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Interlayer composition and interface stability in Mo/Si multilayers studied with high-resolution RBS

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Abstract

Mo/Si multilayers with a period thickness of ~ 7.5 nm have been fabricated by e$^-$-beam evaporation in UHV at a deposition temperature of 150°C [1]. At their interfaces interlayers of mixed Mo and Si are obtained which are thicker at the Mo-on-Si than at the Si-on-Mo interface. The composition of as-deposited Mo/Si multilayers and bilayers and changes in the composition after baking the samples have been studied with high-resolution RBS. Differences in the behaviour of the two interfaces with baking are observed. The interdiffusion of Mo and Si starts mainly at the Mo-on-Si interface. The thickness of the interlayer at the Mo-on-Si interface increases with baking temperature. With the increasing thickness the Si/Mo mixture in the interlayer changes from Mo-rich to Si-rich. After baking at temperatures higher than 600°C strong Si diffusion into Mo is observed for both interfaces.

1. Introduction

Mo/Si multilayers (ML) serve as soft X-ray mirrors in the wavelength range above the Si-L edge [2]. The Mo/Si multilayers and bilayers studied in this work have been fabricated by e$^-$-beam evaporation in UHV onto a Si substrate, which has been kept at a temperature of 150°C during deposition (in situ) [1]. The native oxide layer on the substrate has not been removed. The double layer spacing of the ML is ~ 7.5 nm and the Si/Mo atomic ratio over the whole stack is 1.45. The normal-incidence X-ray reflectivity is stable up to a baking temperature of 500°C [3]. After baking at 600°C the reflectivity is drastically reduced to a negligible value. In order to optimize the MLs with respect to their X-ray reflectivity and temperature stability knowledge about the formation of the interfaces is important. Therefore, much interest has been invested in literature to learn about the structure of the interfaces. It is known that both for sputter deposited [4–9] and for e$^-$-beam deposited [10–12] Mo/Si MLs interfacial layers of mixed Mo and Si exist in the layer system, which are considerably thicker at the Mo-on-Si interface than at the Si-on-Mo interface. For Mo/Si MLs deposited by sputtering it is reported that the composition of the thicker interlayer is not far from $MoSi_2$ [8], whereas it seems to be different from $MoSi_2$ for e$^-$-beam deposited MLs [10]. The interlayer regions broaden if the multilayers are baked at temperatures between 300 and 600°C.

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In this temperature range crystallites of reacted $MoSi_2$ and Mo_5Si_3 have been found by Selected Area Electron Diffraction (SAED) [5,6] and Large Angle X-ray Scattering (LAXS) [6–8]. These methods detect the occurrence of crystallites, but the atomic ratio of Si and Mo in the mixed regions may differ from the stoichiometry of the crystallites because there might exist a mixture of crystallites and unreacted material. Rutherford Backscattering Spectroscopy (RBS) is a method which yields quantitative information about the composition of the analyzed target, especially about the atomic ratio of the materials in mixed regions. Since the thickness of the mixed interlayers in the Mo/Si layer system is below 3 nm, the depth resolution of a standard RBS system with semiconductor detector, which is limited to ~ 10 nm, is not sufficient. Therefore a high-resolution RBS system with a depth resolution below 3 nm is necessary to gain information about the formation of the interlayers.

2. Experimental set-up

The measurements have been carried out with He^+ ions provided by a 350 keV ion accelerator. The targets are mounted on a five-axes high-precision manipulator. The energy of the backscattered ions is determined by means of a toroidal electrostatic analyzer, which is able to detect an angular range of 25° in the scattering plane simultaneously in an angle-resolved manner. The angular resolution of the analyzer is better than 0.3° and the energy resolution $\Delta E/E$ of the whole set-up (including the ion accelerator) is 5×10^{-3}. Taking the effect of energy straggling into account, one obtains a depth resolution of ~ 1.5 nm in a depth of 8 nm in the scattering geometry shown in Fig. 1 for Mo/Si layer systems [13]. A more detailed description of the experimental RBS set-up is given in Ref. [12].

