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Quantitative study of the transition layers in Mo/Si multilayers from the analysis of the Si Kβ x-ray emission band

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The multilayer interferential mirrors consist of alternate layers of heavy (Mo, W, Ni…) and light (Si, C, B…) materials. Among them, the Mo/Si multilayers are useful optical components in the X-UV photon energy region. They are also used as high-reflectivity mirrors, polarizers, beam splitters… Moreover, they are expected to be applied in soft X-ray microscopes, interferometers and projection lithography. The optical properties of these compounds depend on the quality of their interfaces. Indeed, it is known that interdiffusion occurs between the Mo and Si layers, leading to the formation of an interlayer at each interface. The aim of this study is to determine the nature and the thickness of the compounds susceptible to form at interfaces. In this objective, we have analyzed the Si Kβ x-ray emission band which is sensitive to the physico-chemical state of the Si atoms.
The soft X-ray emission spectroscopy induced by electrons is especially suited to study buried layers and interfaces nondestructively. It gives both qualitative (electronic stucture) and quantitative (concentration of the elements) informations. When looking at an emission band, this technique enables to probe the occupied valence states. Because these states involve weakly bound electrons, the emission band is very sensitive to the physico-chemical environment : nature, number and arrangement of the neighbouring atoms. Thus, XES both enables to identify and to quantify the compounds within the multilayer in order to determine the thickness of the interfaces.

We present study of a Mo/Si multilayer upon annealing from 200°C to 600°C. It was prepared using a magnetron sputtering system and consists of 40 bilayers of Mo/Si with d_{Mo} = 2.92 nm and d_{Si} = 4.04 nm. The studied emission band is the Si Kβ emission (3p → 1s transition) which describes the Si 3p density of states. It is analyzed with a high-resolution bent-crystal x-ray spectrometer [1].
The Si 3p density of states of the compounds susceptible to be present near the interfaces are shown in Fig.1 : amorphous silicon, $MoSi_2$ and Mo_5Si_3. The superimposition of the spectral densities of the multilayer unannnealed and annealed from 300°C to 600°C are presented in Fig.2. When the annealing temperature increases up to 500°C, the emission band broadens towards the low photon energies, where are present the maximum of Mo_5Si_3 and the shoulder of MoSi2. At 600°C, the spectrum shape tends towards that of $MoSi_2$. This shows the presence of silicides at the interfaces, whose proportion increases with the annealing temperature.
The emission band of the multilayer is fitted by a weighted sum of the reference compound spectra. As an example, we present in Fig.3 the fit for the unannealed Mo/Si sample. From the contribution of the silicides, the density of silicon in the reference compounds and the number of deposited atoms, the thickness of the transition layer is determined from a diffusion model [2,3]. They are presented in the Table.1 for all the studied multilayers. It is observed that the $MoSi_2$ contibution and thus, interlayer thickness increase with the annealing temperature.

From the study of the valence band, we can determine the interfacial compounds in the multilayers. From the relative intensity contribution of each compound we have calculated the thicknesses of the interlayers between the Mo and Si layers. These results are in agreement with x-ray reflectivity measurements showing a decrease of the reflecting power with the annealing temperature.

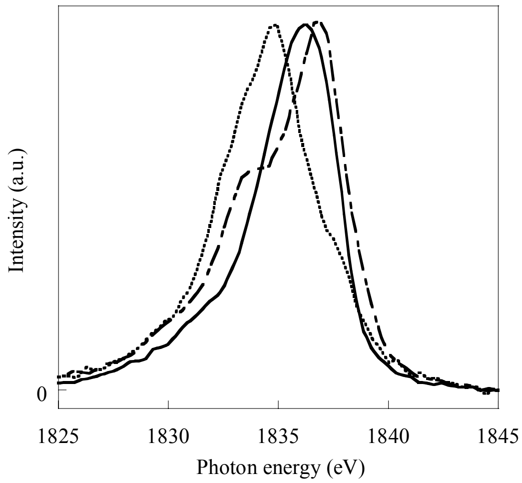

Fig.1 : Si Kβ emission bands of the reference compounds : a-Si (solid line), $MoSi_2$ (dashed line) and Mo_5Si_3 (dotted line).

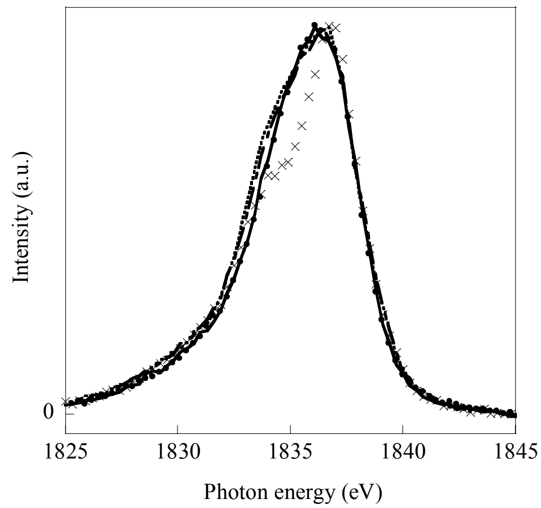

Fig 2 : Si Kβ emission bands of the unannealed Mo/Si sample (dots) and after annealing at 300°C (solid line), 400°C (dashed line), 500°C (dotted line) and 600°C (crosses).

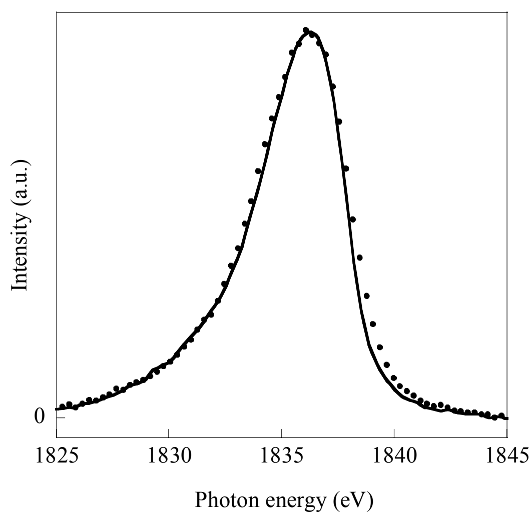

Fig.3 : Fit of the spectrum of the unannealed Mo/Si sample (dots) by a weighted sum of the spectra of the reference compounds (line) 78% a-Si + 14% $MoSi_2$ + 8% Mo_5Si_3.

Table 1 : Contribution of the silicides to the fit of the Mo/Si samples and thickness of the transition layer.

sample	unannealed	annealed 300°C	annealed 400°C	annealed 500°C	annealed 600°C
silicide contribution (%)	22	38	43	58	87
transition layer thickness (nm)	0.4	0.6	1.0	1.5	3.1

[1] P. Jonnard, in these proceedings.
[2] N. Miyata et al., Jpn. J. Appl. Phys. **38**, 6476 (1999).
[3] P. Jonnard et al., J. Phys. IV **118**, 231 (2004).