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Correlation between surface whisker growth and interfacial precipitation in aluminum thin films on silicon substrates

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Abstract When subjected to thermal excursions, aluminum thin films on silicon substrates often show whisker or hillock growth on the film surface, along with formation of Si precipitates at the interface. This study demonstrates that the two effects are related, and that interfacial Si precipitation directly influences the growth of Al whiskers on the film surface during isothermal annealing at 300–550 °C. The density of whiskers and hillocks not only increases with increasing annealing temperatures where the film is under greater compressive stress, but also during longer hold times which should relieve the stress. At high temperatures and long annealing times, extensive Si precipitation, eventually leading to a bi-modal precipitate size distribution, occurs continuously at the interface. The total amount of Si precipitates far exceeds the solubility limit of Si in the Al thin film, and can generate enough compressive stress in the film to drive surface whisker growth. By continuously augmenting film stress, interfacial Si precipitation supplies the driving force for whisker/hillock formation on the Al-film surface.

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Introduction

Aluminum films deposited on silicon substrates are known to be susceptible to the growth of whiskers and hillocks on the film surface following thermal excursions [1–6]. In addition, during heat treatment of Al films on Si, dissolution pits may form in Si due to rapid interdiffusion of Al and Si in localized regions, forming Al 'spikes' [7–10]. Furthermore, during heat treatment, Si may dissolve uniformly into the Al film, and re-precipitate at the interface during cooling, further affecting the interfacial properties [7–13]. Dissolution pitting and Si precipitation have been frequently observed together following isothermal heat treatment at high temperatures (250–500 °C) [e.g., 11–13], while whiskers and hillocks on the film surface has been widely observed following thermal cycling. Although Si precipitation on the substrate surface, as well as whisker/hillock formation on the film surface, can all occur during thermal cycling, a correlation between these effects has not been reported to date.

Whiskers and hillocks grow from Al films on Si substrates in order to relieve in-plane compressive stresses which build up at high temperatures, under conditions where a surface oxide layer prevents stress relaxation via surface diffusion [1–6]. Compressive stresses may form in films due to thermal expansion mismatch between film and substrate, as well as due to the formation of interfacial reaction products [14, 15], or oxide formation in the film [16]. In the absence of an oxide film, surface diffusion on the free surface relieves the film stress with no change in the film surface topography [2, 3]. However, when a protective oxide layer is present, compressive stress accumulates in the film until metal whiskers extrude out through localized cracks, which may form either due to out-of-plane stress gradients produced by local textural perturbations among the oriented columnar grains in the film [15], or due to stress-driven diffusional accumulation of Al at specific locations of the film–oxide interface [17].

In addition to whisker formation during heating, precipitates may form at Al/Si or Al/SiO_2 interfaces during cooling [3, 11–13, 17–21]. Si dissolves into Al uniformly as well as preferentially from specific locations on the substrate, and precipitates out epitaxially at the interface due to supersaturation during cooling [3, 11–13, 17, 18], heterogeneously nucleating at either grain boundaries in the film [18], at re-entrant corners constituted by oxide cuts on the Si substrate [12], or next to dissolution pits on Si [11]. The preferential dissolution of Si into Al, which precedes epitaxial re-growth of Si at the interface, has been ascribed to local compressive stress concentrations in the film due to sample configuration [12], or to the presence of substrate surface defects [11]. There is also some evidence that Si precipitates can form at the interface due to a reduction reaction between Al and surface oxides on Si [19, 21], or from amorphous silicates formed at Al_2O_3–Si interfaces [20].

In this paper, we report on a correlation between Al whisker formation and interfacial Si precipitation and other associated artifacts during heat treatment at temperatures ranging from 382 to 550 °C.

Experimental procedure

Samples were fabricated by thermally evaporating Al films of 0.75 μm thickness on polished undoped single crystal Si substrates with {100} orientation at 200 °C at a chamber pressure of 2×10^{-7} torr. Prior to deposition, the Si substrates were cleaned using standard microelectronic practice (de-greased sequentially in trichloroethylene, acetone, and iso-propanol, and de-oxidized in 10% HF, rinsed, and blow-dried). Immediately after deposition, the films and substrates were removed from the deposition chamber and placed in a furnace where they were annealed for various times in flowing 98% Ar 2% H_2 atmosphere. The annealing temperatures ranged from 300 to 550 °C. After annealing, the samples were slowly cooled in the furnace by losses to ambient (cooling rate ~3–8 °C/min).

After removing the samples from the furnace, the film surfaces were examined by scanning electron microscopy (SEM) and atomic force microscopy (AFM). Later the Al films were removed from the Si substrates by etching with 25 wt% H_3PO_4 at 60–70 °C. It was determined by examining polished Si substrates before and after immersion in 25 wt% H_3PO_4 that this acid did not detectably affect the silicon substrate. After removing the films, the bare Si substrates were again examined by SEM and AFM.