3. Results and discussion

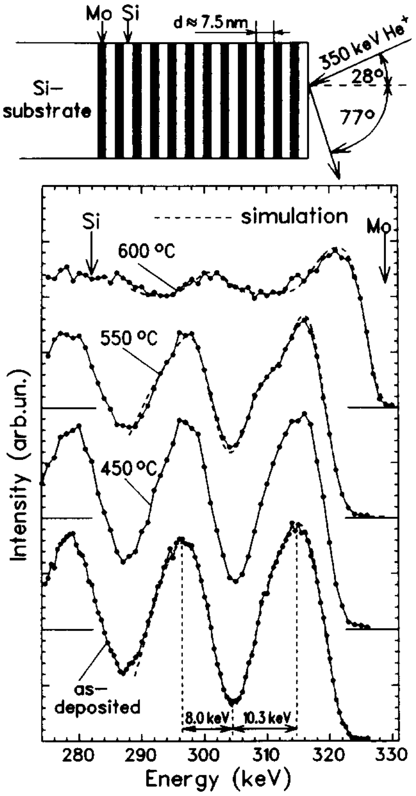

Fig. 1. RBS spectra obtained with 350 keV He^+ ions backscattered from a Mo/Si ML with a double layer spacing of ~ 7.5 nm in a scattering geometry which provides good depth resolution. Spectra are shown for the as-deposited ML and after baking at temperatures of 450, 550 and 600°C. The dashed curves are simulations of the RBS spectra for layer systems that resulted in the best fit. The expected energetic position of ions backscattered from Mo and Si at the surface is indicated by the arrows in the upper part of the figure. The first two oscillations (at high energy) are exclusively due to backscattering from the uppermost two Mo layers in the ML stack.

Fig. 1 shows the backscattering spectra (intensity measured by the analyzer versus the backscatter energy) for a Mo/Si ML with 12 periods and a double layer spacing of ~ 7.5 nm obtained with a 350 keV He^+ beam for the as-deposited ML and after baking at temperatures of 450, 550 and 600°C. The angle of incidence is 28° and the exit angle is 77°, both with respect to the surface normal. This geometry provides high depth resolution. Although the pure Mo layers in this ML system are known to be polycrystalline [14] with a texture in the direction of the film growth, no dependence of the backscattering intensity on the angle of incidence due to the channeling effect has been found.

The arrows in the upper part of Fig. 1 indicate the expected energetic position of the ions backscattered from Mo and Si at the surface. Thus the first two peaks appearing in the spectrum (between 285 and 325 keV) are exclusively due to backscattering from Mo atoms in the first two periods of the ML stack. For lower energies the Si signal is not separated from the Mo signal, therefore the analysis of the spectrum is restricted to the first two Mo oscillations. Since the depth resolution decreases with depth, mainly due to the effect of energy straggling, the first Mo peak is better resolved than the second one. For a quantitative analysis of the multilayer composition we use therefore mainly the first Mo peak. As already has been found before [12], the Mo oscillations show an asymmetric peak shape. This is attributed to interlayers of mixed Mo and Si, which are considerably thicker at the Mo-on-Si than at the Si-on-Mo interface. The slope on the low-energy side of the first Mo peak below the surface (Mo-on-Si interface) exhibits a slight shoulder located in the upper region of the decrease. This indicates that there actually exists an interlayer and not only a linear gradual change from one material to the other. This finding is further supported by a transmission electron microscopy cross section [14] of a sample which was fabricated under the same conditions. The TEM cross section reveals also an interlayer with sharp interfaces at the Mo-on-Si interface and no detectable interlayer at the Si-on-Mo interface.

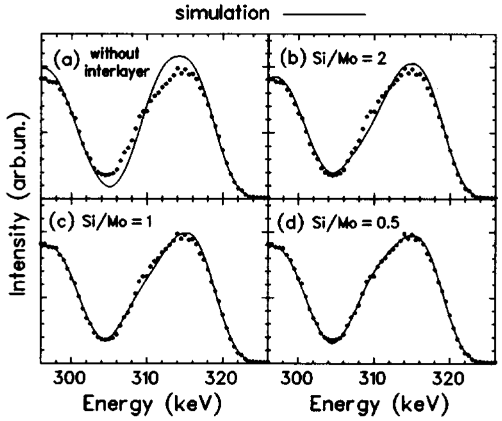

Fig. 2. The first Mo peak of the RBS spectrum for the as-deposited sample of Fig. 1 is shown with an expanded energy scale and compared to calculations for different Si/Mo atomic concentration ratios in the interlayer at the Mo-on-Si interface. The interlayer thickness for the calculations was 1.9 nm and the Si/Mo ratio averaged over one period was kept constant at 1.45. The Si/Mo ratio in the interlayer was assumed to be constant over the interlayer. The solid curves represent the calculations which have been performed (a) without interlayer and with mixing ratios of (b) Si/Mo = 2, (c) Si/Mo = 1 and (d) Si/Mo = 0.5.

