Directed evolution of $\alpha$-grains in thin metastable-$\text{Al}_2\text{O}_3$ films deposited on Si(100) after post-deposition annealing

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A 250-nm-thick $\text{Al}_2\text{O}_3$ film was deposited on a Si(100) by a radio-frequency magnetron sputtering and annealed at 1100 °C for various periods of time in air. In the matrix composed of fine metastable-$\text{Al}_2\text{O}_3$ grains of 50–100 nm in diameter, large $\alpha$-$\text{Al}_2\text{O}_3$ grains of about 2–10 μm in diameter appeared, interestingly aligning themselves along various directions. The compressive stress developed in the alumina films because the thermal expansion coefficient of the film was higher than that of the silicon substrate. The stress distribution in the film is expected to be inhomogeneous due to some discontinuities or defects, such as arrays of dislocation pits and steps on the surface of the Si substrate, which could be generated by intersections of the substrate surface and the slip and twin planes in the Si substrate. The enhanced phase transformation into $\alpha$-$\text{Al}_2\text{O}_3$ along various directions is suggested to arise from such discontinuities or defects.

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**1. Introduction**

Alumina ($\text{Al}_2\text{O}_3$) has many metastable polymorphs, such as $\gamma$ (cubic, defect spinel structure), $\delta$ (tetragonal), $\theta$ (monoclinic), and $\kappa$ (orthorhombic), in addition to the thermodynamically stable $\alpha$-phase. While heated, metastable phases undergo a series of polymorphic phase transformations to form the stable $\alpha$-phase. The transformation into $\alpha$-$\text{Al}_2\text{O}_3$ is irreversible and typically occurs at above 1000 °C by various vapor deposition processes, such as chemical vapor deposition and physical vapor deposition (PVD).

For PVD, the phase transformation sequence is reported to be amorphous or a mix of amorphous and $\gamma \rightarrow \theta + \delta \rightarrow \alpha$ [5,12–14]. Chou and Nieh [14] studied phase transformation in $\text{Al}_2\text{O}_3$ films deposited on NaCl substrates by radio-frequency (rf) magnetron reactive sputtering. After the deposition, the NaCl substrates were dissolved away, and the free-standing films were annealed at temperatures between 800 and 1200 °C. The film annealed at 1200 °C revealed an abnormal grain growth of $\alpha$-phase in the nanometersized $\gamma$-$\text{Al}_2\text{O}_3$ matrix, which might be a mix of $\gamma$, $\delta$, and $\theta$-phases, as suggested by Levin and Brandon based on the transmission electron microscopy (TEM) diffraction pattern [1]. Since the substrates were removed before annealing, the effect of the film/substrate interface on phase transformation during the annealing was excluded.

For a thin film deposition, stresses are developed in the film and the substrate because of differences in lattice parameter or in thermal expansion coefficient between the film and the substrate [15–18]. Such stress fields are closely related to the formation of self-assembled nanodot arrays on various films. Wu et al. [17] observed that NIS quantum dots aligned with cross-hatch patterns on epitaxial $\text{Si}_{0.7}\text{Ge}_{0.3}$ layers on Si(001) substrates. Ge islands were also found to reveal regular arrays on SiGe buffer layers on Si(100) substrates [18]. The alignments were suggested to result from stress fields of the step bunching on the $\text{Si}_{0.7}\text{Ge}_{0.3}$ surface [17] or of the misfit dislocation network in the interface between the SiGe and Si layers [18]. It will be interesting to explore how the stress fields influence the phase transformation and grain growth in $\text{Al}_2\text{O}_3$ thin films.

We deposited an $\text{Al}_2\text{O}_3$ thin film on Si(100) by a rf magnetron sputtering as a model system. The as-deposited film was identified as a mix of amorphous and $\gamma$-phase. When annealed at a temperature of 1100 °C in air, $\alpha$-$\text{Al}_2\text{O}_3$ grains of 2–10 μm in diameter were formed in the matrix composed of fine metastable-$\text{Al}_2\text{O}_3$ grains of 50–100 nm in diameter and were aligned in rows in the matrix film. The purpose of this study is to suggest a model for the aligned evolution of $\alpha$-$\text{Al}_2\text{O}_3$ grains.