For transmission electron microscopy (TEM) examination, sandwich-samples were constructed by diffusion bonding the Al-film sides of two as-deposited samples with each other at 550 °C for 2 h at a pressure of 7 MPa in high vacuum. Subsequently, cross-sectional TEM samples were prepared from this sandwich by cutting out a wedge using a Ga-ion focused ion beam (FIB). The Al–Si interfaces in the samples were characterized in the TEM at an accelerating voltage of 200 kV, and via energy dispersive X-ray spectroscopy (EDS) using an electron probe size of 10 nm.

Results and discussion

Film surface effects

In the as-deposited state and following heat treatment for 1 min at 382 °C, no evidence of hillock or whisker formation was apparent on the film surface. But after heating for 2 h at 382 °C, whiskers and hillocks started appearing, as shown in Fig. 1a. With isochronal heating at increasing temperatures (2 h at 382, 427, and 550 °C), an increasing density of whiskers and hillocks were formed (Fig. 1a–c). This is principally due to faster diffusional transport at higher temperatures.[1] Since whiskers were not observed after 1 min at 382 °C, but appeared after a 2 h hold, it is clear that whisker/hillock growth can occur under isothermal conditions, where creep relaxation processes are expected to relax the compressive stresses which provide the driving force for whiskering. This is supported by the observation that when the sample heat treated at 382 °C for 1 min (which did not produce significant hillocking) was re-heated to only 300 °C for 18 h, a significant hillock density was observed. An additional heat treatment for 50 h at 300 °C led to a further substantial increase in the density of whiskers, and especially hillocks. This suggests that a mechanism which regenerates compressive stresses is operative during the isothermal hold.

Figure 2a and b shows typical morphologies of the whiskers and hillocks, based on the sample treated for 2 h at 427 °C. Some of the whiskers were straight, while others were kinked. Some faceting was observed on the whiskers as well as the hillocks. These facets correspond to the Al{111} planes, the growth direction of the whiskers being ⟨110⟩ [6].

Substrate surface effects

Figure 3a and b shows the surface of the Si substrate after the Al film has been dissolved away, following isothermal

[1] The thermally induced film stress also becomes more compressive with increasing temperature, but levels off beyond ~ 200–250 °C due to plastic yielding and creep of the film [e.g., 6].

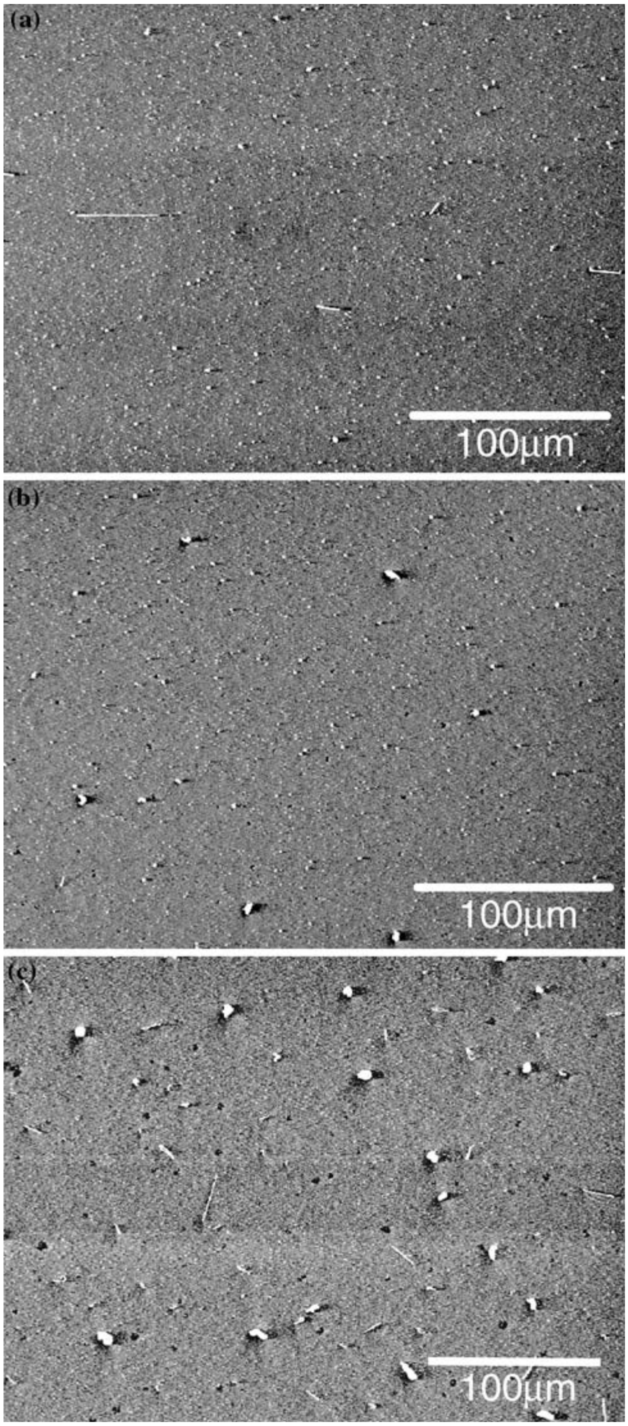

Fig. 1 SEM images of the Al-film surface after 2 h at **a** 382 °C, **b** 427 °C, and **c** 550 °C

