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Thermally enhanced interdiffusion in Mo/Si multilayers

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The formation and development of Mo-Si interfaces in Mo/Si multilayers upon thermal annealing, including a transition to *h*-MoSi₂, have been investigated using high resolution transmission electron microscopy, x-ray reflectivity, and x-ray diffraction measurements. The silicide layers naturally formed at Mo-Si interfaces, i.e., just upon and after the deposition, are amorphous and have different thicknesses for as-deposited samples, with the Mo-on-Si interlayer being the largest. In addition, silicide growth at Mo-Si interfaces during annealing before the phase transformation predominantly takes place at the Mo-on-Si interface and a MoSi₂ interface layer is formed. Diffusion continues until a thick MoSi₂ layer is formed at the interface, at which point the interface crystallizes and diffusion speeds up, finally resulting in an abrupt intermixing and phase transition of the entire interface to *h*-MoSi₂. This model predicts an onset of the phase transition which does not depend primarily on the annealing temperature but on a threshold thickness of the MoSi₂ interface before crystallization takes place. This crystallization threshold is shown to exist not only in the Mo/Si system, but also occurs for Mo/Si multilayers where the natural interfaces are replaced by diffusion barriers. © 2008 American Institute of Physics. [DOI: 10.1063/1.2907964]

I. INTRODUCTION

Extreme ultraviolet lithography (EUVL) systems, utilizing Mo/Si reflective optics, are currently considered as next generation lithography systems for the semiconductor industry.¹ The principal requirement for an EUVL optical system is to produce an image with near-diffraction limited resolution, requiring accurate control of the reflected wave, including its phase, along the optical path. Due to the high dose of EUV radiation incident on the optics during the lithography process, their temperature might easily reach a few hundreds degrees, which would influence properties such as the multilayer period thickness. Although a small change of the period thickness does not dramatically change the peak reflectance, the resulting small change in the reflected wavelength of a full EUVL system composed of ten mirrors leads to phase errors that can add up to several percents, affecting the image quality.

Previous studies on thermally treated Mo/Si multilayers at temperatures in the range of 20–800 °C already revealed the formation of different Mo silicides.^{2,3} Elsewhere, we presented a model based on the minimization of the total free energy to explain the formation of preferred silicides as a function of the Mo/Si ratio in the multilayer.⁴ In this work, we focus on the formation and growth upon annealing of the interdiffusion zones in Mo/Si multilayers, as previously reported.^{5,6} We show that the reported behavior upon annealing in Mo/Si interlayers can be linked to the asymmetry of interlayer interfaces that were formed directly after deposition. From combined grazing incidence x-ray (GIXR) and wide angle x-ray diffraction (WAXRD) measurements, we investigate the process of silicide formation. We also show

that the phase transition in the Mo/Si system depends not on annealing temperature but actually on a minimum amount of silicide formed at the interfaces.

II. EXPERIMENTAL

Multilayers of 50 bilayers of Mo and Si with a period thickness of 6.9 nm with a Mo fraction of 0.4 were deposited by electron-beam evaporation onto 25 × 25 mm² super polished silicon substrates. The base pressure during deposition was better than 2 × 10⁻⁸ mbar. The deposition system has been described elsewhere.⁷ Kr ions were applied after depositing each Si layer to suppress roughness development within the multilayer. As a result of this, a reflectivity of 69% is routinely achieved at 13.5 nm at near-normal incidence.^{8–11}

The multilayers were successively annealed for 48 h from 20 to 400 °C using a halogen lamp in a vacuum chamber (base pressure of 10⁻⁷ mbar) and analyzed by GIXR and WAXRD with a Philips X'Pert double crystal x-ray diffractometer using Cu K α radiation (0.154 nm). During the WAXRD measurements, the sample was rotated by $\varphi=20^\circ$ in the sample plane to suppress the diffraction peak from the monocrystalline substrate and aligned with the incident beam at a fixed angle of $\omega=1^\circ$ to maximize the illuminated area and thereby the diffracted intensity. A detailed analysis of the nanosize crystallites in Mo/Si multilayers is presented in Ref. 12.

