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To cite this version:

H. Maury, Philippe Jonnard, K. Le Guen, J.-M. André, Z. Wang, et al.. Thermal cycles, interface chemistry and optical performance of Mg/SiC multilayers. The European Physical Journal B: Condensed Matter and Complex Systems, 2008, 64 (2), pp.193-199. hal-00256010

HAL Id: hal-00256010

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EPJ manuscript No.
(will be inserted by the editor)

Thermal cycles, interface chemistry and optical performance of Mg/SiC multilayers

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Received: February 4, 2008/ Revised version: date

Abstract. The interplay between optical performance and the thermally activated interface chemistry of periodic Mg/SiC multilayers designed for application at 30.4 nm are investigated by optical (hard x-ray, soft x-ray and ultraviolet ranges, i.e. from 0.154 to 30.4 nm) reflectivity and x-ray emission spectroscopy. The multilayers are prepared by magnetron sputtering and then annealed up to a temperature of 500°C. Two clear changes take place in the multilayer upon annealing. At first, between 200 and 300°C a strong decrease of the reflectivity is observed, due to the development of interfacial roughness following the crystallization of the Mg layers. No interfacial compound is detected. Then, between 350 and 400°C there is formation of the Mg_2Si magnesium silicide at the interfaces following the reaction between the Mg and SiC layers. This also leads to the almost total loss of reflectivity of the multilayer. Thus, this kind of multilayer is thermally stable only for application requiring no heating above 200°C.

PACS. 68.65.Ac Multilayers – 61.05.cm X-ray reflectometry (surfaces, interfaces, films) – 78.70.En X-ray emission spectra and fluorescence – 73.90.+f Other topics in electronic structure and electrical properties of surfaces, interfaces, thin films, and low-dimensional structures – 66.30.Ny Chemical interdiffusion; diffusion barriers – 68.35.Fx Diffusion; interface formation

1 Introduction

Periodic multilayers consisting of an alternating and periodic succession of optically dense and less dense films are widely used nowadays as diffractive optical elements in the X-UV range [1]. For the 30 nm region, the Mo/Si system has been commonly employed because of its high time and thermal stability [2,3]. Its reflectivity ranges from 15 to 20% in the 25-35 nm spectral range. However for applications like solar imaging or photoemission microscopy using the He II line (30.4 nm, 40.7 eV) multilayers with high reflecting power are needed. Thus Si/Mo_2C, Si/B_4C, Si/C and Si/SiC multilayers have been developed for this spectral range [4]. The Si/B_4C system features a good reflectivity but poor temperature and time stabilities, while Si/SiC was found inadequate due to a measured reflectivity of only 16%. It has been proposed to replace silicon by magnesium in multilayers because of the low absorption coefficient of the Mg atoms after the L absorption edge at 25 nm (50 eV). Ejima *et al.* have proposed Mg/SiC multilayers and measured a reflectivity of 20% at 30.4 nm [5]. More recently, Takenaka et al. have obtained a reflectivity as high as 40% with the Mg/SiC multilayer at the same wavelength [6].

Because of its recent development, the Mg/SiC system has been little characterized. For example, with respect to Mo or Si, Mg is highly reactive and has a relatively low melting point (649°C) [7–10]. These properties could lead to a difficulty in controling the time and thermal stability of the Mg/SiC multilayers, despite their high reflectivity. It is thus important to investigate the phenomena that take place at the interfaces and limit the performances of the system, in order to understand how interdiffusion and roughness develop as a function of the annealing. It may then be possible to obtain knowledge-based Mg/SiC structures both optimized for thermal and time stabilities.

In this paper, we present the study of periodic Mg/SiC multilayers prepared by magnetron sputtering and designed for application at 30.4 nm (40.7 eV). They are analysed by a combination of non-destructive techniques [11–14]: x-ray reflectivity (XRR) in the hard and soft x-ray ranges, UV reflectivity (UVR) and x-ray emission spectroscopy (XES), in order to obtain structural and chemical informations about the Mg/SiC stack together with the monitoring of the optical performances in the working range. The study is performed for the sample obtained by magnetron sputtering and for samples annealed at temperatures up to 500°C.

2 Experimental details

The Mg/SiC multilayers, consisting of 30 bilayers, were deposited onto silicon substrates by an ultrahigh vacuum direct current magnetron sputtering system with four 100-mm-diameter sputtering sources (JGP560C, made in China). The base pressure of this sputtering system reaches $\sim 1 \times 10^{-4}$ Pa before deposition. The working gas is argon (99.999% purity) at the constant pressure of 0.1 Pa. The sputtering sources work at the constant power mode, regulated at 10 W and 60 W for Mg and SiC targets, re-

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spectively. Substrates in size of 20 mm × 30 mm were cut from a polished single-crystal Si (100) wafer. During the deposition of each layer, the substrate was kept stationary over the sputtering source for the time required to deposit the desired thickness. The multilayer was fabricated by alternate facing the substrate in front of each target. The deposited multilayer was then measured, for quality control and depositing rate control, by using a small angle x-ray diffractometer (D1 system, Bede Ltd., UK). The measurements show that the layer thicknesses of Mg and SiC are 12.1 nm and 4.7 nm, respectively, see table 1.