treatments at 427 °C for 1 min and 2 h, respectively.[2] After 1 min at 427 °C, the surface of the Si substrate is smooth, but several highly faceted dissolution pits are observed

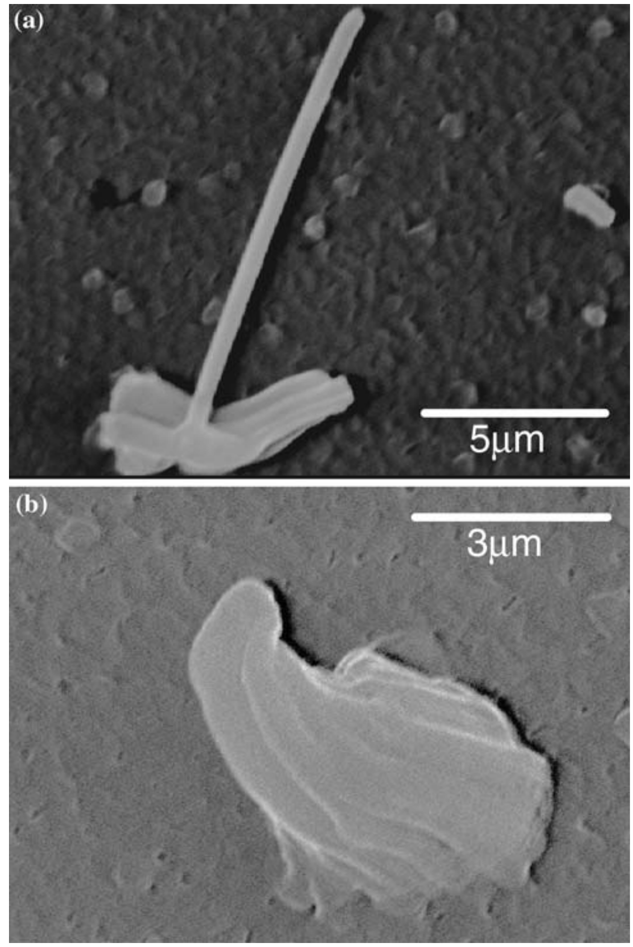

Fig. 2 Morphology of the whiskers (**a**) and hillocks (**b**) formed on the Al film after 2 h at 427 °C

where Al had 'spiked' into Si. The facets of the pits correspond to the {111} planes of Si, which are inclined to the {100} surface at ~55°, while the edges of the pit correspond to the two perpendicular ⟨110⟩ directions which lie on the substrate surface [11]. After 2 h at 427 °C (Fig. 3b), the pits are less sharply faceted, and the substrate surface is generally rough as compared to Fig. 3a. The roundness of the pits is indicative of extensive dissolution of Si into Al during the hold at this temperature, and the associated surface roughness is due to prolific precipitation of Si on the substrate surface. Thus, when there is limited dissolution of Si into the Al film without precipitation (e.g., at 1 min at 427 °C), no whisker or hillock growth is noted. Conversely, substantial Si dissolution followed by precipitation is associated with whisker/hillock growth on the Al-film surface.

Figure 4a and b shows the Si-substrate surface following film-dissolution after a 2 h treatment at 550 °C. At this point, the pits are a little larger and more rounded than those obtained after the 427 °C treatment. Importantly, the substrate is populated by an array of coarse Si precipitates,

[2] For reference, the sample treated for 1 min at 427 °C showed no film-surface whiskering, whereas the one treated for 2 h showed considerable whisker growth.