Several multilayers were annealed both in successive runs at temperatures in the range of 300–350 °C, as well as directly to these temperatures to check if there is any effect of annealing history. No significant difference was observed between the two methods, and only successively annealed samples will be reported on here. Multilayer bilayer thicknesses were determined from theta-2theta scans using the above x-ray spectrometer. Transmission electron microscopy (TEM) was used in bright field mode (Philips CM30T) at

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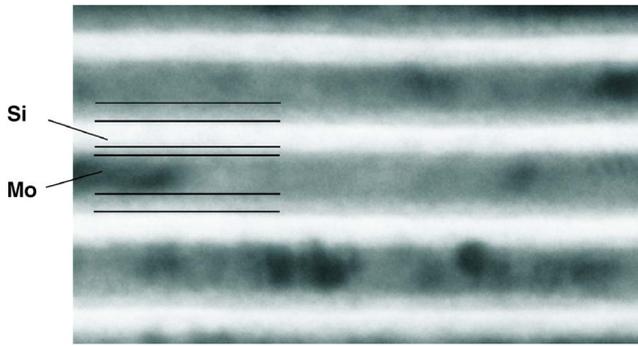


FIG. 1. (Color online) Cross-sectional bright field TEM image of a Mo/Si multilayer, showing 1.0 nm Mo-on-Si and 0.5 nm Si-on-Mo interlayers.

300 kV to indicate the layer structure. Specimens for TEM were prepared by Ar ion milling of multilayer cross sections glued on a copper grid. The Si $K\beta$ emission coming from the silicon atoms present in the samples was analyzed from x-ray emission spectroscopy (XES). This emission corresponds to the $3p-1s$ transition and describes the occupied valence states having the Si $3p$ character. The soft x-ray spectrometer with which the measurements are performed was the IRIS apparatus.¹³ An InSb (111) crystal was used at the first diffraction order. In these conditions, the spectral resolution $E/\Delta E$ is about 2000. The Si $1s$ core holes, whose binding energy is 1840 eV, are created by an electron beam generated by a Pierce gun. The energy of the incident electrons was set at 7.5 keV. It was verified that this energy is sufficiently low so that the electrons cannot reach the substrate.

III. RESULTS AND DISCUSSION

A. Diffusion through Mo-Si interfaces at enhanced temperatures

The initial thicknesses of the Mo-Si interfaces were determined from TEM measurements. Figure 1 shows a cross-

section bright field electron micrograph taken from a Mo/Si multilayer with a Mo fraction of 0.4. From this, it was determined that a 1 nm thick interface is formed at the Mo-on-Si interface, while a thinner interlayer of 0.5 nm is formed at the Si-on-Mo interface. Similar interfaces were previously reported for multilayers deposited by magnetron sputtering.^{5,14,15} During thermal treatment in the temperature range of 20–300 °C, the diffusion at Mo-Si interfaces is enhanced and the as-deposited intermixed interfaces gradually expand.

Figure 2 (solid squares) shows the compaction of the multilayer period upon annealing in the temperature range of 150–300 °C, as determined by GIXR measurements. Also shown are data for $T > 300$ °C, exhibiting a phase transition, which will be discussed in Sec. III B.

Figure 2 (open squares) also shows the reduction of the Mo crystallite size in the growth direction upon annealing, which is taken to represent the amount of Mo that is “consumed” into the compound interfaces. Upon this process the interfaces then expand. It is apparent that, below 300 °C, there is a one-to-one correlation between the consumed Mo amount and the reduction in multilayer period. From this, we uniquely determined the type of silicide formed during annealing at Mo-Si interfaces. In Table I for all Mo-Si compounds, we list the molecular volumes calculated from tabulated density values¹⁶ and the period compaction given for the formation of 1 nm silicide. For example, 0.4 nm Mo and 1 nm Si would be consumed in forming 1 nm of MoSi_2 , thereby leading to a period compaction of 0.4 nm. Actually, MoSi_2 is the only silicide where the period compaction would be equal to the amount of consumed Mo, and therefore equal to the reduction of the Mo crystallite size in the growth direction, as illustrated in Fig. 2(a).