Some samples are annealed during 1.25 h (this does not include the times for increasing and decreasing the temperature) under argon atmosphere or in vacuum in the preparation chamber of the XES apparatus. The annealing temperatures range from 100 to 500°C with a 50°C step. For the samples dedicated to the XRR analysis at 0.154 nm the temperature step is 100°C. The samples are analysed by specular optical reflectivity at three different wavelengths ranging from hard x-rays to ultra-violet. XRR at 0.154 nm (Cu Kα emission at 8048 eV) is performed with a home-made apparatus [15] for the annealed samples. All the multilayers are analysed by XRR at 1.33 nm (Cu Lα emission at 932 eV) with a home-made spectrogoniometer [16].

For the as-prepared samples, UVR measurements at ∼30.4 nm were taken in $\theta - 2\theta$ mode in the 5°-35° range in s-incidence at BEAR beamline [17] at Elettra. The photon energy was calibrated by a He gas absorption cell; the calibrated value resulted 40.70 eV (30.4 nm). The θ goniometer angular resolution was $\frac{1}{100}^{\circ}$. Energy resolution $\frac{E}{\Delta}$, beam divergence and spot dimension of the incident beam at the sample were respectively: ∼1000, 20 mρ× 20 mρ (horizontal × vertical), 400μm × 200 μm (horizontal × vertical). Impinging and reflected intensities were measured by an IRD SXUV100 solid state diode within two separate runs; incident intensities were monitored by a Au mesh inserted in the beam path whose drain current was used for normalisation. The reflectometer features an overall accuracy on the absolute reflectivity of ∼1%.

The x-ray spectra are measured with the IRIS (Instrument for Research on Interfaces and Surfaces) equipped with a high-resolution wavelength dispersive spectrometer [18]. The x-rays are produced by electron irradiation. In the case of the analysis of the Si emission band, the electron energy (4 keV) is carefully chosen so that no signal can come from the silicon substrate. No evolution of the intensity and lineshape of the emission bands is detected with the number of accumulations, indicating that the electron irradiation conditions have no significant effect on the sample.

3 Results and discussion

3.1 As-prepared samples

The Mg 3p and Si 3p valence states are studied by XES through the Mg Kβ and Si Kβ emissions originating from the 3p - 1s transition. In fact, the Mg and Si 3p valence states are very sensitive to the chemical state [19,20] of the Mg and Si atoms and give information about the com-

pounds formed at the interfaces of the Mg/SiC multilayers. We compare the spectra from the multilayers to those from reference compounds: a Mg polished plate, a Mg_2Si powder (from Alfa Æsar, 99,5% purity), a SiC crystalline powder (from Alfa Æsar, 99,8% purity), a 250 nm thick SiC film deposited in the same conditions as the SiC layers of the Mg/SiC stacks and a MgO single crystal. If no interaction takes place within the multilayer, one should observe the Mg lineshape of Mg in pure metal and that of Si in SiC.

We present in figure 1 the spectra from the reference compounds that demonstrate the chemical dependence of the x-ray valence band emissions. The Mg Kβ spectra (figure 1a) of Mg and Mg_2Si can be easily distinguished by their shape and the position of their maximum. In the same way, the Si Kβ spectrum (figure 1b) of Mg_2Si is clearly distinct from those of the SiC samples. The two SiC samples present differences: for the crystalline powder the main peak is narrower and the structure around 1827 eV is better resolved. The high energy broadening could be ascribed to the presence of occupied defect states in the forbidden gap of the amorphous sample, the slight low energy narrowing to the difference between the crystalline and amorphous states and the poor resolution of the 1827 eV structure for the amorphous samples to the fact that the stoichiometry of the film is not exactly that of SiC [21].

The spectra of the as-prepared multilayers are shown in figure 2 in comparison with Mg metal for the Mg Kβ emission (Fig. 2a) and with crystalline SiC for the Si Kβ emission (Fig. 2b). For the sake of clarity, only a limited number of references is displayed. It is clear, within the sensitivity of the method, that the magnesium atoms in the multilayer are in the metallic state and the silicon ones are on the carbide state, i.e. there is no interaction or diffusion between the Mg and SiC layers.