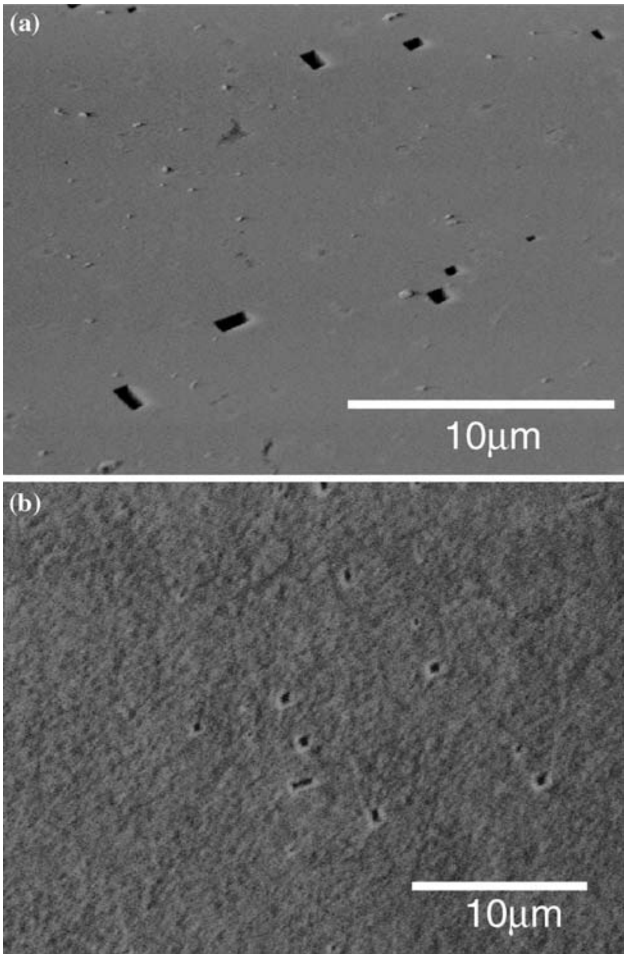

Fig. 3 Surface of the Si substrate after the Al film has been dissolved away, following heat treatment at 427 °C for **a** 1 min and **b** 2 h

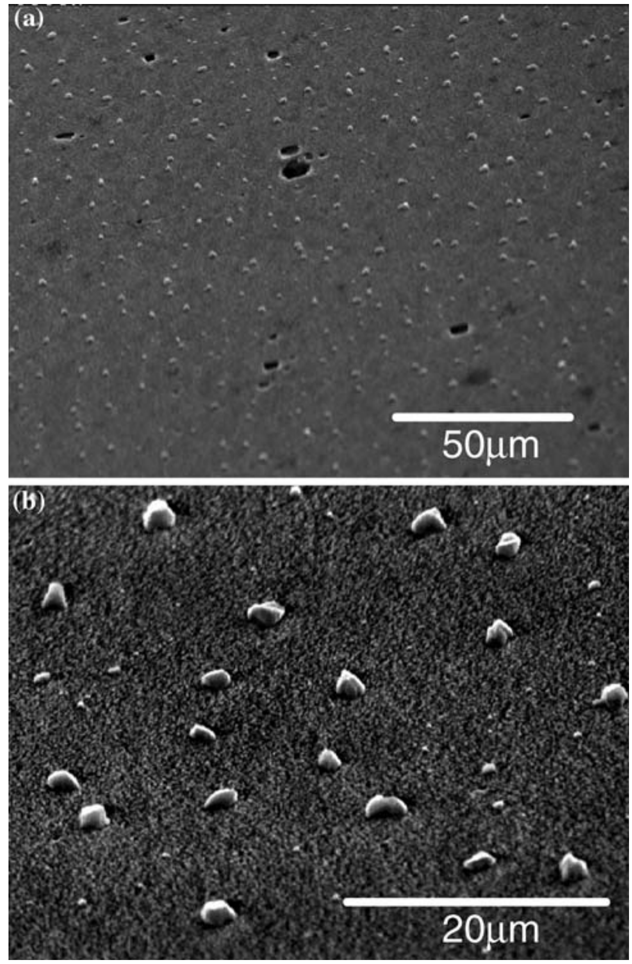

Fig. 4 Si-substrate surface following film-dissolution after a 2 h treatment at 550 °C, showing irregularly shaped pits (**a**) and coarse Si precipitates (**b**)

with a nearest neighbor spacing of ~10 μm. Figure 4b which shows a higher magnification micrograph of the same substrate, shows that the Si precipitates are ~1–3 μm in size. In addition to these coarse precipitates, a very fine precipitate densely populates the entire substrate surface, similar to that after 2 h at 427 °C (Fig. 3b). This is more clearly shown in the AFM image of the Si-substrate surface in Fig. 5a, where nearly 50% of the substrate surface area is observed to be covered by the precipitates.[3] The AFM line scan of the substrate surface shown in Fig. 5b reveals that the fine precipitates are about 40–80 nm thick. Thus, following the 550 °C/2 h treatment, a bi-modal distribution of Si precipitates is formed on the Si substrate (i.e., at the interface), with the fine precipitates being 0.2–0.4 μm in diameter and 40–80 nm thick, whereas the coarse precipitates are roughly 1–3 μm in diameter, and 0.5–0.7 μm in height. Since the Al thin film is only 0.75 μm thick, the large Si precipitates penetrate the entire Al film, while the small precipitates are all at the interface only. Although localized precipitation of epitaxial Si along the grain boundaries of Al thin films on Si has been observed frequently [3, 11–13, 17, 18], the bi-modal distribution observed in this study, and in particular, the prolific fine-scale precipitates, have not been reported earlier.