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2. Experimental procedure

A 250-nm-thick Al₂O₃ film was deposited on a Si(100) 4-inch wafer (525 µm thick) by a rf magnetron sputtering system with a base pressure of 0.67 mPa. The sputtering was done using an Al₂O₃ target with 99.995% purity at an rf power of 500 W at room temperature in Ar ambient. The Ar flow rate was 100 sccm, and the working pressure in the chamber was 0.29 Pa. The film-deposited wafer was cleaved into 10×10 mm² square specimens with the edges parallel to the natural <110> cleavage directions. Each specimen was annealed in air at 1100 °C for 1 h–48 h. Microstructures were characterized by TEM in a JEM4010 (JEOL Ltd., Co., Japan). Surface images were obtained using a field-emission scanning electron microscope (FESEM) (JSM 6330F, JEOL Co., Ltd., Japan). For phase identification, X-ray diffractometry (XRD) was employed at Bruker D8 DISCOVER (Bruker AXS GmbH, Karlsruhe, Germany; Cu Kα radiation, 40 kV, 30 mA). Grain orientations were determined using a commercial electron backscatter diffraction (EBSD) setup (INCACrystal, Oxford Instruments, UK) equipped in a FESEM (JSM 6500F, JEOL Co., Ltd., Japan).

3. Results

The as-deposited film was amorphous (Fig. 1). Fig. 1 shows an area of the interface between the film and substrate. When annealed at 1100 °C for 48 h, as shown in Fig. 2, the film was fully crystallized, appearing to be composed of γ and θ. The peak at 2θ = 61.7° corresponds to Si(400) Kα reflection. At 1100 °C, rows of large grains appeared along various directions (Fig. 3). The directions indicated by arrows were indexed referred to the <110> edge directions of the cleaved, film-deposited wafer specimen. The average diameter of the large grains increased from 2 µm to 10 µm with increasing annealing time from 1 h to 48 h. The change in grain size is clearly seen in band contrast images (Figs. 4–6). The matrix of the annealed structure fully consisted of grains of 50–100 nm in diameter, whose size remained unchanged during annealing. Fig. 3(b), taken from the specimen annealed for 1 h, shows cracks in a large grain. In addition, its surface appeared rugged, which might be a sign of the beginning stage of surface faceting. After annealing for 48 h, surface faceting of the large grains became clear as shown in Fig. 3(f).

The phase identification of the large grains after annealing at 1100 °C was carried out using EBSD. They were easily identified as α-phase. Phases of the small matrix grains were not identified by EBSD because of their small grain size. Band contrast maps from the specimens annealed at 1100 °C (Fig. 4–6) show darker grey pixels near the grain boundaries, indicating low pattern quality due to the overlap of backscattered diffraction patterns from the grains adjoining the grain boundaries. The local distributions of grain orientations are shown in (b) in Figs. 4–6. In the orientation maps, the grain colors represent crystallographic directions of the α-grains, which are the specimen surface normal direction (ND), the in-plane direction of the alignment of the α-grains (longitudinal direction, LD), and the other in-plane direction normal to LD (transverse direction, TD). A direction color key for the orientation maps is shown in Fig. 4. The two-dimensional orientation distributions in the orientation maps can be statistically represented by inverse pole figures ((c) in Figs. 4–6). The corresponding inverse pole figures are drawn referred to X0, Y0, and Z0, which correspond to TD, LD, and ND, respectively. As shown in Figs. 4–6, the texture of the α-grains is nearly random when annealing time is 1 h, and the density of the [0001] // ND orientation increases with increasing annealing time, while the two in-plane directions tend to cluster near or along the arc between [1010] and [0110], which are normal to [0001], indicating fiber texture of the α-grains. In the orientation maps (Figs. 4–6), the measurement points within an α-grain often vary inhomogeneously in color, even containing no information (thus appearing blank), possibly because of surface defects, such as cracks and surface faceting shown in Fig. 3(b) and (f).