In addition, by comparing TEM pictures before and after annealing at 300 °C, we determined that indeed 1 nm Si was consumed upon formation of a 1 nm interface, supporting

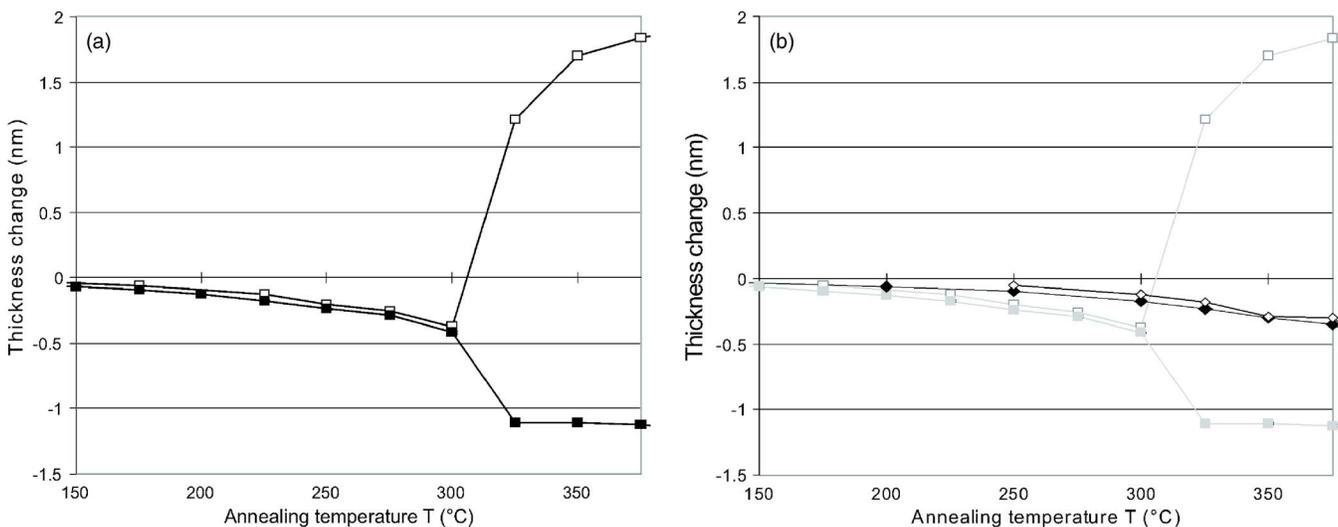


FIG. 2. (Color online) (a) Period compaction (solid squares) and change in transverse crystallite size (open squares) upon annealing. The direct relation between the two curves up to an annealing temperature of 300 °C signifies the formation of additional MoSi_2 at the interfaces. (b) Period compaction (black solid circles) and change in transverse crystallite size (black open circles) for a Mo/Si multilayer with a Si_3N_4 diffusion barrier. For reference, the period compaction (gray solid squares) and change in transverse crystallite size (gray open squares) for a standard Mo/Si multilayer as in (a) is also shown. The presence of Si_3N_4 significantly reduces diffusion processes, preventing of at least postponing the phase transition to $h\text{-MoSi}_2$ to a temperature outside the probed range.

TABLE I. Molecular volumes of Mo and Si existing in all Mo_xSi_y compounds divided to the silicide's molecular volume that are used to calculate the period compaction.