Based on the size and area-coverage of the small precipitates, it can be estimated that the Si precipitates occupy approximately 3.3 volume percent of the 0.75 μm thick Al film, whereas the large precipitates occupy around 5.5 volume percent of the film. Thus, after the 550 °C/2 h treatment, the fine and coarse Si precipitates together occupy ~8.8 volume percent of the film, corresponding to a weight percent of 7.6 in the Al film. This is much greater than the equilibrium solubility limit of 1.46% Si in Al at 550 °C [22]. Therefore, it is clear that most of the Si precipitates could not have formed due to Si dissolution into Al at high temperature and subsequent precipitation due to

[3] Since AFM images tend to overestimate the size of the precipitates due to the finite curvature of the cantilever tip, the actual precipitate coverage is estimated to be ~40% of the substrate surface area.

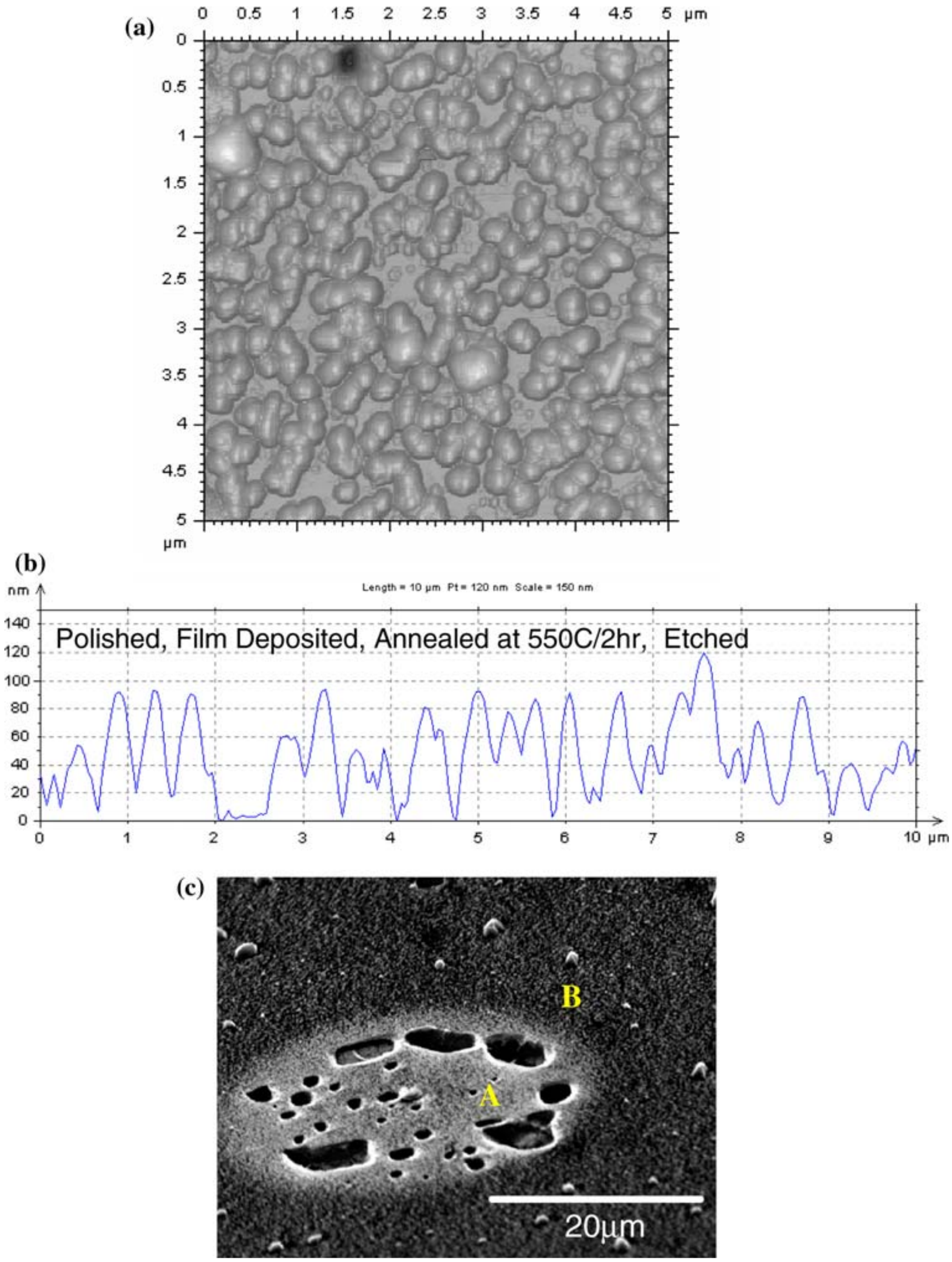

Fig. 5 **a** AFM image and **b** AFM line scan of the Si-substrate surface after removal of the Al film following the 550 °C/2 h treatment. **c** SEM image showing pit-clusters in the sample treated at 550 °C for 2 h