The XRR obtained at 0.154 nm (8048 eV) with the as-prepared sample is shown on figure 3a. The Bragg peaks are narrow, intense and well-defined up to the fifth diffraction order. Some secondary peaks are also observed between the Bragg peaks. We suggest that their existence is due to a small defect or super-structure in the SiC layers, every two periods. Based on indications from XES, a fit of the experimental XRR curves is made using a two units period. Fitted parameters, i.e. the thickness and density of the Mg and SiC layers and interface roughness, deduced using the IMD code [22] are given in table 1. It can be seen that the *rms* interfacial roughness is quite large, specially for the Mg layers and that the density of the SiC layers is that of the bulk SiC whereas the Mg layers have a density slightly smaller than the one of the Mg metal.

In figure 4 the UV reflectivity at 30.4 nm (40.7 eV) obtained in a $\theta - 2\theta$ scan for an as-prepared sample is shown. The simulated reflectivity for the corresponding ideal structure with thickness parameters of table 1 is reported. The comparison with the experimental curve shows a higher reflectivity of the order of 20%. A much better agreement with the experiment is obtained when the interface roughnesses reported in table 1, as obtained from XRR measurements at hard x-ray energy, are intro-

duced into the simulation. It is important to stress that, in agreement with the indication coming from XES (i.e. absence of important interdiffusion or chemical reaction at the interfaces), the optical performance of as-prepared multilayers is substantially limited by interface roughness.

3.2 Annealed samples

We present the Mg Kβ and Si Kβ x-ray analysis of the annealed Mg/SiC multilayers in figure 5. Because it is not possible to acquire both emissions at the same time, the Si emission band is obtained only at 500°C. It is observed in figure 5a that the Mg valence states are those of the metal up to a temperature of 300°C. At 350°C a slight shoulder begins to grow at a photon energy of 1296 eV where the lineshape of Mg_2Si presents its maximum (see Fig. 1). At 400°C this shoulder is well developed and its maximum is slightly shifted toward the low photon energies, consistent with the shift between the maxima of pure Mg and Mg_2Si spectra. Then, there is no more evolution up to 500°C. The silicon emission band, figure 5b, shows that at this highest temperature the silicon atoms are in a chemical environment like the one of Mg_2Si. This behavior of the magnesium and silicon atoms evidences the formation of the magnesium silicide at the various interfaces of the stack.

This is confirmed by fitting the Mg Kβ lineshape by a weighted sum (linear combination) of the reference spectra as shown in the figure 6. An equal contribution of pure Mg and Mg_2Si is necessary to account for the shape of the spectrum of the sample after an annealing at 500°C. This means that a part of the magnesium atoms has reacted with the silicon atoms from the SiC layers to form the silicide. This is consistent with the observation that the silicon atoms have reacted to form Mg_2Si with the magnesium atoms of the Mg layers (Fig. 5b). It could be presumed that the shoulder appearing on the Mg spectrum of the highly annealed multilayers is due to an oxidation of the Mg atoms because the maximum of the MgO emission is at the same emission energy as the shoulder of the Mg_2Si emission (see Fig. 1a). However, we can rule out this possibility because even when introducing some oxide contribution in the fit, it is not possible to account for the energy shift of the maximum and to improve the fit in the range of the shoulder.

The evolution of the reflectivity obtained at 1.33 nm (932 eV) at the first Bragg order as a function of the annealing temperature is presented in figure 7. The same behavior is observed for the second Bragg order. First, by annealing at 200°C, the reflectivity decreases slightly. Between 200 and 250°C a strong decrease (80%) of the reflectivity occurs. This decrease continues up to a temperature of 350°C. Another step occurs between 350 and 400°C after which the reflectivity diminishes almost entirely. This last step is in agreement with the chemical transformation that occurs within the sample between 350 and 400°C as indicated by XES.

We present the evolution of the XRR curves at 0.154 nm (8048 eV) as a function of the annealing temperature in figure 3a-f up to 500°C. Five Bragg peaks can be observed. There is no significant evolution of the intensity and width

of the various Bragg peaks up to 200°C. From 300°C, the Bragg peaks are less intense and well-defined only up to the third diffraction order. This change is correlated with a decrease of the background intensity as shown in figure 8a. The simulation of these reflectivity curves shows that the contrast of the refractive index decreases between the Mg and SiC layers when going from 200 to 300°C. This can be explained by a development of the roughness or of the interdiffusion between the layers [23]. However, the latter can be ruled out because no chemical diffusion has been evidenced by XES. The absence of diffusion is also confirmed by looking at the value of the critical angle for the total reflection (Fig. 8a). It is almost the same (0.211°) for both annealing temperatures, meaning that the mean optical index and threfore the composition of the sample does not change. Thus, the evolution of the reflectivity at 0.154 nm between 200 and 300°C can be explained by an increase of the interfacial roughness. This is probably due to the crystallization of the Mg layers and should be confirmed by diffraction measurements. When annealed at 400°C, see figure 8b, it is observed that the Bragg peaks are less intense and become broader. The structure of the stack has almost disappeared due to the formation of the Mg_2Si compound as evidenced by XES. This is also confirmed by the shift of the critical angle down to 0.174° at 400°C, showing that the mean index and thus the composition of the multilayer has changed.

