COATING REMOVAL OF SILICON-BASED MIRROR IN SYNCHROTRON RADIATION BY SOLUBLE UNDERLAYER

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Abstract

Multilayer optics is widely used for the x-ray beam monochromatization, focusing, and collimation in synchrotron light source. However, the multilayer coatings might be damaged by the high heat loads, the poor film adhesion, the high internal stress, or the inadequate vacuum conditions. As a result, it is essential to develop a method to make the optical substrate reusable without compromising its quality. In our published work, we successfully prepared a W/B_4C multilayer coating with a 2 nm Cr buffer layer on a small-sized Si wafer. The coating was stripped from the Si substrate by dissolving the Cr buffer layer using an etchant. After the etching process, the sample's roughness was comparable to that of a brand-new substrate. We have since utilized this method to clean the multilayers on the surface of a 20 cm × 5 cm silicon-based mirror for High Energy Photon Source (HEPS). The surface roughness and shape were measured, and they reached the level of a brand-new mirror.

INTRODUCTION

The surface of the mirror is coated with a single or multilayer coating of different materials, so that the mirror has high reflectivity or spectral selectivity. Monocrystalline silicon is an ideal substrate for synchrotron radiation optics due to its low density, high mechanical strength, and good thermal stability. Silicon substrates are typically polished to a roughness of only a few angstroms and have an excellent surface shape before being coated with a single or multilayer coating. After long-term service, the coating will deteriorate or even fail due to contamination, mishandling, instantaneous temperature changes, poor adhesion between the coating and the substrate, and high internal stress of the film. The optics need to be updated after a period of service. Therefore, there is a need to study ways to remove optical films to make the expensive high-precision Si substrates reusable.

There are many ways to remove films, such as liquid etching, vapor etching, laser etching, and soluble underlayers [1]. In the preparation of optics with coating in synchrotron radiation, researchers usually prepare a Cr buffer layer on the substrate, and then prepare various optical thin films to reduce the stress of the film and enhance the adhesion force. Therefore, for synchrotron radiation optics, there is an inherent advantage to using the method of soluble underlayers to remove the film. In our published work in Optics Express [2], we successfully prepared a W/B_4C multilayer coating with a 2 nm Cr buffer layer on a small-sized (2 cm × 1 cm) Si wafer. As is shown in Fig. 1, the coating was stripped from the Si substrate by dissolving the Cr buffer layer using an etchant. After the etching process, the sample's roughness was comparable to that of a brand-new substrate. The W/B_4C multilayer coatings with a Cr buffer layer were recoated on the etched samples, and the results of X-ray reflection (XRR) show that the interface roughness was not damaged by the etching process.

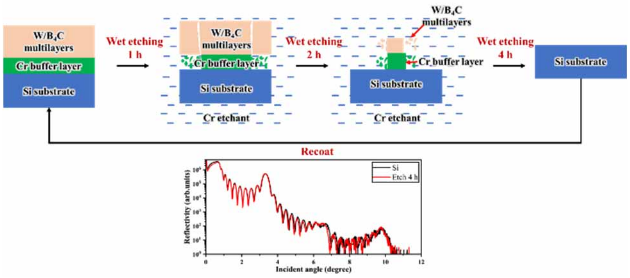

Figure 1: Schematic diagram and XRR results of a refurbished coated silicon wafer with a Cr buffer layer.

The optics used in synchrotron radiation are usually large-size silicon stripes, it is necessary to investigate the applicability of the method of soluble underlayers for refurbishing large-size silicon strips. We have since utilized this method to clean the multilayers on the surface of a 20 cm × 5 cm silicon-based mirror for High Energy Photon Source (HEPS), and the surface roughness and shape at different stages were measured.

EXPERIMENTAL

Films were deposited on the surface of a 20 cm × 5 cm silicon-based mirror by magnetron sputtering at the Platform of Advanced Photon Source Technology R&D (PAPS) in Huairou, Beijing. The deposition parameters of the Cr buffer layer and W/B_4C multilayer film and the etching process were the same as those previously applied on small-sized wafers. To compare the effects of the etching process on different coatings, Pt/ B_4C multilayer film with a Cr buffer layer were also deposited in different areas of the same silicon strip. A mask was used to allow the film to be deposited in a designated area of the silicon stripe. The deposition parameters of the Cr buffer layer in both coatings are the same. The surface roughness was investigated using a non-contact 3D optical surface profiler. The optical figure was measured by Long Trace Profiler (LTP).