4. Discussion

According to XRD shown in Fig. 2, the crystalline phases in the matrix after annealed at 1100 °C for 48 h appeared to be composed of γ and θ. XRD data obtained from specimens annealed for 1 h, 24 h, and 72 h (not shown here) were almost the same as for 48 h (Fig. 2), indicating that crystallization was fully completed before reaching 1100 °C. However, the XRD could not unambiguously identify phases, because the transition alumina structures have similar d-spacings. The very small size of the matrix grains (up to 100 nm in diameter) in the annealed structure as shown in Fig. 3(f) also seems to strongly indicate that the matrix is composed of γ-grains, because the γ-phase is known to be surface-energy-stabilized for small grain sizes, due to its low surface energy [19].

Although during annealing at 1100 °C the alignment of abnormally large α-Al₂O₃ grains was observed and their surface normal directions were close to [0001], the peak corresponding to the basal plane normal orientation (2θ = 41.67°; JCPDS No. 46-1212) did not appear, as shown in Fig. 2. This may be due to the poor relative XRD intensity of the basal plane of α-Al₂O₃. Furthermore, the area fraction of the transformed area was roughly measured at a low magnification of 100× to be as small as 5%, which also explains the absence of the α-Al₂O₃ peak.

The phase transformation from the amorphous Al₂O₃ phase into the stable α-Al₂O₃ phase needs activation energy. The activation energy is usually supplied by heating the system. Heating supplies the system with the thermal energy and the strain energy as well. The thermal energy is self-explainable and isotropic and, thus, cannot explain the anisotropic phenomenon, i.e., the alignment of the large α-grains, which is thus attributed to the contribution of the strain energy. Since the thermal expansion coefficient of the alumina film differs from that of the Si substrate, the film and substrate can be stressed during heating at 1100 °C. Assuming that the matrix is only
composed of γ-grains, the Al2O3 film would receive the compressive stress during annealing, because the thermal expansion coefficient of γ-phase is given by

$$\alpha_\gamma(T) = 2.6068 \times 10^{-6} + 1.1499 \times 10^{-8}(T - 298.15) \text{ at } T = 298.15 \text{ to } 900 \text{ K}$$

and that of Si is given by

$$\alpha_\text{Si}(T) = (3.725(1 - \exp[-5.88 \times 10^{-3}(T - 124)]) + 5.584 \times 10^{-4}T) \times 10^{-6} \text{ at } T = 120 \text{ to } 1500 \text{ K}.$$  

For lack of measured data, the thermal expansion coefficients of metastable alumina phases are assumed to be the same as that of γ-alumina. The thermal strains of γ-phase and Si are as follows:

$$e_\gamma = \int_0^T \alpha_\gamma(T) dT = 2.6068 \times 10^{-6}(T - T_0)$$

$$+ 1.1499 \times 10^{-8}[T^2 - T_0^2]/2 - 298.15(T - T_0)$$

$$e_\text{Si} = \int_0^T \alpha_\text{Si}(T) dT = [3.725(T - T_0) + (\exp[5.88 \times (124 - T) \times 10^{-3}]) - \exp(5.88 \times (124 - T_0) \times 10^{-3})]/(5.88 \times 10^{-3}) + 2.792 \times 10^{-4}(T^2 - T_0^2)] \times 10^{-6}. \quad (4)$$

Fig. 3. SEM micrographs of the films annealed at 1100 °C for 1 h (a, b), 24 h (c) and 48 h (d, e, f). Abnormally grown α-grains, appearing dark as compared with the matrix, are aligned along various directions. (b) shows cracks within an α-grain. (f) A higher magnification of the interface between an α-grain and fine matrix grains, where distinct faceting is revealed on the surface of the α-grain.
Setting $T_0 = 298.15$ K and $T = 1373.15$ K in the above equations, we obtain $e_f = 0.009426$ and $e_{Si} = 0.004599$. Therefore, the $\gamma$-phase layer is expected to be under compressive stress at 1100 °C.