4 Conclusion

We studied the thermal stabilty of periodic Mg/SiC multilayers up to 500°C by the combination of XRR, UVR and XES techniques to show the possible thermally activated evolution of the system both through morphology and interface processes. We have shown that the as-prepared samples do not present interfacial compounds but have quite large interfacial *rms* roughness.

Upon annealing, a critical temperature for a strong decrease of the reflectivity has been reported in the literature, but the various authors do not agree on its value, 200°C [5] or 300°C [6]. In this work, we have found two critical temperatures for which we have observed an important change of the optical and chemical properties. The reflectivity is quite constant up to 200°C after which it drops drastically, very probably due to the development of the interfacial roughness. Thus, this kind of multilayer is thermally stable only for application requiring no heating higher than 200°C.

A second critical temperature has been observed between 350 and 400°C that corresponds to the destruction of the multilayer due to the formation of magnesium silicide resulting from the chemical reaction between the magnesium and silicon atoms. This leads to another very strong loss of reflectivity.

This paper shows that the knowledge and control of the microscopic processes governing the interface chemistry and morphology are the key aspects to be improved in order to optimize the optical performances and the overall stability of Mg/SiC multilayers.

Acknowledgments : Pr. R. Gauvin from McGill University is thanked for providing us with the polished Mg sample.

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Table 1. Parameters of the as-prepared Mg/SiC multilayer deduced from XRR at 0.154 nm. The relative density is the density of the layer divided by the density of the bulk corresponding material.

layer	thickness (nm)	relative density	roughness (nm)
Mg	12.1	0.94	1.7
SiC	4.7	1.00	0.9

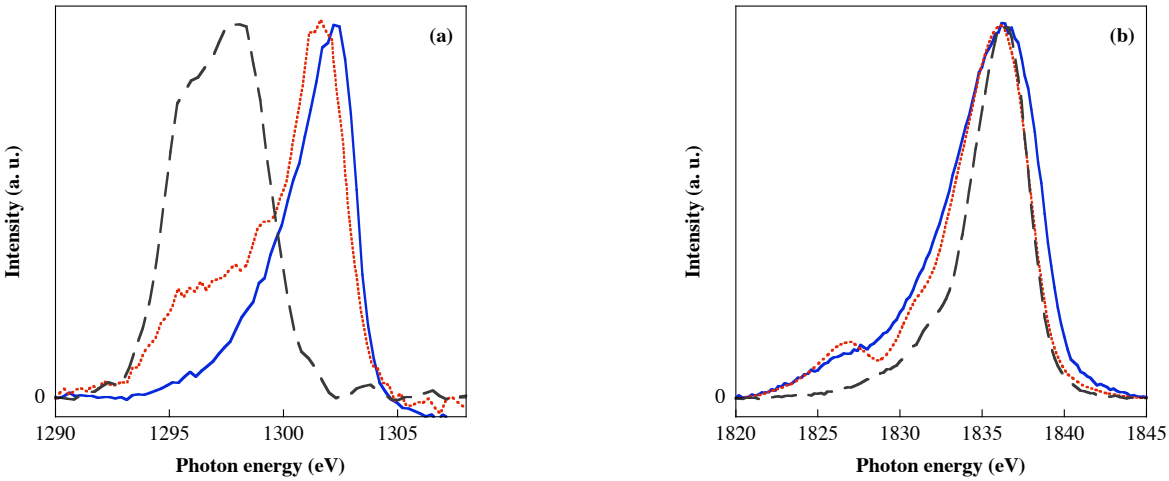

Fig. 1. (a) Mg Kβ x-ray emission bands from reference compounds: metal Mg (solid line), Mg_2Si (dotted line) and MgO (dashed line); (b) Si Kβ x-ray emission bands from reference compounds: Mg_2Si (dashed line), cystalline SiC (solid line) and amorphous SiC (dotted line).