RESULTS AND DISCUSSION

Figure 2 is the image of the coated silicon stripe. The W/B_4C multilayer coating was intact, while the surface of the Pt/B_4C multilayer coating was crazing, which might be caused by the high internal stress in the film. Figure 3 shows the etching process. Figure 3(a) shows that the

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middle area of Pt/B$_4$C multilayer coating, which originally had many cracks, quickly fell off after two minutes etching, while at the same time, the W/B$_4$C multilayer coating was still intact. After 16 h static etching (Fig. 3(b)), the W/B$_4$C multilayer coating peeled off completely, while the Pt/B$_4$C multilayer coating changed little compared to that in Fig. 3(a). After wiping with absorbent cotton for two minutes, the Pt/B$_4$C multilayer coating peeled off completely quickly. This indicates that different coatings require different etching conditions, static etching is sufficient for the W/B$_4$C multilayer coating, and for Pt/B$_4$C multilayer coatings, the etchant diffused from the edge is not enough to complete the etching, it is difficult for the etchant to pass through the Pt//B$_4$C multilayer coating, and it is necessary to add stirring to create a dynamic etching environment to accelerate the diffusion of etchant.

Figure 2: Si stripe after coating.

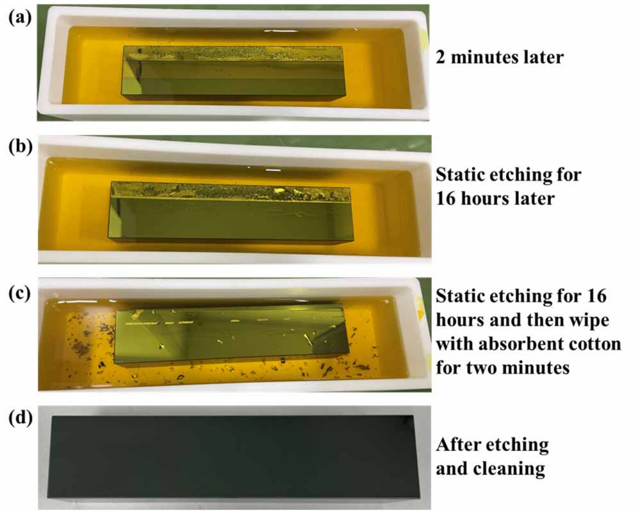

Figure 3: The etching process.

Figures 4 and 5 show the results of the area of W/B$_4$C multilayer coating measured by 3D optical surface profiler and LTP, respectively. The roughness of eight points was randomly taken to take the average value, and the results are shown in Fig. 6. The standard deviation (SD) of roughness at each stage is also plotted in Fig. 6. Figure 4 (c) shows that the etched surface is homogeneous and free of obvious defects. This is also indicated by the SD in Fig. 6. The specific values of roughness and figure of different stages obtained by 3D optical surface profiler and LTP are listed in Table 1. The roughness and figure of the etched surface do not change much compared to the original surface. Dissolving the buffer layer under static condition can well remove the W/B$_4$C multilayer coating on the surface of large-size silicon strips.