In order to obtain more quantitative information about the thickness and the composition of this interfacial layer we have performed calculations of the RBS spectra. They are based on the equations given by Chu et al. [15] and use the stopping-power values tabulated in Ref. [16]. They include the effect of energy straggling, the isotopes of Mo and Si and the energy-dependent fraction of singly ionized projectiles to all projectiles leaving the target. In Fig. 2 the first Mo peak of the experimental RBS spectrum in Fig. 1 (as-deposited sample) is compared with four calculations, one without interlayer (Fig. 2a) and three for different compositions of the interlayer at the Mo-on-Si interface with Si/Mo atomic concentration ratios of 2, 1 and 0.5 (Figs. 2b–2d). The interlayer thickness used for the calculation is 1.9 nm [17]. The Si/Mo ratio was assumed to be constant over the interlayer. The thicknesses of the Mo and Si layers were modified in such a way that the double layer spacing and the Si/Mo ratio for the whole double layer remain constant (7.5 nm and 1.45). Assuming a layer system without interlayers at the Mo-on-Si interface results in a by far stronger slope on the left side of the Mo peak than in the experimental spectrum (Fig. 2a). A Si/Mo ratio of 2 yields a shoulder on the left side of the peak in the lower part of the decrease, which is also in disagreement with the experimental data (Fig. 2b). The other two calculations approach the experimental spectrum by far closer. The calculation for a mixing ratio Si/Mo = 1, however, also does not reproduce the shape of the left side of the Mo peak (Fig. 2c). The best agreement between experiment and calculation is obtained with a Si/Mo ratio of 0.5

(Fig. 2d). This calculation is also shown by the dashed curve in Fig. 1 (as-deposited sample). As a clear step is not observed in the experimental spectrum, a nonlinear gradual change of the composition from Mo to Si can also not be excluded from the RBS data, but it is not compatible with TEM cross sections of the sample [14] which reveal clearly an interlayer with sharp interfaces (slight concentration variations within the interlayer can, however, not be excluded, but in any case the average composition of the interlayer must be Mo-rich, i.e. far from $MoSi_2$ for the as-deposited sample). A system without interlayer and an interfacial roughness of 1.5 nm between Mo and Si layers might as well be considered to explain the RBS data. This model is, however, neither compatible with TEM cross sections nor with the high reflectivity for soft and hard X-rays [3,14]. Corresponding calculations with interlayers at the Si-on-Mo interface of the as-deposited sample yield the result that a possible interlayer is smaller than 1 nm [17].

In the upper part of Fig. 1 spectra of the same ML are shown after baking for 20 min at 450, 550 and 600°C under vacuum in a separate chamber. After baking at 450°C almost no change in the ML structure is detectable in the RBS spectrum. After baking at 550°C the deepness of the Mo oscillations is reduced. The major change compared to the non-baked sample has occurred at the Mo-on-Si interface, where the spectrum exhibits a distinct shoulder. This shows that the interfacial layer at the Mo-on-Si interface has broadened significantly due to interdiffusion of Mo and Si, whereas no significant change is observed at the Si-on-Mo interface. An interdiffusion at this interface would result in a shift of the high-energy edge of the RBS spectrum to higher energies. Therefore, the diffusion in the Mo/Si ML with baking starts at the Mo-on-Si interface, where the originally thick interfacial layer has been found. The simulation of the RBS spectrum after baking at 550°C yields the best fit for an interfacial layer at the Mo-on-Si interface with a thickness of 3.0 ± 0.5 nm and a Si/Mo ratio close to 1. After baking at 600°C the layered structure is almost destroyed. Only slightly Mo-richer regions exist in the sample, which might be the residue of the originally pure Mo layers, but they are located closer under the surface. The simulation of this spectrum yields a Si/Mo atomic ratio of ~ 1.7 for the Si-rich regions, which is close to the stoichiometric composition of $MoSi_2$.

To get more detailed information about the behaviour of the interfaces with baking, bilayers of Mo deposited on Si and Si deposited on Mo on a Si substrate (covered with the native oxide layer) at a deposition temperature of 150°C have been examined. Now both interfaces can be studied separately. In addition, the projectiles backscattered from Si are separated from those backscattered from Mo and the signal from the Mo layer is not influenced by the signal of the underlying next Mo layer.