super-saturation during cooling. Clearly, in contrast to the commonly held view that Si precipitation occurs during cooling [11–13], most of the Si precipitation occurs during the high temperature dwell, particularly when the temperature is above 400 °C.

Figure 5c shows an additional artifact, which was observed at a few scattered locations on the substrate after the 550 °C/2 h treatment. In addition to the coarse and fine Si precipitates and isolated pits discussed above, a few clusters of relatively large pits were observed. These clusters appeared in roughly circular patterns, and contained both large and small pits, the smaller ones being more distinctly faceted. AFM scans revealed that the large pits were nearly 1 μm deep. Importantly, it is apparent that the substrate in the immediate vicinity of the cluster (region A in Fig. 5c) is largely devoid of the small Si precipitates, although just outside the clusters (region B in Fig. 5c), fine Si precipitates formed in copious quantities.

Such pit-clusters have not been reported before. It is likely that they form near occasional strain centers (i.e., regions with high defect concentration) that exist at or close to the substrate surface [11], where Si can rapidly dissolve into the Al film at high temperatures, producing regions of high Si concentration in the Al. Concurrently, Al diffuses into the pits, forming Al spikes into the substrate. Because of extensive interdiffusion, these regions of the Al film become highly defected, and an amorphous interfacial phase is formed on the Al side of the interface. Upon cooling, this amorphous phase undergoes transition into a glass, thereby retaining the excess Si, thus explaining the absence of Si precipitation within the clusters. Amorphous interfacial regions have been previously observed to form at bulk Al–Si interfaces during diffusion bonding at 833 K, penetrating to various depths (30–200 nm) on the Al side, depending on the local surface oxygen concentration [23]. Interfacial amorphous Al-silicate may also form via reaction between the [100]Si and thin alumina films [20], which exist at the Al–Si interface. During etching, the amorphous region gets etched away along with the Al film, revealing the structures shown in Fig. 5c.

One possible mechanism by which Si precipitation may occur at high temperature is by reduction of the native SiO_2 layer which exists on Si prior to Al deposition. During heat treatment after Al deposition, the following reaction may occur [21]:

$$SiO_2 + Al \rightarrow Al_2O_3 + Si \quad (1)$$

leading to the growth of epitaxial Si at the interface [21].

The following TEM results provide some support for this mechanism. Figure 6a shows a bright field image of the region near an Al–Si interface. A thin (25–40 nm) interfacial layer, which contains Al, Si, and O, is present all along the interface, as evident from the EDS spectrum. A large Si precipitate is present on the Al side (outlined with dashed line A), as apparent from the selected area diffraction pattern (SADP) obtained from it (Fig. 6b). The broken lines B and C outline other smaller Si precipitates behind the large precipitate. Comparing Fig. 6b with the SADP for the Si substrate shown in Fig. 6c, it is clear that the orientations are nearly identical, indicating that the precipitate is epitaxial. Just under the Si precipitate, a small dissolution pit (encircled with a dashed line) is observed on the Si side of the interface. The pit is filled with Al (i.e., it corresponds to an 'Al spike'), as evident from the SADP obtained from within it (Fig. 6d). Figure 7 shows a high magnification bright field image of a region of the interface with a Si precipitate and a dissolution pit, along with X-ray maps of Al, Si, and O. From the elemental EDS maps, it is apparent that the interface consists largely of Al and O, with very little Si, suggesting possible oxidation of Al along with precipitation of Si according to Eq. 1. However, the amount of aluminum oxide under the precipitate is clearly not sufficient to for the entire Si precipitate to have formed via the reaction in Eq. 1. Furthermore, although the Si precipitate appears to be associated with the dissolution pit, not enough Si could have dissolved from the pit to form the entire precipitate. Finally, it is worth noting that the growth of native SiO_2 on cleaned Si substrates saturates at a thickness of only ~12–15 Å [24]. Therefore, the reaction in Eq. 1 cannot produce enough Si and/or Al_2O_3 to account for all the fine precipitates observed in Figs. 4 and 5a.

