in the same photoresist material in which the gratings are fabricated. A top-plate support structure with the shallow depth of 60 μm is formed. After post exposure bake, the unexposed part is removed by the developer. To obtain the gold absorption structures, the photoresist pattern should be electroplated in gold potassium citrate solution with a current density of 0.5 A/dm^2 at 50 ℃. The photoresist can be stripped in a remover made of N-methyl-2-pyrrolidone although it is not required from the point of the optical performance, since the SU-8 photoresist is transparent to X-rays.

In order to satisfy the condition of the X-ray imaging, the requirements for each absorption grating on the duty cycle and period are quite different. Thereby grating sizes are required to be controlled precisely. The linewidth of grating structure resulting from photolithography changes with the exposure dose, so it is important to investigate the relationship between the surface linewidth broadening and exposure dose. A variety of gratings with different periods from 20 μm to 100 μm and different duty cycles are designed during the facbrication process. By calculating the average and standard deviation of the linewidth broadening values under different exposure doses, the linewidth broadening (seen in Fig.4) along with the change of the exposure dose can be obtained.

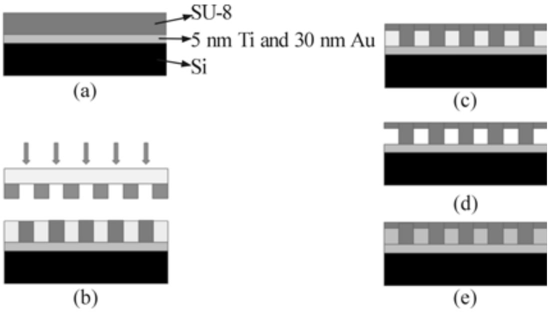
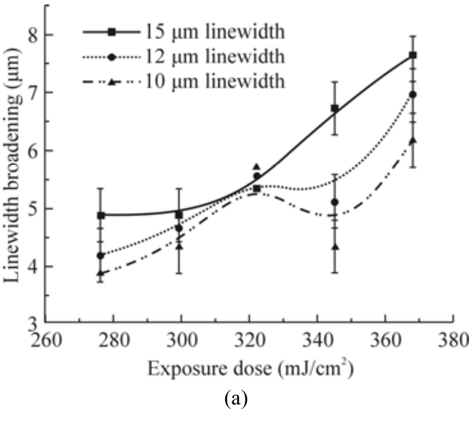
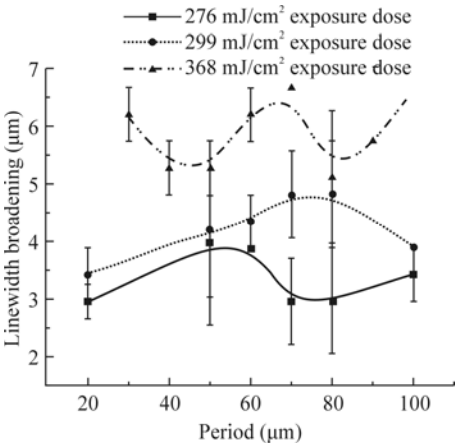


Fig.3 Fabrication process used to manufacture the absorption grating: (a) Original structure; (b) UV exposure through a mask with a suitable size; (c) Low-dose UV exposure using a mask which links the structures exposed in step (b); (d) Development; (e) Electroplating

A graph of average linewidth broadening as a function of exposure dose is shown in Fig.4(a), which shows the overall trend of the linewidth broadening increases with exposure dose. From Fig.4(b), it can be seen that the same grating linewidth will broaden a similar value under the same exposure dose even if the periods and duty cycles of gratings are different. The grating sizes can be controlled precisely by optimizing the linewidth and the UV exposure dose. In this experiment, the exposure dose about 300 mJ/cm^2 and the feature size of 25 μm of the mask are selected to fabricate the grating with a linewidth of 30 μm. The gold grating structure (shown in Fig.5) with a duty cycle of 0.5 is fabricated successfully.



(a)



(b)

Fig.4 (a) Linewidth broadening as a function of UV exposure dose with the period of the grating of 60 μm for different linewidths; (b) Linewidth broadening as a function of grating period with the linewidth of 10 μm for different exposure doses

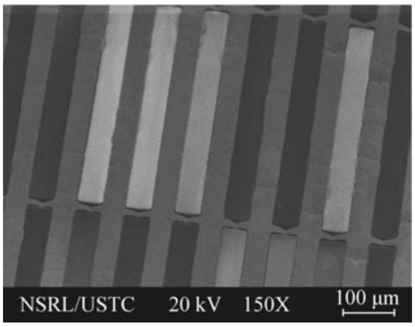


Fig.5 SEM image of the gold grating structure with a duty cycle of 0.5

The process parameters of SU-8 photoresist are optimized by investigating the relationship between the grating linewidth broadening and the exposure dose. It indicates that the linewidth of gratings can be controlled precisely. Moreover, it is probable to fabricate high aspect ratio gratings with a support structure at the top of the gratings, which is used to prevent the stiction. Finally, the gold grating structures are successfully fabricated using the standard lithography and the electroplating technology.

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