In Fig. 3 the RBS spectra for a 4 nm Mo layer deposited on a 4.5 nm Si layer are shown for the non-baked sample and after baking at temperatures up to 780°C. For baking temperatures below 630°C the experimental spectra (dots) are compared with the experimental spectrum of the as-deposited sample (solid curve) in order to display the changes in the spectra due to baking. The baking of the bilayers has been performed for 20 min in the scattering chamber under UHV conditions by electron impact. The spectra have been obtained with 300 keV He^+ ions in the same scattering geometry as the spectra in Fig. 1. Below the Si signal in the spectrum some background counts are found, which are mainly due to plural scattering events [18]. For the analysis of the spectra the background, which has been assumed to be constant over the energy, is one main limiting factor for the accuracy. Again the Mo part in the spectrum shows a shoulder at the low-energy side, which is located at the upper part of the decrease. Correspondingly, the Si part shows a shoulder at the high-energy side, which is located at the lower part of the decrease. The position of these shoulders, which are due to the thick interlayer at the Mo-on-Si interface, indicates that this interlayer is Mo-rich. Upon baking the shoulders become much more distinct both for the Mo and the Si part of the spectrum. The position of the shoulders on the intensity scale shows that the composition of the interlayer changes with baking from Mo-rich to Si-rich.

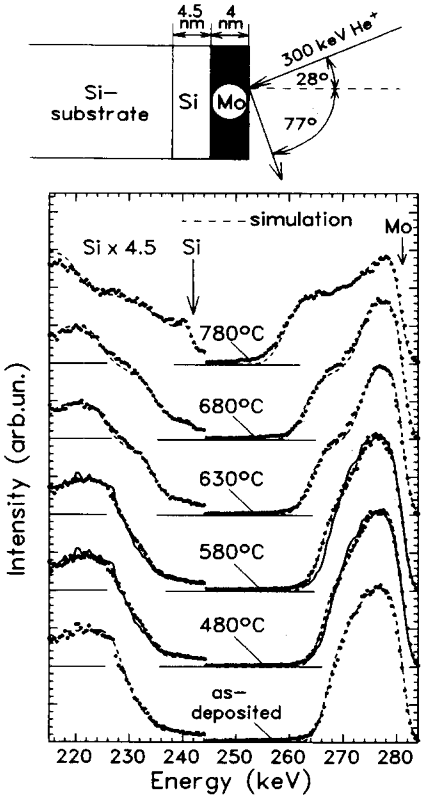

Fig. 3. RBS spectra obtained with 300 keV He$^+$ ions backscattered from a bilayer Mo on Si in the same scattering geometry as in Fig. 1. This bilayer is used to study the Mo-on-Si interface separately. Spectra are shown for the as-deposited bilayer and after baking at temperatures up to 780°C. The Si signal has been multiplied by the factor 4.5. The dashed curves are simulations of the RBS spectra for a layer system that resulted in the best fit of the experimental data. The spectra after baking at 480 and 580°C are additionally compared to the spectrum of the non-baked sample (solid curve).

Interdiffusion has already occurred at 580°C, and the increase in the width of the Mo peak shows that this interdiffusion takes place at the expense of the pure Si layer. As no shift of the high-energy edge of the Si signal to higher energy is observed at 580°C the interdiffusion is limited to a thin layer of less than 1 nm around the original interlayer, in agreement with the assumption for the RBS calculation that the interlayer has increased from ~ 1.7 to ~ 2.2 nm after baking at 580°C.

After baking at 630°C again interdiffusion is observed. The average composition of the interlayer region is now Si-rich. The best fit of this spectrum is obtained by use of an almost pure Mo layer (Si content $\leq 5\%$) with a thickness of ~ 2.7 nm. This is only ~ 0.2 nm smaller than the thickness value that has been used to obtain the best fit for the spectrum of the non-baked sample. Therefore, the spectra are compatible with an almost stable thickness of the pure Mo layer up to a baking temperature of 630°C. This can also be deduced from the almost stable energetic position of the high-energy edge of the Si signal. This observation is consistent with the change in the composition of the interlayer. Therefore, the main interdiffusion process up to 630°C in this layer system is expected across the interface Si/interlayer, and only weaker interdiffusion may take place at the interface Mo/interlayer.

After baking at 680°C a small backscattering contribution of Si in the surface region is found, which corresponds to an amount of ~ 13% Si in the Mo layer on top.