	A (g/mol)	ρ (g/cm ³)	V (cm ³ /mol)	V_{Mo} (cm ³ /mol)	V_{Si} (cm ³ /mol)	$V_{\text{Mo}}/V_{\text{silicide}}$	$V_{\text{Si}}/V_{\text{silicide}}$	Period compaction: $V_{\text{Mo}}/V_{\text{silicide}}$ $+V_{\text{Si}}/V_{\text{silicide}}-1$
Mo	95.9	10.2	9.40					
Si	28.08	2.3	12.21					
Mo_3Si	315.78	8.97	35.21	28.21	12.21	0.80	0.35	0.15
Mo_5Si_3	563.74	8.2	68.75	47.01	36.63	0.68	0.53	0.21
MoSi_2	152.06	6.24	24.37	9.40	24.42	0.39	1.00	0.39

the previous finding of MoSi_2 as the type of interface being formed. The TEM analysis performed at samples annealed at 300 °C suggests that the growth of MoSi_2 predominantly takes place at the Mo-on-Si interface, which also showed the largest amount of intermixing for the as-deposited multilayer. We suggest, similar to the results in Ref. 14 collected at room temperature, that during annealing the crystallinity of Mo may play a dominant role in determining the growth of the interfaces. During Mo growth, the initial growth of Mo takes place in an amorphous phase, while only after several nanometers a crystalline phase occurs.^{4,14,15} Therefore, during annealing in the case of the amorphous Mo, due to the high mobility of the Mo atoms, this interface may be more susceptible to additional silicide formation than the Si-on-crystalline-Mo interface.

To confirm that diffusion takes place preferentially at the Mo-on-Si interface, we treated each as-deposited Si surface with N ions, chosen as an arbitrary example material. This treatment resulted in the formation of a Si_3N_4 interlayer as a diffusion barrier on the top side of the Si layers, which was confirmed by x-ray photoemission spectroscopy (XPS) measurements. The alternate interface was not treated. Figure 2(b) shows the period compaction and the Mo crystallite size change for two Mo/Si multilayers with and without Si_3N_4 at the Mo-on-Si interface.

We observe that due to the presence of Si_3N_4 at the Mo-on-Si interface the diffusion rate is significantly reduced during annealing up to 375 °C and the crystallization is absent in this range, confirming that this interface is indeed the most “active” interface during annealing. The remaining compaction is due to the diffusion at Si-on-Mo interface,

which was not passivated, and possibly at the not-fully passivated Mo-on-Si interface. In the next section we will discuss the diffusion and crystallization processes for Mo/Si multilayers annealed in the range of 300–400 °C in more detail.

B. Abrupt phase transition to $h\text{-MoSi}_2$

At temperatures higher than 300 °C, the “standard” Mo-Si multilayer, composed by the deposition of only Mo and Si materials, suffers an abrupt phase transformation to $h\text{-MoSi}_2$ ^{2,3,5} which consumes entirely the available amounts of bulk components,^{4,16} as indicated in Fig. 2 by the abrupt reduction in period thickness and the sharp increase in crystallite size (actually the MoSi_2 crystallite size is measured here, since above 325 °C all bulk Mo is transformed into MoSi_2).

The phase transformation of bulk Mo and Si into $h\text{-MoSi}_2$ is in agreement with XES measurements performed on multilayers annealed at 400 °C. In Fig. 3 a clear change is noticed between the Mo-Si multilayer spectrum after deposition and the multilayer annealed at 400 °C. The Si 3*p* spectral density of the annealed multilayer at 400 °C shows a close resemblance to the reference spectrum of MoSi_2 (Ref. 17) suggesting a significant evolution in the physical-chemical environment of the silicon atoms between 20 and 400 °C towards MoSi_2 formation.

Figure 4 depicts the changes in the Mo-Si multilayer structure during annealing in the range of 20–400 °C. As discussed before, a 1 nm thick interface is initially formed at the Mo-on-Si interface, which increases to 2 nm after being

