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Design and fabrication of highly heat-resistant Mo/Si multilayer soft X-ray mirrors with interleaved barrier layers

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Introducing interleaved carbon barrier layers improves the heat-resistance of Mo/Si multilayers. The soft X-ray reflectivities of the multilayers were calculated, and the effects of heating on both the reflectivities and layer structures of Mo/Si multilayers with and without barrier layers were investigated using X-ray diffraction and transmission electron microscopy. The results show that, for applications using intense soft X-ray beams, Mo/Si multilayers with interleaved carbon barrier layers are better mirrors than Mo/Si multilayers because they have much better heat resistance and almost the same soft X-ray reflectivity as the Mo/Si multilayers.

Keywords: X-ray mirrors; multilayers; heat resistance; barrier layers; Mo/C/Si/C.

1. Introduction

The Mo/Si multilayer soft X-ray mirror is widely used in many X-ray applications, such as monochromators, thermal filters and bandpass filters for synchrotron radiation, and mirrors in X-ray lasers, because of its spectral selectivity and high normal-incidence reflectivity. The use of intense X-rays in these applications makes heat resistance an important factor in ensuring reliable performance (Ziegler *et al.*, 1989; Kortright & DiGennaro, 1989). However, since the multilayer mirrors are inherently metastable material composites with nanometre-order periodicities, they are not stable in high-temperature environments. In particular, Mo/Si multilayer mirrors do not have good inherent heat resistance because large amounts of interdiffusion easily occur between the Mo and Si layers (Holloway *et al.*, 1989; Stearns *et al.*, 1990; Rosen *et al.*, 1993; Takenaka, Kawamura, Ishii *et al.*, 1995). Therefore, the reflectivity and periodic length of this multilayer mirror are easily reduced even under a low heat load. We tried to fabricate Mo/Si multilayers with interleaved carbon (C) thin barrier layers to improve the heat resistance. This structure is similar to that of the Si/W/Si/C multilayer reported by Ziegler *et al.* (1989).

2. Design of Mo/Si multilayers with barrier layers

We calculated the X-ray reflectivities of Mo/Si multilayers with C barrier layers, and experimentally investigated the effects of heating on both the reflectivity and the layer structures of a Mo/Si multilayer and several Mo/Si multilayers with interleaved C barrier layers of differing thicknesses. Fig. 1 shows the calculated reflectivities of both types of multilayer, each having 40 layer pairs, at wavelengths of around 13 nm at near-normal incidence, assuming that the multilayers have ideal structures. The calculations were performed using the Fresnel equation and Henke's optical data (Henke *et al.*, 1988). In these multilayers, the periodic length was 7 nm and the thickness ratio between the Mo layer and the light-element layer was 0.4:0.6. The calculated peak reflectivity of the Mo/Si multilayer was 72.6% and that of the Mo/C/Si/C multilayer, with a 0.5 nm-thick barrier layer, was around 68.9%. The reflectivity of the Mo/C/Si/C multilayer decreased with increasing C barrier-layer thickness.

3. Sample fabrication and heat treatment

An r.f. magnetron sputtering deposition system was used to fabricate the Mo/Si multilayers and Mo/C/Si/C multilayers (Takenaka, Kawamura, Haga *et al.*, 1995). The thickness ratio of the Mo layer to the Si layer (or Si layer and two C barrier layers) was between 0.35 and 0.65. There were 40 layer pairs. The thickness of the barrier layer of the deposited Mo/C/Si/C multilayer was sub-nanometre in our experiment. The characteristics of as-deposited Mo/Si multilayers with and without barrier layers are given in Table 1.

The samples were then heated in an IR furnace for 1 h at various temperatures up to 1073 K in an Ar atmosphere. The gas flow rate was 200 cm^3 min^{-1}. The heating temperature was monitored by three thermocouples in contact with the sample surface; the monitored temperature was accurate to within ±5 K below 1073 K. The samples were heated to the desired temperature in 10 min. After being held at the desired temperature for 1 h, they were cooled to room temperature by a 200 cm^3 min^{-1} room-temperature Ar gas flow.

4. Characterization by X-ray diffraction

The Cu $K\alpha$ first-order Bragg-peak reflectivities of these multilayers were measured at room temperature after heating at

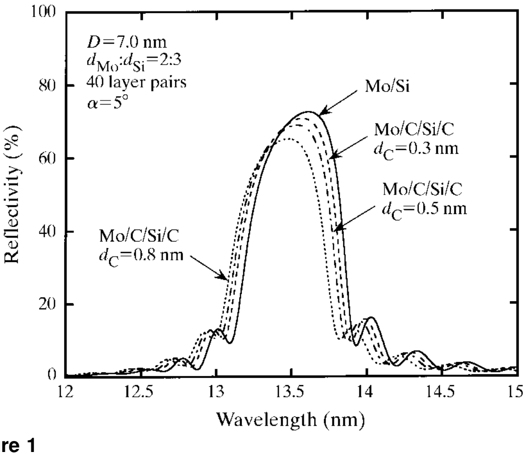

Figure 1
Calculated reflectivities of Mo/Si and Mo/C/Si/C multilayers at wavelengths around 13 nm at near-normal incidence.