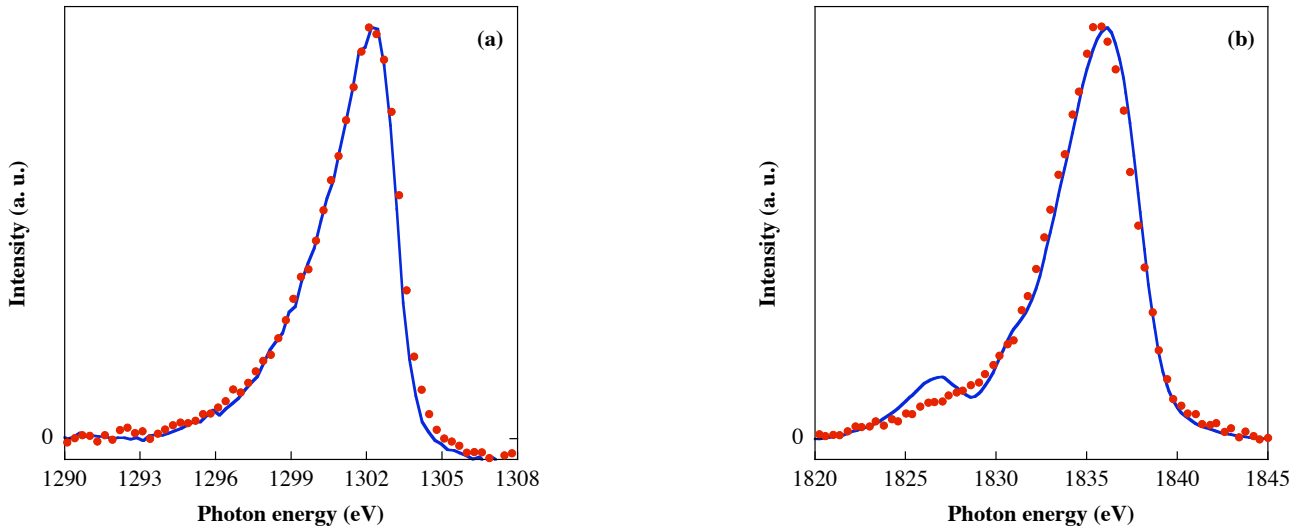

Fig. 2. (a) Mg Kβ x-ray emission bands from an as-prepared Mg/SiC multilayer (dots) and metal Mg (solid line); (b) Si Kβ x-ray emission bands from from an as-prepared Mg/SiC multilayer (dots) and crystalline SiC (solid line).