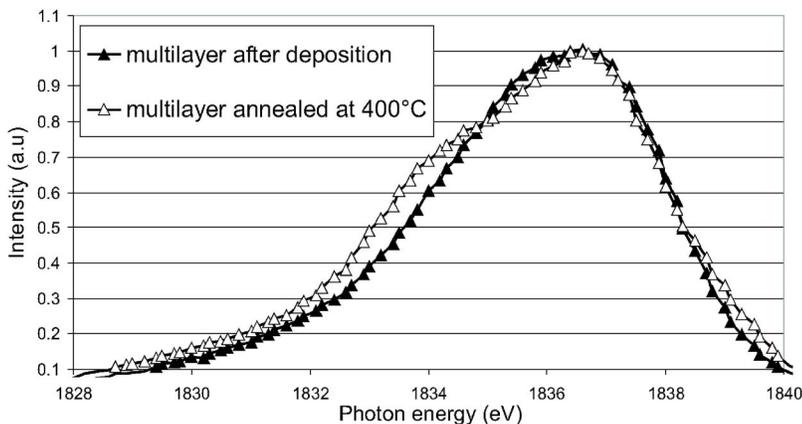


FIG. 3. (Color online) The change in Si 3*p* spectral densities between the Mo/Si multilayer after deposition (solid triangles) and after annealing at 400 °C (open triangle) suggests the multilayer transformation into MoSi_2 .

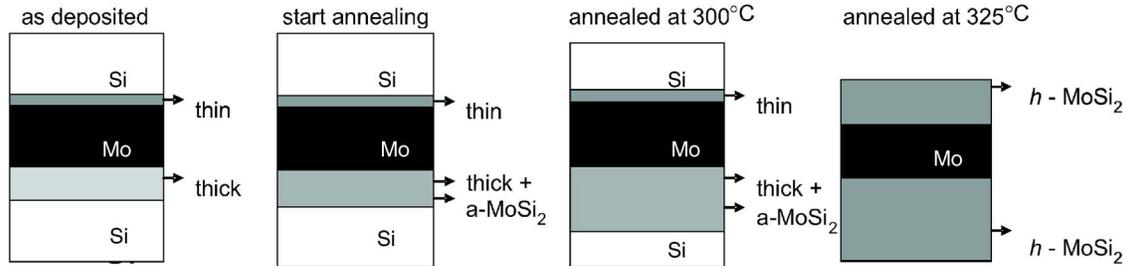


FIG. 4. (Color online) A model of the changes in the structure of a Mo/Si multilayer during thermal treatment is given here, illustrating the gradual formation of amorphous MoSi_2 up to a critical thickness of 2 nm, where crystallization of the interface occurs and all available bulk Mo and Si transforms into $h\text{-MoSi}_2$.

annealed at 300 °C. It was observed before that in the Mo/Si system a thickness threshold of 2 nm exists for the abrupt crystallization of Mo, attributed primarily to the interfacial and bulk excess energies of amorphous clusters and secondly to the Si concentration into Mo which should be below the solid solubility limit.¹⁵

In our case, the multilayer GIXR data could not be modeled considering the composition of the original Mo-Si interface layers formed after deposition as being only MoSi_2 . Instead, a combination of silicides (Mo_5Si_3 and MoSi_2) was required to successfully model the GIXR measurements.¹⁸ The Mo-on-Si interface is initially 1 nm as shown in Fig. 1. Since it most likely consists of a combination of silicides, more amorphous material has to be formed at this interface during annealing with the Mo:Si ratio of 1:2 which would favor the MoSi_2 crystallization.¹⁹ It was shown in Sec. III A that for temperatures up to 300 °C the amorphous material formed at Mo-Si interfaces is MoSi_2 . At 300 °C, besides the 1 nm silicide combination formed at room temperature, the amorphous environment with the 1:2 Mo:Si ratio formed during annealing is approximately 1.2 nm at Mo-on-Si interface which is sufficient to cause the abrupt interlayer crystallization into $h\text{-MoSi}_2$ for temperatures higher than 300 °C ($a=4.64$ Å and $c=6.53$ Å), see Ref. 19. We here suggest that the abrupt phase transformation to $h\text{-MoSi}_2$, evident in Fig. 2, is not linked to a threshold value of the annealing temperature but actually to the abrupt crystallization that occurs after a critical thickness of 2 nm Mo-on-Si interlayer is formed. The diffusion rate will increase dramatically⁵ along the grain boundaries in the now crystalline Mo-on-Si interface and all bulk available Mo and Si will interact and form $h\text{-MoSi}_2$. Annealing at temperatures higher than 325 °C does not change the multilayer formed structure, confirming this intermixing saturation phenomenon.