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Table 1

Characteristics of as-deposited Mo/Si multilayers with and without C barrier layers.

Number of layer pairs is 40; d_{Mo} = thickness of Mo layer *etc*; D = periodic length.

Materials	d_{Mo}	d_{C}	d_{Si}	d_{C}	D
Mo/Si	2.8 nm	–	4.2 nm	–	7.0 nm
Mo/C/Si/C	2.1 nm	0.2 nm	4.4 nm	0.3 nm	7.1 nm
Mo/C/Si/C	2.3 nm	0.5 nm	3.5 nm	0.5 nm	6.8 nm

various temperatures. The reflectivities were measured at a spot near the centre of the multilayers. Fig. 2 shows the change in the reflectivity *versus* the heating temperature and the periodic lengths of these multilayers after heating at various temperatures. The reflectivities and periodic lengths have been normalized by the initial reflectivities and periodic lengths of as-deposited multilayer samples.

The reflectivities of Mo/Si multilayers with about 7 nm periodic lengths markedly decreased after heating above 573 K, falling after heating at 673 K to about 60% of the value for the as-deposited multilayers. On the other hand, the change in reflectivity of the Mo/C/Si/C multilayer, with 0.3 and 0.5 nm-thick C barrier layers, was much less. The reflectivity remained over 80% of the value of the as-deposited multilayer even after heating at 873 K.

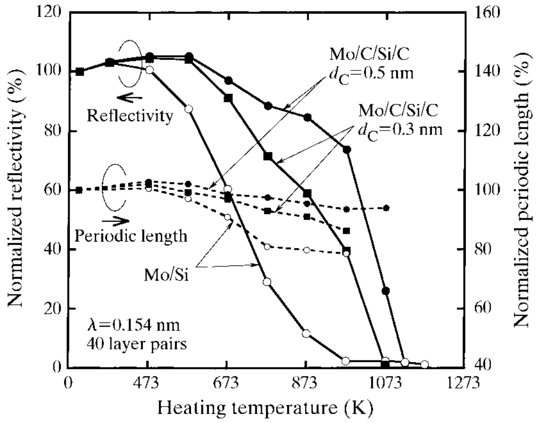
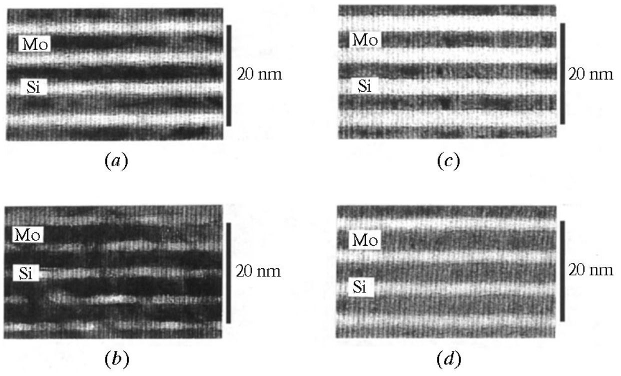


Figure 2

Relationship between normalized Cu $K\alpha$ first-order Bragg-peak reflectivity and relationship between normalized periodic lengths of Mo/Si and Mo/C/Si/C multilayers and heating temperature.



(*a*) (*c*)

(*b*) (*d*)

Figure 3

TEM cross sections for (*a*) as-deposited Mo/Si, (*b*) 873 K-heated Mo/Si, (*c*) as-deposited Mo/C/Si/C and (*d*) 873 K-heated Mo/C/Si/C.

The periodic lengths of both Mo/Si multilayers decreased as the heating temperature increased to 673 K, falling to about 90% of the value of the as-deposited multilayer. On the other hand, that of the Mo/C/Si/C multilayer changed only slightly as the heating temperature increased. Heating between room temperature and around 573 K induced a slight increase in reflectivity and periodic length, though the reason for this is unclear. However, there is the possibility that these phenomena are due to decreasing interface roughness and the evolution of periodic length in this heating temperature range.