The thermal stress of the $\gamma$-phase layer, $\sigma^\gamma$, can be given by

$$\sigma^\gamma = \frac{E^\gamma \varepsilon^\gamma}{1-\nu^\gamma}$$

where $E^\gamma$ and $\nu^\gamma$ are Young's modulus and Poisson's ratio, respectively, and $\varepsilon^\gamma (= e_{Si} - e_f)$ is the thermal strain of the $\gamma$-phase layer. Since the elastic constants of the $\gamma$-phase are not available, we use Young’s modulus (346.1 GPa) and Poisson’s ratio (0.25) of the $\alpha$-Al$_2$O$_3$ at 1350 K [22]. Then we obtain $\sigma^\gamma = -2.2275$ GPa, assuming that the alumina film coated Si(100) wafer behaves elastically. Because of the compressive stress, the specimen is curved as shown in Fig. 7.

**Fig. 4.** (a) Band contrast map obtained from EBSD analysis of the films annealed at 1100 °C for 1 h. The local distribution of crystallographic grain orientations is shown in (b). (c) The corresponding contour inverse pole figures in reference to X0, Y0, and Z0, which correspond to TD, LD, and ND, respectively.
The in-plane stress in the Si substrate, \( \sigma_s \), with respect to the neutral plane is given by Eq. (6) [23]

\[
\sigma_s(x_3) = \sigma_{s11}(x_3) = \sigma_{s22}(x_3) = -\frac{1}{R} x_3 \frac{1}{S_{11} + S_{12}} = -\beta x_3 \quad \text{with} \quad \beta = \frac{1}{R(S_{11} + S_{12})}
\]

(6)

where \( R \) and \( S_{ij} \) are the radius of curvature of specimen and the compliances of Si(100) wafer.

The force- and momentum-balance in Fig. 7 can be given by

\[
\int_{x_3=0}^{x_3} \sigma_s(x_3) dx_3 + \alpha^t t_f = 0 \quad \text{and} \quad \int_{x_3=0}^{x_3} \sigma_s(x_3) dx_3 + \alpha^t \sigma_s^t = 0.
\]

(7)

(8)

It follows from Eqs. (6) to (8) that \( \alpha = 2/3 \). It also follows from Eqs. (6) and (7) that

\[
\beta = \frac{1}{R(S_{11} + S_{12})} = \frac{6\alpha^t t_f}{t_s^2}. \quad \text{(9)}
\]

Setting \( \sigma_s^t = -2.2275 \times 10^9 \text{ Pa}, \ t_f = 250 \times 10^{-9} \text{ m}, \) and \( t_s = 525 \times 10^{-6} \text{ m}, \) we obtain \( \beta = -1.2122 \times 10^{10} \text{ Pa/m}. \) Therefore, \( \sigma_s(0) = 0 \) and \( \sigma_s(2t_s/3) = 4.243 \text{ MPa}. \) That is, the Si substrate near the film/substrate interface is under a tensile stress of about 4 MPa.

The critical resolved shear stress for dislocation generation at 1000 °C or above, which is in the same temperature range as in the present study, is 1–10 MPa [24] and that for dislocation movement is
3–20 MPa [25,26]. Therefore, dislocation generation and movement are likely to take place near the film/substrate interface of the Si substrate at 1100 °C. In other words, slip and twinning take place in the Si substrate by the dislocation generation and movement. Dislocations propagate along the slip planes of the \{11\text{I}\}<110> slip systems. Twinning is reported to be activated along the \{111\} and \{123\} planes [27,28].

For Si, four unique slip planes intersecting the (100) surface are the (111), (11\text{I}1), (1\text{I}11), and (\text{I}\text{I}11) planes. Dislocation lines running along the slip planes meet the (100) surface, forming discontinuities such as pits or steps along the intersection line of the surface and the slip planes. The intersection line directions on the (100) surface are determined by the cross product of the surface normal (i.e., [100]) and the four slip plane normals. The possible unique directions are [0\text{I}1] and [011], which cross each other at right angles. The twin planes intersecting the (100) surface produce the twin plane re-entrant edges [29] on the surface. The possible intersection line directions for (111) twin planes are the same as for the slip planes. The possible intersection line directions for \{123\} twin planes are [03\text{I}2], [032], [02\text{I}3], [023], [031], [031], [0\text{I}32], [013], [012], and [021].