C. Impurities effect on the abrupt phase transformation to $h\text{-MoSi}_2$

It was shown in Fig. 2 that the diffusion is significantly reduced by passivating the top of each Si layer after deposition with N ions. According to our model, a phase transition to $h\text{-MoSi}_2$ would still be expected in the entire multilayer when the critical interlayer thickness of 2 nm is reached. However, this could not be reached below 400 °C because of slowed down diffusion through the passivated part of the Si layer. Thus, additional experiments were performed at temperatures higher than 400 °C to verify the existence of a

critical crystallization threshold in systems with a passivated Si surface. We prepared multilayers with different Si_3N_4 thicknesses and the annealing results are summarized in Fig. 5.

The curves indicate that the period compaction increases abruptly for the same critical thickness of the Mo-on-Si interface zone, namely, 2 nm, consequently confirming our finding of the thickness threshold required for silicide crystallization. In Fig. 6 we present the WAXRD spectra for the multilayers with thin and thick Si_3N_4 layers deposited on top of each Si layer and the multilayer without Si_3N_4 layer for temperatures corresponding to a Mo-on-Si interlayer thickness equal to 2 nm (before crystallization) and higher than 2 nm (after crystallization). The WAXRD spectra of the $h\text{-MoSi}_2$ are also shown (black lines). The vertical dotted lines point towards the peak position in these spectra where the presence of a crystalline silicide $h\text{-MoSi}_2$, can be identified in the annealed multilayers with a Mo-on-Si interlayer thicker than 2 nm.

Although it is expected that the phase transformation to $h\text{-MoSi}_2$ occurs in the entire multilayer, the x-ray experimental data show that up to 500 °C (highest temperature applied) multilayer crystallization does not occur. This might be caused by the lack of Si in the multilayers after Si_3N_4 layer

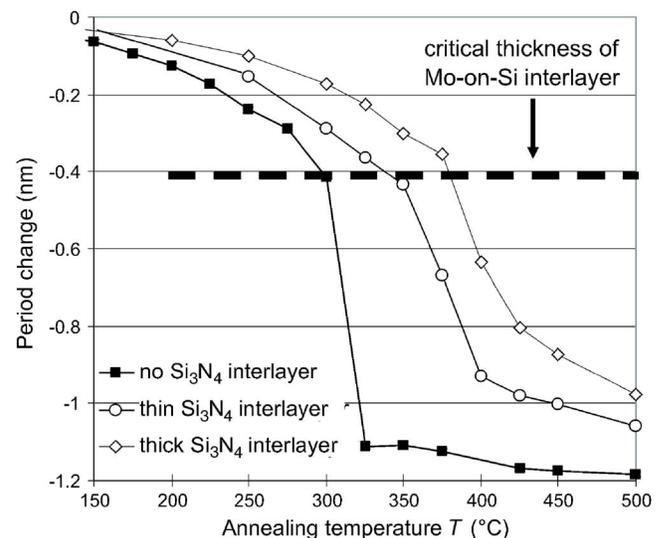


FIG. 5. (Color online) Period compaction during annealing up to 500 °C for multilayers with different Si_3N_4 thicknesses. The same period compaction is observed before the transition, suggesting a critical value for the thickness of the interface before crystallization takes place.