5. Characterization by TEM observation

Figs. 3(*a*) and 3(*b*) show TEM cross sections for as-deposited and 873 K-heated Mo/Si multilayers. The as-deposited Mo/Si multilayer formed fairly well with interdiffusion layer structures between the Mo and Si layers. The 873 K-heated Mo/Si multilayer had larger interdiffusion layer structures that were crystallized and aggregated, promoting the development of interface roughness. In addition, with 973 K heating, the Mo/Si multilayer crystallized further, promoting agglomerations of Mo or Mo

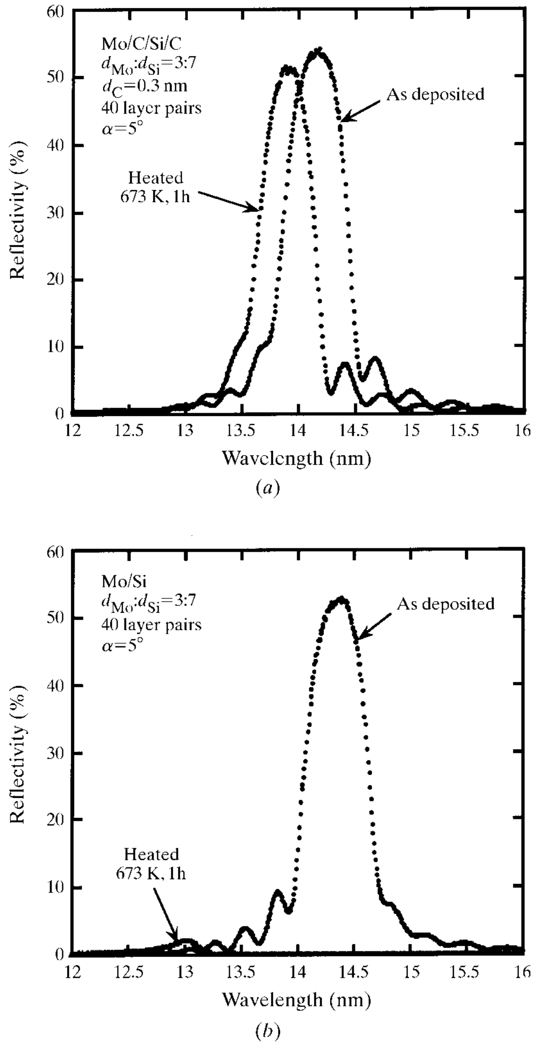


Figure 4

(*a*) Measured soft X-ray reflectivities for the as-deposited and 673 K-heated Mo/C/Si/C multilayers. (*b*) Measured soft X-ray reflectivities for the as-deposited and 673 K-heated Mo/Si multilayers.

silicide and Si. In this heated multilayer, large grains were observed. In contrast, the Mo/C/Si/C multilayer with 0.5 nm-thick C layers had well defined discrete multilayer structures for the as-deposited (Fig. 3*c*), 873 K-heated (Fig. 3*d*) and 973 K-heated samples (not shown).

6. Soft X-ray reflectivities

Fig. 4(*a*) shows the measured soft X-ray reflectivities for the as-deposited and 673 K-heated Mo/C/Si/C multilayers with 0.3 nm-thick C layers. The peak reflectivity of the as-deposited Mo/C/Si/C multilayer was 54.0% at a wavelength of 14.2 nm and only decreased to 51.3% with a slight decrease in the peak wavelength (13.9 nm) with 673 K heating. Fig. 4(*b*) shows the reflectivities for the as-deposited and 673 K-heated Mo/Si multilayers. The peak reflectivity of the as-deposited Mo/Si multilayer was 52.8% at a wavelength of 14.4 nm. However, it dropped to 2.0% with a large decrease in the peak wavelength (13.0 nm) with 673 K heating. It is clear that the changes in reflectivity and the wavelength position of the peak of the Mo/C/Si/C multilayer with 0.3 nm-thick C barrier layers were much smaller than those of the Mo/Si multilayer after heating at 673 K.

7. Conclusions

These results suggest that, for applications using intense soft X-ray beams, Mo/Si multilayers with interleaved C barrier layers are better mirrors than simple Mo/Si multilayers, because they have much better heat resistance and almost the same reflectivity in as-deposited structures.

References

Henke, B. L., Davis, J. C., Gullikson, E. M. & Perera, R. C. C. (1988). LBL-26259, UC-411. LBL, CA, USA.

Holloway, K., Do, K. B. & Sinclair, R. (1989). *J. Appl. Phys.* **65**, 474–480.

Kortright, J. B. & DiGennaro, R. S. (1989). *Rev. Sci. Instrum.* **60**, 1995–1998.

Rosen, R. S., Stearns, D. G., Viliardos, M. A., Kassner, M. E., Vernon, S. P. & Cheng, Y. (1993). *Appl. Opt.* **32**, 6975–6980.

Stearns, D. G., Stearns, M. B., Cheng, Y., Stith, J. H. & Ceglio, N. M. (1990). *J. Appl. Phys.* **67**, 2415–2427.

Takenaka, H., Kawamura, T., Haga, T., Kinoshita, H. & Ishii. Y. (1995). *Jpn. J. Appl. Phys.* **34**, 5027–5031.

Takenaka, H., Kawamura, T., Ishii, Y. & Asagiri, S. (1995). *J. Appl. Phys.* **78**, 5227–5230.

Ziegler, E., Lepetre, Y., Joksch, S., Saile, V., Mourikis, S., Viccaro, P. J., Rolland, G. & Laugier, F. (1989). *Rev. Sci. Instrum.* **60**, 1999–2002.