

# Thermal Stability of Cr/Sc-based Soft X-ray Multilayer Mirrors

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## ABSTRACT

Reflective multilayer optical elements may be subject to elevated temperatures in their application as elements in instrumentation based on intense synchrotron light, free-electron laser, or laser plasma sources. Moreover, at-wavelength characterization, usually performed with synchrotron radiation, may impose a temperature rise of the sample by hundreds of degrees. Thus, if the optical element is temperature sensitive, the reflective performance may change during use or during characterization, influencing the measurement being carried out.

Therefore, in this work, the thermal stability of Cr/Sc-based multilayer X-ray mirrors has been investigated. The interdiffusion process has been studied quantitatively by monitoring the evolution of low-angle Bragg peak intensities in hard X-ray reflectivity and diffuse scattering measurements.

Cr/Sc multilayers with multilayer periods ranging from  $\Lambda=0.8-6.4$  nm, layer thickness ratios  $\Gamma$  between 0.2-0.8 and number of periods from  $N=50$  to  $N=300$  have been deposited by dual-target unbalanced magnetron sputtering onto Si substrates. The substrate was maintained at an ambient growth temperature ( $<50^\circ\text{C}$ ), and ion-energy assistance was applied during deposition in order to produce optimally smooth and sharp interfaces, thereby leading to maximum performance. This deposition technique routinely provides near-normal incidence reflectivities in excess of 20% at the Sc absorption edge at  $\lambda=3.115$  nm.<sup>2</sup> In addition to these pure metallic Cr/Sc multilayers, also multilayers with N or B<sub>4</sub>C interdispersed in the structure have been produced using the same deposition technique. State-of-the-art Cr/Sc multilayers, using intralayers of B<sub>4</sub>C on top of the Cr layer,<sup>3</sup> have also been investigated.

Isothermal annealing was performed on all samples for temperatures up to 850°C. The temperature was increased in steps of 50°C and kept for ~12 hours, while *in-situ* X-ray reflectivity and diffuse scattering measurements were continuously recorded. For pure metallic Cr/Sc multilayers the Bragg peak intensity decreased exponentially with annealing time at each temperature, in accordance with linear diffusion theory. The intensity decrease corresponds to activation energies of  $0.5\pm 0.1$  eV for interface broadening, and the multilayers were stable up to 350°C. After further annealing up to 600°C the multilayer structure was completely destroyed.

For the other Cr/Sc-based multilayers a non-linear diffusion behaviour was observed, implying that interdiffusion of these multilayers can not be described by ordinary diffusion couple theory. For the nitrided multilayers even a slight increase in the peak intensity was found for annealing temperatures up to 400°C. All Cr/Sc multilayers containing N or B<sub>4</sub>C showed an improved thermal stability up to 450°C, and B<sub>4</sub>C-containing multilayers stayed amorphous even after annealing up to 650°C according to electron diffraction. The microstructural changes during the annealing process, as investigated using X-ray diffraction, cross-sectional high-resolution transmission electron microscopy (HR-TEM), scanning TEM (STEM) and electron diffraction, is also presented.

## References

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