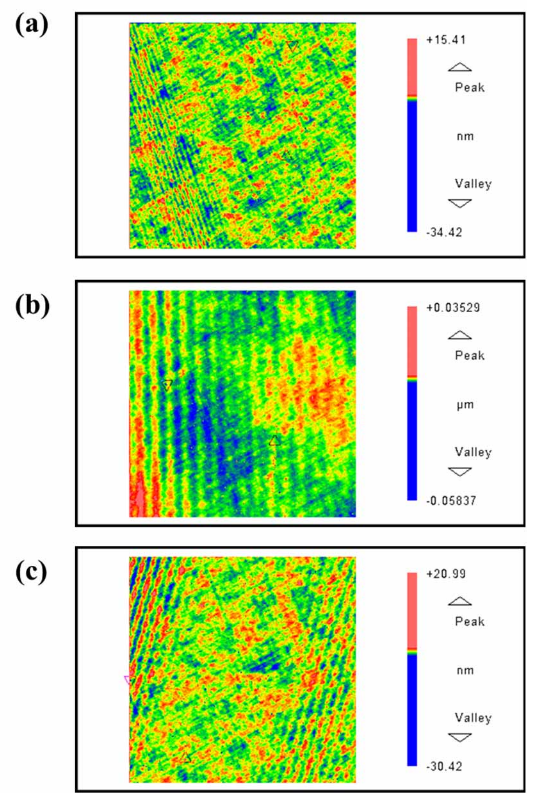

Figure 4: Surface morphology of samples measured by 3D optical surface profiler: (a) Si stripe, (b) after coating and (c) after etching.

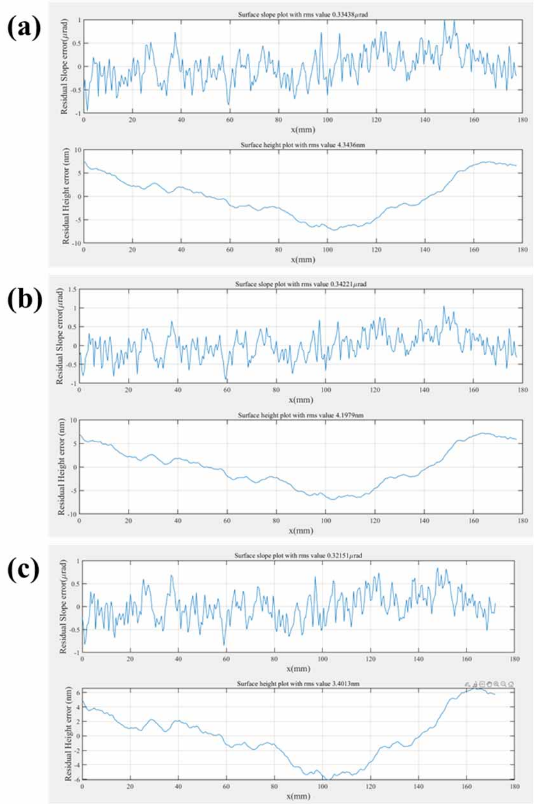

Figure 5: Results measured by LTP: (a) Si stripe, (b) after coating and (c) after etching.

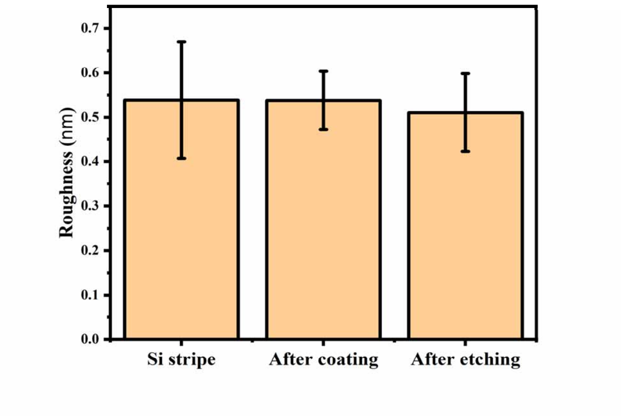

Figure 6: The surface roughness of the Si stripe at different stages.

Table 1: Results Obtained by 3D Optical Surface Profiler and LTP

Silicon-based mirror	Roughness (nm)		Surface slope rms (μrad)
	Average	SD	
Silicon stripe	0.54	0.13	0.33
After coating	0.54	0.07	0.34
After etching	0.51	0.09	0.32

CONCLUSION

Multilayer coatings on large-size Si mirror could be removed by dissolving the Cr buffer layer. Different multilayer coatings need different etching condition. For Pt/B_4C multilayer coatings, it is necessary to add stirring to create a dynamic etching environment to accelerate the diffusion of etchant. Static etching is sufficient for W/B_4C multilayer coatings, and the surface roughness and shape could reach the level of a brand-new mirror by dissolving the Cr buffer layer using an etchant.

ACKNOWLEDGEMENT

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