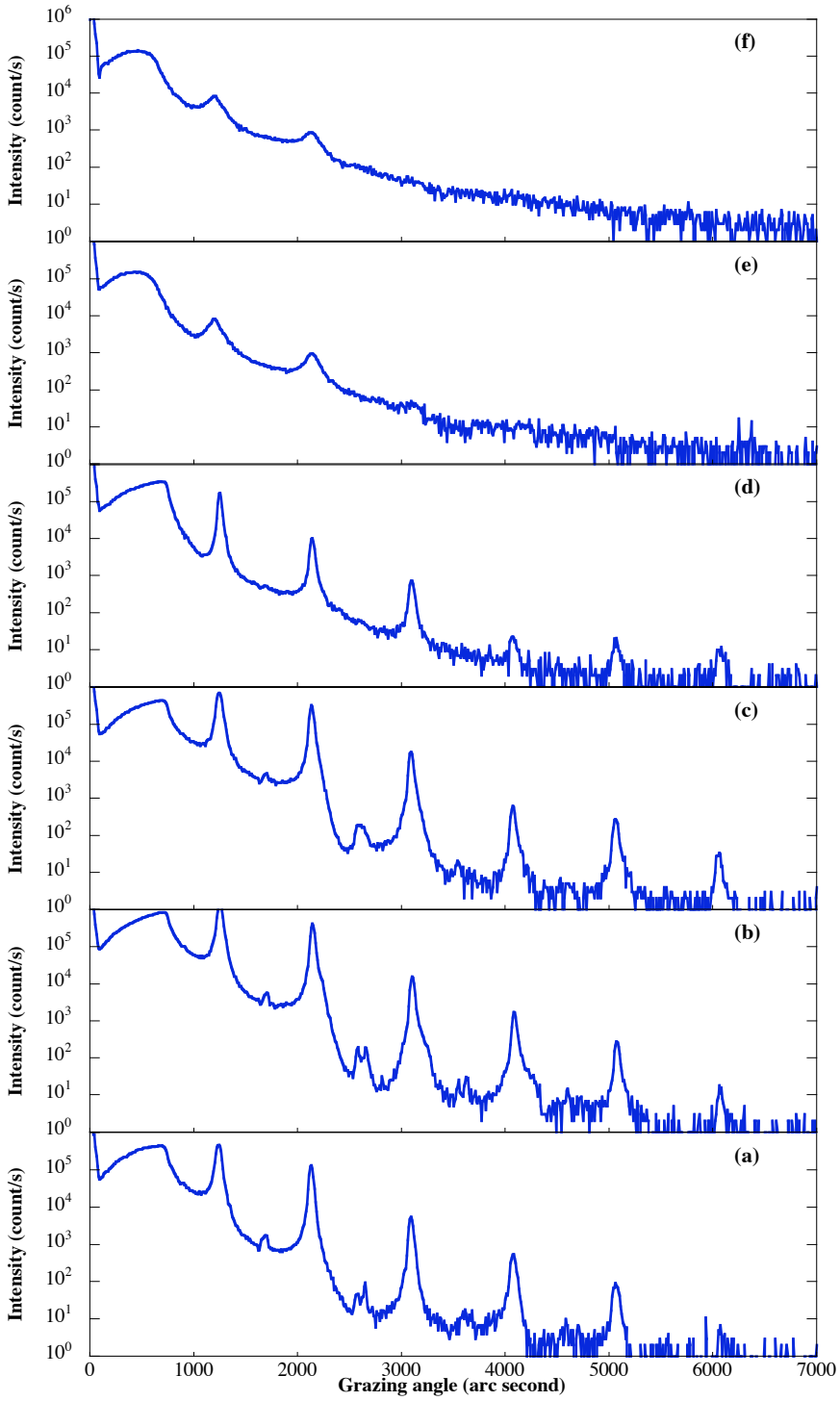

Fig. 3. Reflectivity curves at 0.154 nm (8048 eV) of the Mg/SiC multilayers: as-prepared (a), annealed at 100°C (b), 200°C (c), 300°C (d), 400°C (e) and 500°C (f).

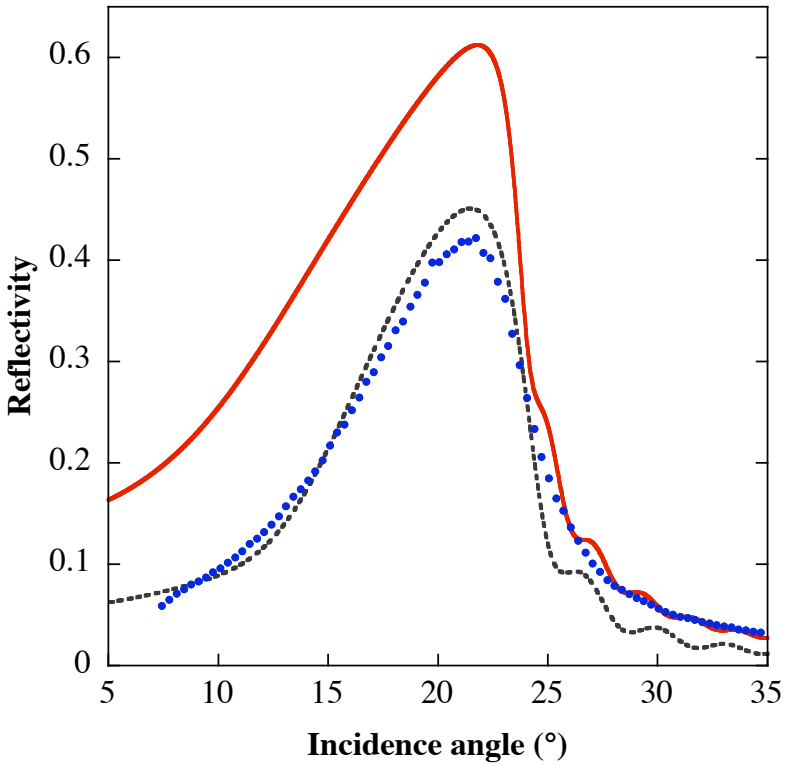

Fig. 4. Reflectivity curve at 30.4 nm (40.7 eV) of the as-prepared Mg/SiC multilayer (dots) compared to simulated curves for a perfect structure (solid line) and for a structure with the parameters listed in the table 1 (dotted line).