Transformation of the Al$_2$O$_3$ film deposited on the Si(100) surface is influenced by the defects such as pits and steps on the Si substrate surface, which can act as stress raisers and increase accommodation strains between the Si substrate and the alumina deposit. The increase in strain gives rise to an increase in strain energy of the film, which in turn enhances the transformation into $\alpha$-phase. In other words, the discontinuities or defects along the intersection lines are likely to induce higher strain energies in the Al$_2$O$_3$ deposits on them. Therefore, the metastable-Al$_2$O$_3$ deposits on the defects are likely to be transformed into stable $\alpha$-Al$_2$O$_3$ during annealing earlier than those on other regions because the deposits on the defects are supplied by strain energy in addition to thermal energy. It is interesting to note that the rows of $\alpha$-grains in Fig. 3 are along the intersection lines calculated above and appear at 1100 °C, which is lower than 1200 °C as observed for the free-standing Al$_2$O$_3$ films by Chou and Nieh [14]. The decrease in critical temperature for nucleation and grain growth of $\alpha$-phase is attributed to the strain energy contribution caused by the surface defects.

The surface of the Si(100) wafer used in the study was thoroughly examined before deposition by optical and scanning electron microscopy (SEM) and X-ray diffraction (XRD) to determine the perfection of the Si substrate. The Si substrate was found to be highly (100) oriented with a maximum misorientation of 0.1°.

**Fig. 6.** (a) Band contrast map, (b) orientation maps, and (c) inverse pole figures obtained from the films annealed at 1100 °C for 48 h.

**Fig. 7.** Alumina film-deposited silicon substrate is curved due to stress in film. $\alpha t_f$ and $t_s$ indicate neutral plane, film thickness, and substrate thickness, respectively.
microscopies. However, structural singularities such as surface steps or scratches, which were likely to induce specific running directions of the aligned $\alpha$-grains as shown in Fig. 3, were not found, clearly suggesting that the alignment of the $\alpha$-grains originated from surface defects caused by slip and twinning of the Si substrate during post-deposition annealing.

Based on the suggestion that surface defects caused by slip and twinning of the Si substrate during post-deposition annealing can act as stress raisers, we speculate that the formation of the $\alpha$-regions starts at the deposit/Si interface. Of course, more rigorous study, e.g., in situ TEM observation at high temperatures, is required to explore the formation and transformation mechanisms.

If $\alpha$-Al$_2$O$_3$ phase is elastically anisotropic, its orientation can also influence the strain energy [30]. However, the elastic properties of $\alpha$-Al$_2$O$_3$ phase are nearly isotropic (Appendix A), and its orientation effect is excluded. This is manifested by the random orientation of $\alpha$-Al$_2$O$_3$ crystals, which developed during annealing for 1 h (Fig. 4).

When the annealing time increases to 24 h and 48 h, the density of the $\alpha$-regions increases (Figs. 5 and 6), and the density of the [0001] // ND component increase to minimize the system energy.

5. Concluding remarks

All the thin film specimens annealed at 1100 °C exhibited $\alpha$-Al$_2$O$_3$ grains of 2–10 μm in diameter and their alignments in various directions. The directions are in agreement with the rows of surface defects caused by slip and twinning of the Si substrate during annealing, due to the thermal stress developed in the substrate. The texture of $\alpha$-Al$_2$O$_3$ grains could be approximated by random orientation when they were annealed for 1 h. As the annealing time increased, the texture approached the basal plane orientation, resulting from its lowest surface energy.

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Appendix A. Elastic anisotropy of $\alpha$-Al$_2$O$_3$

Suppose that the uniaxial stress is oriented along $x_i$, then Young’s modulus $E$ is given by

$$ E = 1 / S_{1111} $$

(A1)

where the compliances $S_{1111}$ can be expressed in terms of the compliances $S_{ijkl}$ referred to the symmetry axes of the material. From the transformation law

$$ S'_{1111} = a_{i1}a_{j1}a_{k1}a_{l1}S_{ijkl} = 1 / E. $$

(A2)

The compliances of crystals with hexagonal symmetry have a form of Eq. (A3) when referred to the crystal axes as shown in Fig. A1.