The interlayer thickness and the Si/Mo atomic concentration ratio of the mixed interlayer, which has been determined by means of simulations of the spectra (dashed curves in Fig. 3), are shown in Fig. 4. In these simulations the Si/Mo ratio has been assumed to be constant over the interlayer. Fig. 4a shows the dependence of the amount of atoms/cm^2 in the interlayer and the corresponding thickness of the interlayer on the baking temperature [17], Fig. 4b shows the relative concentration of Si and Mo in the interlayer versus baking temperature. The amount of Mo atoms in the interlayer has been kept almost constant up to 630°C, as can be verified from Figs. 4a and 4b. The error bars contain systematic errors. For example, in the spectrum after baking to 580°C clearly detectable interdiffusion has already occurred, as can be seen in Fig. 3. The interlayer thickness increases with baking temperature from initially 1.7 nm for the non-baked sample up to 3.8 nm after baking at 680°C. The Si/Mo atomic ratio in the interlayer increases from initially ~ 0.5 for the non-baked sample to ~ 1.7 after baking at a temperature ≥ 630°C. The major change in the thickness and the composition occurs fast between 580 and 630°C.

The atomic ratio of 1.7 in the interlayer above a baking temperature of 600°C is close to the composition of $MoSi_2$. Since the reaction temperature for crystallization of $MoSi_2$ is in the range of 630°C after Refs. [8,19], the interlayer may mainly consist of crystallized $MoSi_2$ at a temperature ≥ 600°C. LAXS studies of the ML samples indicate indeed the existence of $MoSi_2$ crystallites after baking to a temperature higher than 600°C [20]. After baking at 780°C a large increase of the Si signal near the surface is found, therefore the originally pure Mo layer is now strongly involved in the diffusion process.

In Fig. 5 the set of RBS spectra for a bilayer consisting of 4.5 nm Si on 4.5 nm Mo evaporated on a silicon substrate is shown for the as-deposited sample and after baking to temperatures in the range from 580°C up to 780°C. In order to show the changes in the layer system after baking the spectra for baking temperatures of 580 and 630°C are again compared with the spectrum of the as-deposited sample (solid curve). The spectrum for the non-baked sample shows a steep increase at the high-energy edge of the Mo part, which corresponds to the Si-on-Mo interface. The steepness is mainly determined by the depth resolution of the set-up, therefore the thickness of an interfacial layer will be less than 1 nm. Baking to

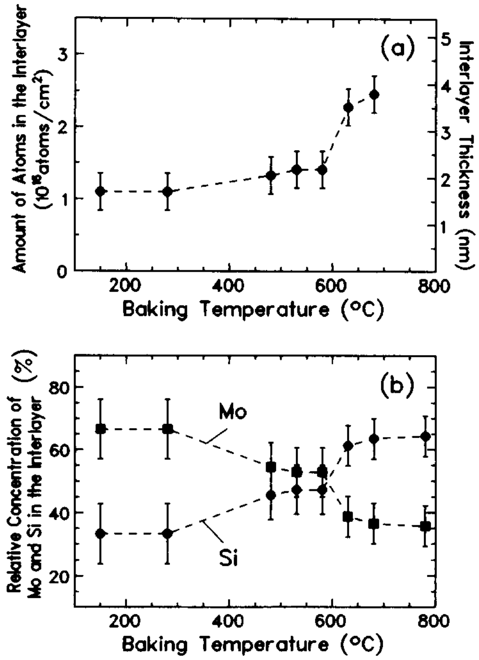

Fig. 4. (a) Amount of atoms per cm^2 in the interlayer at the Mo-on-Si interface versus baking temperature. Corresponding values for the thicknesses are also given [17]. (b) Relative concentration of Si and Mo atoms in the Mo-on-Si interlayer versus baking temperature. The amount of atoms per cm^2 and the relative quantity of Si and Mo atoms has been extracted from the spectra in Fig. 3 by means of simulations.