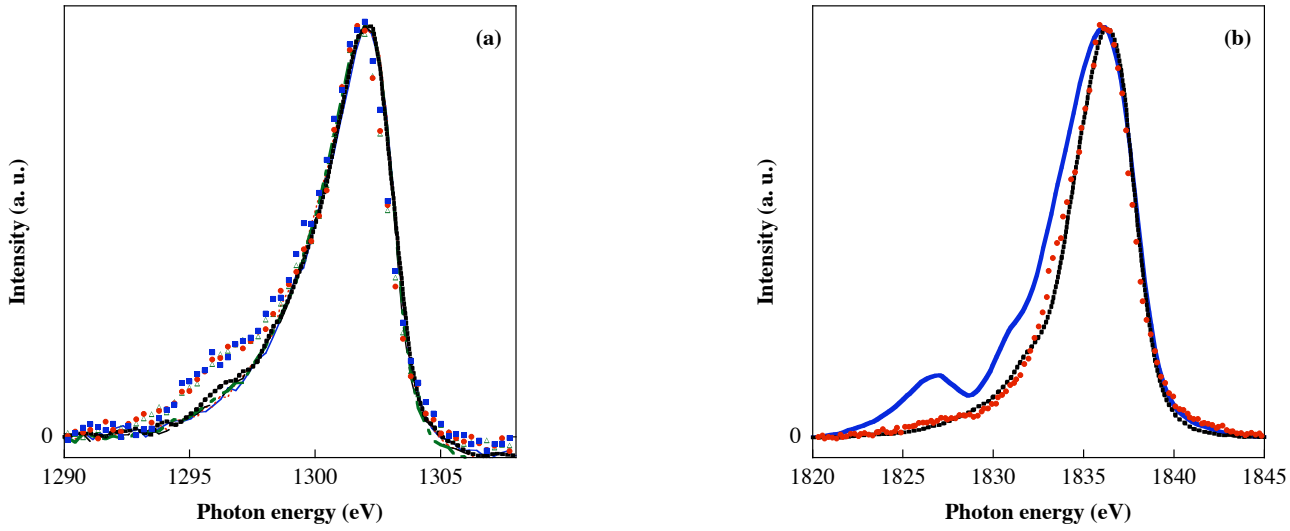

Fig. 5. (a) Mg Kβ x-ray emission bands from Mg/SiC multilayers: as-prepared, 200, 250 and 300°C (thin solid lines); 350°C (thick dotted line); 400, 450 and 500°C (dots). (b) Si Kβ x-ray emission bands from from the Mg/SiC multilayer annealed at 500°C (dots) compared to the spectra of Mg_2Si (solid line) and cystalline SiC (dotted line).

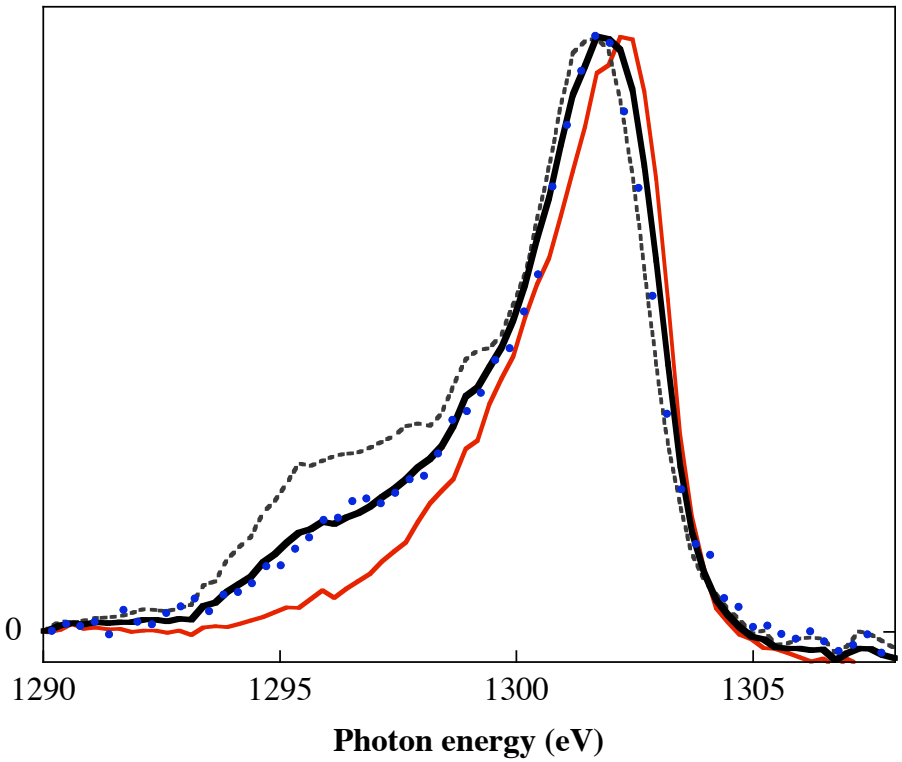

Fig. 6. Mg Kβ x-ray emission band from the Mg/SiC multilayer annealed at 500°C (dots) compared to the ones of pure Mg (thin solid line) and Mg_2Si (thin dotted line) and to a weighted sum of corresponding to 50%Mg + 50% Mg_2Si (thick solid line).