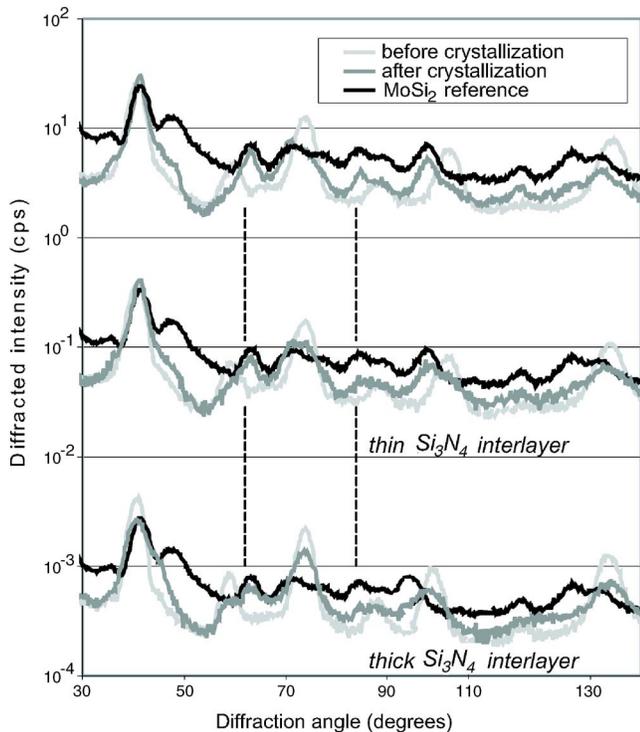


FIG. 6. (Color online) The x-ray diffraction spectra (WAXRD) of annealed Mo/Si multilayers with thin and thick Si_3N_4 layer in top of each Si layer, compared to a Mo/Si without Si_3N_4 layers. The $h\text{-MoSi}_2$ spectrum (black line) is used to identify in these multilayers the presence of this silicide. The vertical dotted lines point to the $h\text{-MoSi}_2$ diffraction peaks where the presence of crystalline Mo-on-Si interlayer is visible from the temperature corresponding an interlayer thickness equal to 2 nm (light gray line) to the next successive annealing temperature corresponding an interlayer thickness higher than 2 nm (dark gray line).

formation by passivating the Si surface. The multilayers studied here consist of 3 nm Mo and 4 nm Si after deposition. To form $h\text{-MoSi}_2$ over the entire multilayer, all Si should be consumed due to the Mo:Si ratio of 1:3 in MoSi_2 . Therefore, in the case of thick (>2 nm) Si_3N_4 layers, after N treatment, from the original 4 nm Si layer thickness, less than 2 nm remains. This would lead to much less $h\text{-MoSi}_2$ formation, which is also visible in the crystallographic spectrum.

We attribute the multilayer crystallization absence to the presence of N in the system based on previously reported delay effects on crystallization by some materials, such as N, O, and Ar.²⁰ Due to the defects created by these elements in the original structure, the crystallization is mitigated. This is in agreement with the $h\text{-MoSi}_2$ formation in multilayers with thick Si_3N_4 layers, where more N is present in the system and, consequently, the crystallization into $h\text{-MoSi}_2$ is less observed in the crystallographic pattern in Fig. 6 simultaneously with significantly more crystalline Mo left after crystallization than in the case of thin Si_3N_4 layers or no Si_3N_4 layers.

IV. CONCLUSIONS

Mo-Si multilayers, consisting of 50 periods of Mo and Si, were deposited by electron-beam evaporation and sequentially annealed up to 500 °C. From TEM, we show that

an asymmetry exists between the as-deposited Mo-on-Si (1 nm) and Si-on-Mo (0.5 nm) interfaces. During thermal treatment, diffusion is found to take place preferentially through the thick Mo-on-Si interface. We find that the cause of this higher diffusion rate is the same before and after annealing, and consists of a different mobility of the Mo atoms linked by the crystalline structure at the bottom (amorphous intermixture) and the top (polycrystalline film) of the deposited Mo layers.

At enhanced temperatures, the formation of amorphous silicide interfaces continues until a critical thickness of approximately 2 nm is reached, whereupon crystallization of the interface takes place. Diffusion increases along the crystalline grains of the silicide interface and the process of $h\text{-MoSi}_2$ formation continues until all available Si is consumed, resulting in a sharp phase transformation to $h\text{-MoSi}_2$ for the multilayer annealed at temperatures higher than 300 °C.

The dependence of the phase transition on a critical thickness, instead of on a critical temperature, was validated by experiments using Si_3N_4 passivation layers of different thickness to reduce diffusion at the Mo-on-Si interface. All systems exhibit the same behavior, namely, an abrupt enhanced diffusion upon annealing at the Mo-on-Si interface, simultaneously with interface crystallization linked to its critical thickness.

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