The above observations suggest that the extensive Si precipitation observed here occurs via a combination of four possible mechanisms: (1) reduction of the native oxide layer on Si during isothermal holding, (2) continuous solution of Si uniformly from the substrate surface into the Al film and re-precipitation thereof during the hold, (3) localized dissolution of Si at near-surface strain centers

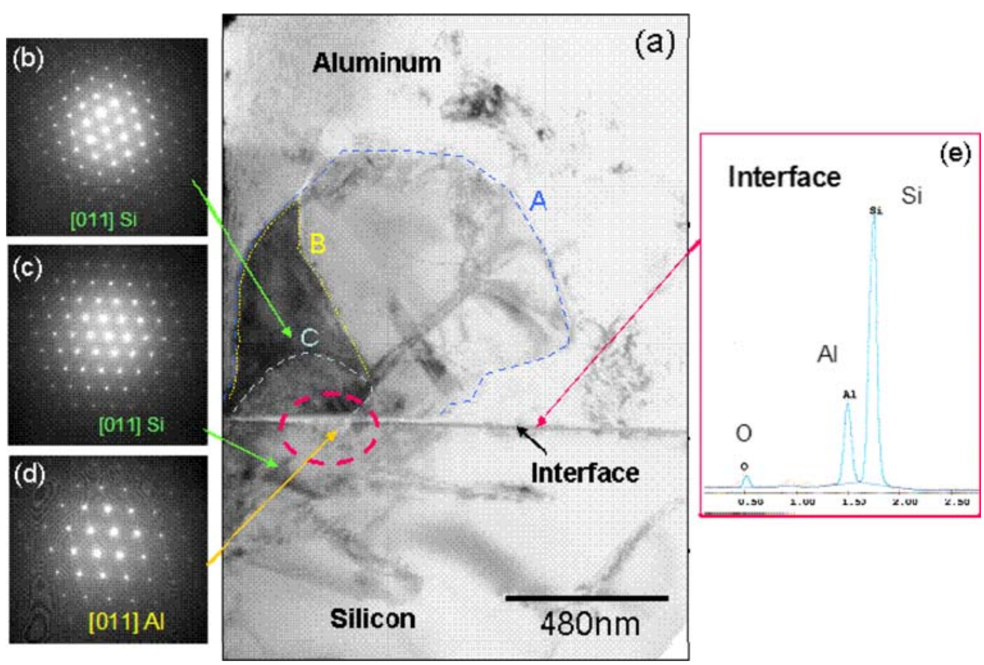

Fig. 6 **a** TEM bright field cross-sectional image of Al–Si interfacial region after 2 h at 550 °C. Selected area diffraction patterns from the Si precipitate (**b**), Si substrate (**c**), and Al spike corresponding to pit in Si (**d**). **e** EDS spectrum showing Al, Si, and O at the interface

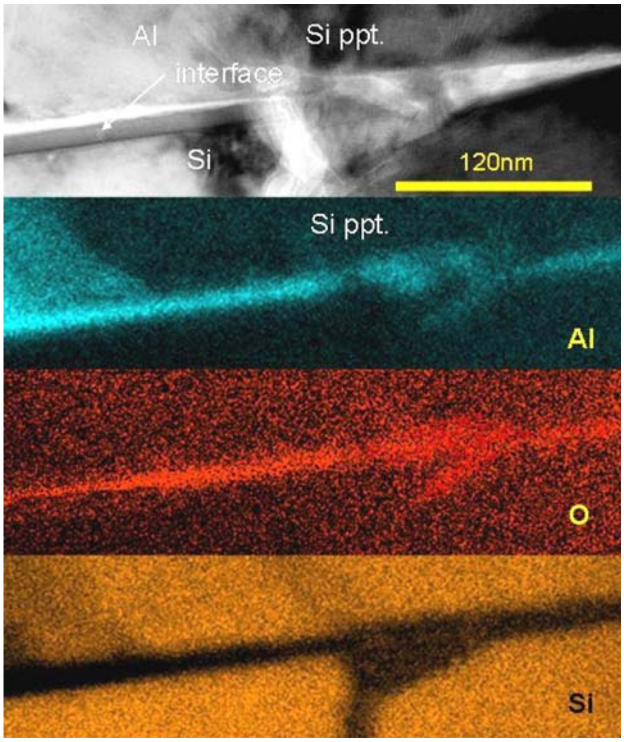

Fig. 7 Bright field TEM image of the interfacial, with elemental EDS maps of Al, O, and Si. A big Si precipitate is present on the Al side of the interface, and a small Al filled Si dissolution pit is seen on the Si side. The top part of the pit contains significant O

(with concomitant pitting) and re-precipitation during holding, and (4) additional Si precipitation due to supersaturation during cooling. From the evidence presented above, it appears that a majority of the Si precipitates is produced by mechanisms 2 and 3, with only a small proportion being produced via mechanisms 1 and 4.