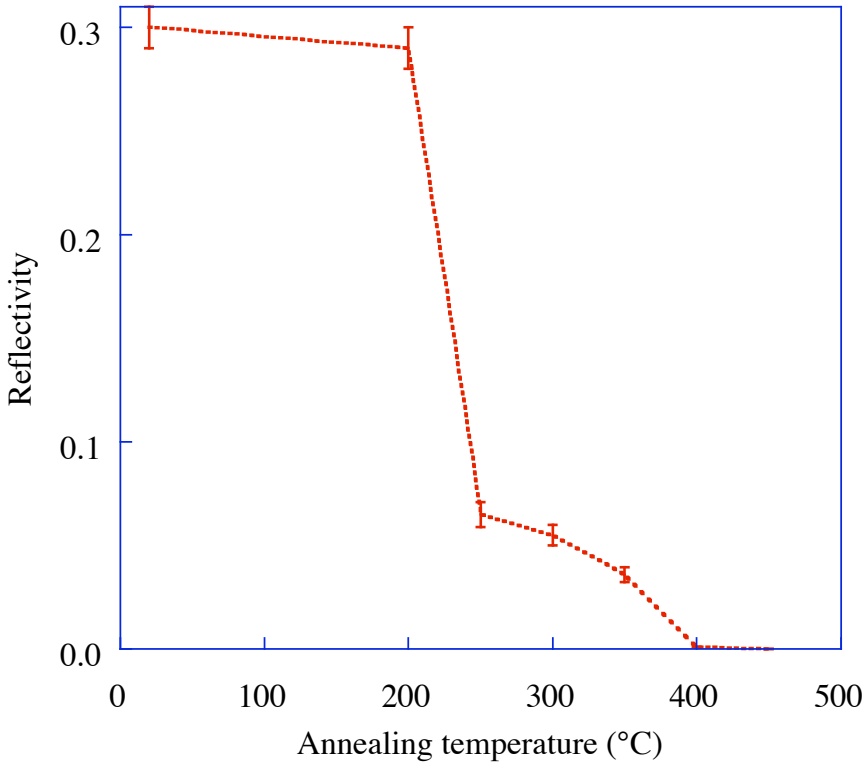

Fig. 7. Evolution of the reflectivity at 1.33 nm (932 eV) of the Mg/SiC multilayer as a function of the annealing temperature.

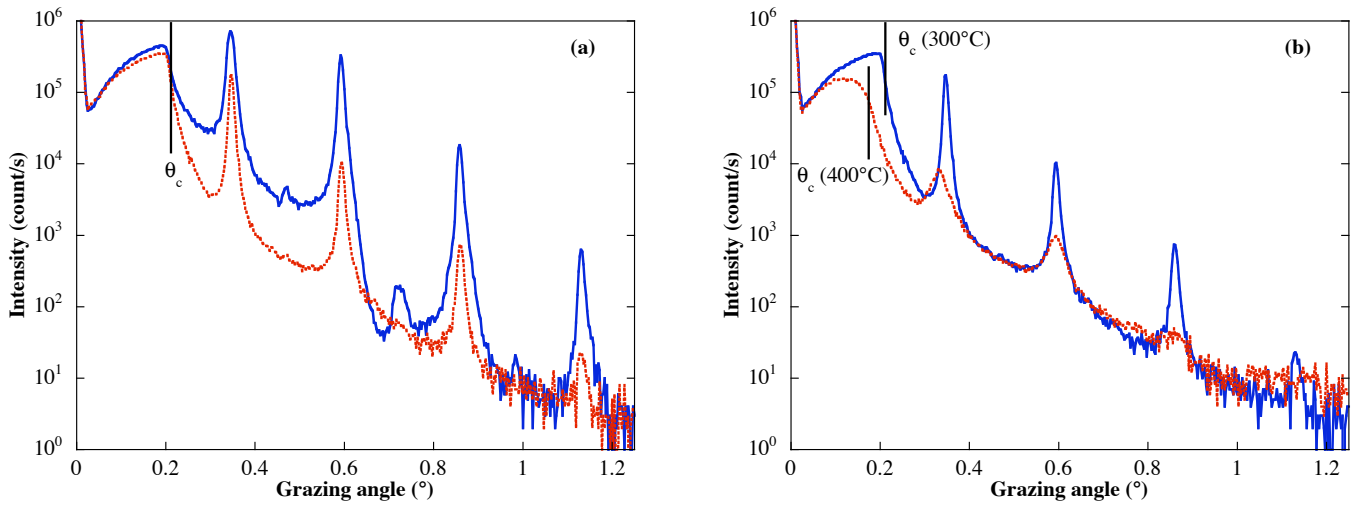

Fig. 8. Reflectivity curves at 0.154 nm (8048 eV) of the Mg/SiC multilayer: (a) annealed at 200 (solid line) and 300°C (dotted line); (b) annealed at 300 (solid line) and 400°C (dotted line). The vertical bars indicate the critical angles for total reflection.