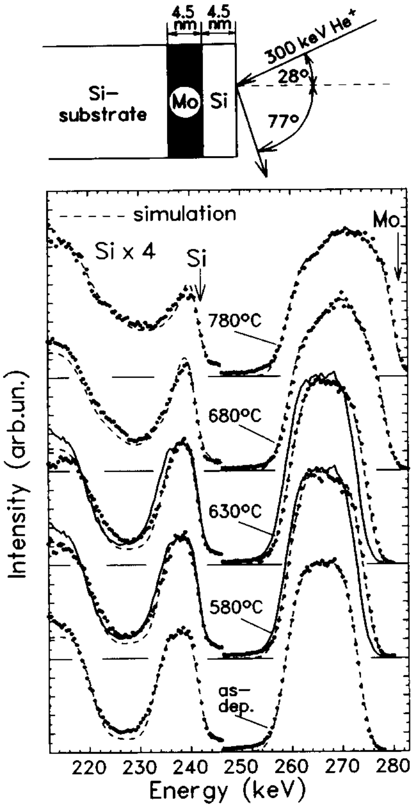

Fig. 5. RBS spectra obtained with 300 keV He$^+$ ions backscattered from a bilayer Si on Mo in the same scattering geometry as in Fig. 1. This bilayer is used to study the Si-on-Mo interface separately. Spectra are shown for the as-deposited bilayer and after baking at temperatures up to 780°C. The Si signal is multiplied by the factor 4. The dashed curves are simulations of the RBS spectra for the layer system that resulted in the best fit of the experimental data. The spectra after baking at 580 and 630°C are additionally compared to the spectrum of the non-baked sample (solid curve).

550°C does not result in a shift of the high-energy edge of the Mo part, which corresponds to the Si-on-Mo interface, as can be seen in the spectra of the ML in Fig. 1. No detectable interdiffusion occurs therefore across the Si-on-Mo interface up to this temperature. After baking to 580°C (in Fig. 5) the width of the Mo peak has increased and a slight shift of the Mo part to higher energy is observed. The width of the Si layer on top is slightly reduced and a small amount of Si seems to be accumulated close to the interface Mo/Si-substrate. The low-energy edge of the Mo signal shows also a shift to a higher energy. This can be explained by the Si accumulation at the interface Mo/Si-substrate. A loss of Si from the surface, however, cannot be excluded in this case, but the amount of material lost will be less the 5% of the Si layer. The increase of width of the Mo peak, however, shows that interdiffusion has occurred at 580 and 630°C.

Strong interdiffusion at this interface occurs at temperatures above 630°C. After baking at 680°C the steepness of the Si-on-Mo interface is clearly reduced. The total backscattering intensity of the Si layer has decreased, the Mo peak has broadened significantly and has shifted again to higher energy. The shape of the Mo peak has also changed. At the low-energy side a shoulder has developed and a corresponding change in the Si signal is found. This can be explained by diffusion of Si atoms from the Si layer on the top through the Mo, leading to a Si enrichment at the interface of the Mo with the substrate. Diffusion from the substrate is not expected, since the Si-oxide layer on the substrate forms an effective diffusion barrier [21]. In addition, all calculations in Fig. 5 have been performed within $\pm 2\%$ with the same amount of material in the bilayer. The area of the backscattering contribution from the Si layer after baking at 680°C is reduced by $\sim 40\%$ compared to the non-baked sample. If this Si material would be lost during baking, the agreement between experiment and calculation requires that the loss is exactly compensated by Si from the substrate, which is very unprobable. The best fit of the spectrum after baking at 680°C is obtained with a Si/Mo atomic ratio of 0.5 at the substrate side and a ratio of 0.2 in the most Mo-rich region of the originally pure Mo layer. Since the Si enrichment at the interface Mo/substrate takes place in the opposite direction of the concentration gradient for Si, there must be a driving force so that this Si enrichment is energetically favourable. After baking at 780°C further diffusion has occurred. Almost all Si from the top layer has intermixed with the Mo layer, but the Si enrichment at the interface with the substrate is still obvious.

Comparing the behaviour of the two interfaces we find that up to a baking temperature of 550°C the diffusion occurs mainly at the Mo-on-Si interface, where an increase in thickness of the mixed interlayer is found. This increase in interlayer thickness is mainly at the expense of the pure Si layer, and the atomic ratio Si/Mo of the interlayer changes from ~ 0.5 for the non-baked samples to about 1 after baking in the range of 550°C. At the Si-on-Mo interface the interlayer thickness after baking to 550°C is still below 1 nm. After baking at 580°C and higher interdiffusion is observed both across the Mo-on-Si and the Si-on-Mo interface. The Si/Mo ratio between the originally pure Mo layers in the ML and in the interlayer of the bilayer Mo on Si approaches that of $MoSi_2$. From the fact that a Si enrichment is found at the substrate side of the Mo layer for the Si on Mo sample and that for both bilayers firstly a small content of Si in the originally pure Mo layer is found, we can conclude that Si is the dominant diffusing species in the polycrystalline Mo layer, perhaps along grain boundaries of the Mo crystallites.

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