While the rationales for mechanisms 1 and 4 are obvious, the driving forces for mechanisms 2 and 3 are less readily apparent. It is known that Si diffuses rapidly along grain boundaries in Al thin films at temperatures as low as 300 °C because this results in a reduction of the grain boundary energy of Al [9]. At higher temperatures, large amounts of Si may dissolve into the Al film along with simultaneous nucleation and growth of Si crystallites in Al [9, 25]. Recently, experiments have shown that the diffusion of Si into Al thin films during continuous isothermal annealing can be so extensive that it can result in complete positional exchange of Al and Si thin films in Si/Al bi-layers deposited on a Si substrate [26, 27]. It has been further noted that the driving force for this layer-exchange mechanism increases with increasing with temperature [26, 27]. This is consistent with the present observations, which provide evidence of substantial diffusion of Si into Al during high temperature isothermal holds, which is far greater than that permitted by the equilibrium solubility of Si in Al at the relevant temperature. Therefore, we may conclude that interdiffusion of Si into Al can occur continuously without being limited by the solubility limit of Si in Al, because of simultaneous precipitation of Si within the Al film.

Correlation between film surface and substrate surface effects

As discussed earlier, hillocking/whiskering at the film surface occurs in order to relieve compressive stresses in the film when a surface oxide layer inhibits stress relaxation by surface diffusion [1–6]. Since the film stress becomes increasingly compressive beyond $\sim$200 °C, it is generally thought that hillocking/whiskering occurs primarily during heating. However, as discussed above, the extent of whiskering and hillocking clearly increases with increasing annealing temperature, as well as increasing annealing time at a given temperature. Since during a high temperature hold, creep relaxation mechanisms are likely to relax the compressive stresses in the Al thin film rapidly, a mechanism for re-generation of compressive stresses must be operational in order to lead to whiskering/hillocking during isothermal annealing. This mechanism is provided by the continuous formation and growth of Si precipitates within the naturally self-passivated (and therefore constrained) Al film during heat treatment. Thus, the Si precipitation at the interface within the Al film during heat treatment has a direct correlation with the hillocking/whiskering at the film surface, similar to the manner in which the continuous formation of interfacial Cu_6Sn_5 intermetallics influence whiskering in tin platings on Cu substrates [14, 15].

It was noted earlier that the Si precipitates constitute a volume fraction (V_{Si}) of around 0.08 of the 0.75 μm thick Al film following a 550 °C/2 h. heat treatment. If we assume that the Al film is self-passivated and cannot expand to accommodate the Si crystallization, the Si precipitates would introduce a compressive volume strain ($\Delta V/V$) in the film, which is roughly equal to $-V_{Si}$. This would produce a compressive equi-biaxial plane stress in the film, given by $\sigma = -[E/(1-\nu)](V_{Si}/3)$ where E and ν are the Youngs modulus and Poisson's ratio of Al, and $E/(1-\nu)$ represents the biaxial modulus of the Al film. Estimating E at 550 °C to be 38 GPa [28] and ν to be 0.3, and assuming V_{Si} to be 0.08 gives $\sigma = -1.44$ GPa. Clearly, this level of stress cannot be supported in the film, and must be relieved by yielding, cracking of the surface oxide film and the extrusion of whiskers and hillocks. Thus, it is evident that the whiskering and hillocking observed on the film surface is directly related to the Si crystallization in the Al film. This is supported by the observation that as the heat treatment temperature or time is increased, both hillocking and Si precipitation increase.

Summary

Artifacts arising on the film surface and at the interface in Al thin films deposited on Si substrates during heat treatment have been studied. During heating, thermally induced compressive stresses in the film are relieved by the formation of whiskers and hillocks on the film surface. Evidence has been presented that most of the hillock and whisker formation occurs during the isothermal hold. Following heat treatment at low temperatures (<300 °C) or short times, few whiskers or hillocks are observed on the Al-film surface, while the Si-substrate surface remains generally smooth, but shows faceted dissolution pits where Al has 'spiked' into the Si. During heat treatment at progressively higher temperatures, the substrate surface becomes rougher because of fine Si precipitation, commensurate with increased film-surface whisker/hillock growth. After heat treatment at 550 °C, when the whisker/hillock density is very high, the substrate surface is densely populated by a bi-modal precipitate distribution, the total amount of Si precipitates far exceeding the solubility limit of Si in the Al thin film. Thus, Si precipitation on the substrate surface bears a direct correlation with whisker/hillock formation on the Al-film surface. It is reasoned that the volume expansion associated with Si precipitation provides a mechanism to continuously regenerate compressive stresses within the film during annealing to drive whisker growth. A new substrate surface artifact, constituting clusters of dissolution pits with little Si precipitation within the clusters was also observed following heat treatment at 550 °C, suggesting non-equilibrium retention of Si in localized amorphous regions at the interface.

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