$$
\begin{bmatrix}
S_{11} & S_{12} & S_{13} & 0 & 0 & 0 \\
S_{12} & S_{11} & S_{13} & 0 & 0 & 0 \\
S_{13} & S_{13} & S_{11} & 0 & 0 & 0 \\
0 & 0 & 0 & S_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & S_{44} & 0 \\
0 & 0 & 0 & 0 & 0 & 2(S_{13} - S_{12})
\end{bmatrix}
$$

(A3)

Writing Eq. (A2) in full,

$$
S'_{11} = S_{11} = a_{11}a_{11}a_{11}a_{11}S_{11} + a_{11}a_{11}a_{11}a_{11}S_{12} + a_{11}a_{11}a_{11}a_{11}S_{13} + a_{11}a_{11}a_{11}a_{11}S_{144} + a_{11}a_{11}a_{11}a_{11}S_{33} + a_{11}a_{11}a_{11}a_{11}S_{44} + a_{11}a_{11}a_{11}a_{11}S_{55} + a_{11}a_{11}a_{11}a_{11}S_{66}
$$

(A4)

where $a_{ij}$ are the direction cosines (cosines of angles between the uniaxial stress direction $x_i$ and the symmetry axes shown in Fig. A1).

It follows from relations of $S'_{11} = S_{11} = S_{12} = S_{13} = S_{23} = S_{33} = S_{44} = S_{55} = S_{66}$ that Eq. (A4) reduces to

$$
S'_{11} = S_{11}(1 - a_{13}^2) + S_{13}a_{13}^2 + (S_{44} + 2S_{13})a_{13}(1 - a_{13}^2).
$$

(A5)

We want to calculate the maximum and minimum values of $S_{11}$. We have:

$$
\frac{dS'_{11}}{da_{13}} = -4a_{13}S_{44}a_{13} + 4a_{13}S_{44} + 2(S_{44} + 2S_{13})a_{13}(1 - 2a_{13}^2)
\]

$$
= 2a_{13}^2 - 2a_{13}S_{44} - 2S_{13}a_{13}(1 - 2a_{13}^2) = 0
$$

For $\alpha$-Al$_2$O$_3$ (hexagonal), $S_{11} = 0.002323$, $S_{33} = 0.002171$, $S_{44} = 0.006803$, $S_{12} = -0.0006709$, $S_{13} = -0.0003849$ (in GPa$^{-1}$) [32].

$$
\frac{dS'_{11}}{da_{13}} = -4S_{11}(1 - a_{13}^2) + 12a_{13}S_{44} + 2(S_{44} + 2S_{13})(1 - 6a_{13}^2)
$$

$$
= -4S_{11} + 2(S_{44} + 2S_{13}) = 0.002774 > 0.002774 > 0 at a_{13} = 0
$$

$$
\frac{d^2S'_{11}}{da_{13}^2} = -4S_{11} - 2S_{44} < 0 at a_{13} = \pm 0.4506.
$$
Therefore, \( S_{11}' = S_{11} \) at \( a_{13} = 0 \), which is the minimum value of \( S_{11}' \).

That is,

\[
[S_{11}']_{\text{min}} = S_{11} = 0.002323 \text{ GPa}^{-1} \text{ or } E_{\text{max}} = 430.5 \text{ GPa.} \tag{A6}
\]

\( S_{11} \) is the maximum at \( a_{13} = \pm 0.4506 \). That is,

\[
[S_{11}']_{\text{max}} = 0.002541 \text{ GPa}^{-1} \text{ at } a_{13} = \pm 0.4523 \text{ or } E_{\text{min}} = 393.5 \text{ GPa.} \tag{A7}
\]

The difference between \( [S_{11}']_{\text{max}} \) and \( [S_{11}']_{\text{min}} \) or between \( E_{\text{max}} \) and \( E_{\text{min}} \) is small, specifically \( [S_{11}']_{\text{max}}/[S_{11}']_{\text{min}} = E_{\text{max}}/E_{\text{min}} = 1.09 \), which indicates that \( \alpha\)-Al\(_2\)O\(_3\) is almost elastically isotropic.

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