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ABSTRACT

Epitaxial film quality is critical to the success of high-performance α -Ga₂O₃ vertical power devices. In this work, the origins of threading dislocation generation and annihilation in thick α -Ga₂O₃ films heteroepitaxially grown on sapphire by the mist-CVD technique have been examined by means of high-resolution X-ray diffraction and transmission electron microscopies. By increasing the nominal thickness, screw dislocations exhibit an independent characteristic with a low density of about $1.8 \times 10^6 \text{ cm}^{-2}$, while edge dislocations propagating along the c-axis are dominant, which decrease down to $2.1 \times 10^9 \text{ cm}^{-2}$ in density for an 8 μm -thick α -Ga₂O₃ layer and exhibit an inverse dependence on the thickness. In the framework of the glide analytical model, parallel edge dislocations are generated at the interface due to the misfit-induced strain relaxation, while the dislocation glide and coalescence result in the annihilation and fusion behaviors. The optimal thick α -Ga₂O₃ with low dislocation densities may provide a prospective alternative to fully realize α -Ga₂O₃ power devices.

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Gallium oxide (Ga₂O₃) is an emerging material of rapidly increasing importance for power device applications.^{1–3} Field effect transistors (FETs) and Schottky rectifiers have been demonstrated mainly on monoclinic β -Ga₂O₃ material due to its most thermodynamically stable phase and the availability of large scale wafers for homoepitaxy. In comparison, metastable corundumlike α -phase Ga₂O₃ has a relatively larger bandgap, smaller electron effective mass, higher breakdown field, and larger Baliga's figure of merit.^{4–6} It also exhibits a relatively small lattice mismatch with many functional oxides such as sapphire, Cr₂O₃, and Fe₂O₃, hence allowing the design of advanced semiconductor heterostructures.^{7–9} Thus, it is expected that α -Ga₂O₃ is a promising alternative for power devices with higher output power and lower power consumption. In recent years, high quality α -Ga₂O₃ epilayers and heterostructures have been realized on sapphire substrates by mist-CVD⁶ and halide vapor phase epitaxy (HVPE).^{10–12} Consequently, vertical Schottky diodes with a low

on-resistance of $0.4 \text{ m}\Omega \text{ cm}^2$ and a breakdown voltage of 855 V have been reported.¹³ Due to the large in-plane lattice mismatch of about 4.81%, α -Ga₂O₃ epilayers deposited on sapphire substrates typically result in a high density of dislocations of the order of 10^{10} cm^{-2} .¹⁴ High leakage current still occurs in these devices, and the corresponding breakdown field is still far from the theoretical limit of α -Ga₂O₃. The observed threading dislocations (TDs) present in heteroepitaxial layers usually act as carrier killer defects and scattering centers, which act as conduction channel leakage paths, severely deteriorating carrier mobility.¹⁵ Recently, Oshima *et al.* have performed epitaxial lateral overgrowth (ELO) of α -Ga₂O₃ with an edge dislocation density down to $5 \times 10^6 \text{ cm}^{-2}$ and found that both tilt and twist structures in the layer decreased with increasing thickness.¹⁶ However, the reaction kinetics of dislocation generation and annihilation have not yet been clarified, which is the fundamental ground for suppressing dislocations toward the improvement of vertical α -Ga₂O₃ power devices. Here, we evaluate the

dependence of tilt and twist structures in α -Ga₂O₃ heteroepitaxial layers on epitaxial thicknesses. The related threading dislocation reaction kinetics are revealed to understand the crystal defects associated with this mismatched heteroepitaxial system in the framework of a dislocation glide analytical model.

In this work, all α -Ga₂O₃ films were grown on c-plane sapphire substrates using a hot-wall atmosphere mist-CVD system. The substrates, cleaned by standard solvents, were directly loaded into the reactor without any surface treatment. The precursor, which is gallium acetylacetonate [(C₅H₇O₂)₃Ga] solved in de-ionized (DI) water (0.05 mol/l) with 1.5% HCl, was atomized into micrometer-sized particles by a 2.4 MHz ultrasonic transducer and then carried into the growth chamber by the mixture of N₂ and O₂ as the carrier gas. The growth temperature was optimized at 470 °C, and the growth rate is about 1 μ m/h. The detailed growth conditions have been described elsewhere.¹⁷ By increasing the growth time, a series of α -Ga₂O₃ films were achieved with their nominal thickness ranging from 80 nm to 8 μ m as determined from optical reflection spectra recorded using a UV-visible spectrometer (Lambda 950, PerkinElmer). It was reported that cracks were often formed on the surface of thick α -Ga₂O₃ films (>3 μ m) without any buffer layer, which result from the accumulated thermal stress induced by the thermal expansion mismatch between α -Ga₂O₃ and sapphire.⁴ In this work, we tried to slow the cooling rate (<50 K/h) after the growth so that the accumulated thermal strain can be relaxed gradually and crack-free thick α -Ga₂O₃ films (up to 6.0 μ m) without any buffer layer have been achieved.¹⁷ The microstructures were characterized by high resolution X-ray diffraction (HRXRD) using a D8 system diffractometer with a high resolution of 0.0001°. The surface morphology was characterized using atomic force microscopy (AFM) operating in the tapping mode. The cross-sectional TEM images were taken using a Talos F200X system working at 200 kV. Cross-sectional TEM specimens were prepared by a conventional combination of mechanical grinding and ion thinning.

Figure 1(a) shows the X-ray diffraction (XRD) $2\theta/\omega$ scan spectrum of a 2.5 μ m-thick α -Ga₂O₃ sample. Only two diffraction peaks corresponding to the (0006) planes of α -Ga₂O₃ and Al₂O₃ are observed. This indicates that the resultant epilayer is single crystalline with the corundum-structured α phase. Perfect lattice ordering in the dislocation-free region is observable in the high-resolution TEM (HRTEM) image shown in the inset of Fig. 1(a). From the ϕ -scan results shown in Fig. 1(b), we determined that the in-plane epitaxial relationship between the epilayer and the substrate is α -Ga₂O₃ (10–10)//Al₂O₃ (10–10). Aside from the distinct peaks of three-fold rotational symmetry, another set of weak peaks is seen at positions rotated by 60° with respect to the predominant peaks, which is possibly a result of double positioning domains formed in the initial nucleation and lateral coalescence stage.⁶ It is noted that all samples investigated here are pure phase α -Ga₂O₃ as determined by XRD measurements. The atomic force microscopy images in Figs. 1(c) and 1(d) exhibit the surface morphologies of α -Ga₂O₃ epilayers with thicknesses of 250 nm and 2.5 μ m, respectively. The randomly distributed array of spiral hillocks is dominant on the surface of the 250 nm-thick layer, which are formed from the spiral ramp created by atomic terraces associated with pinned steps. Each of the pinned steps exhibits a height of about 0.22 nm, corresponding to $c/6$ of α -Ga₂O₃.⁸ It was reported that spiral growth hillocks arise from the lateral growth of pinned steps

FIG. 1. (a) High resolution X-ray diffraction (HRXRD) $2\theta/\omega$ scan spectrum of a 2.5 μ m-thick α -Ga₂O₃ epilayer. The inset displays the HRTEM image; (b) XRD ϕ -scan measurement for the (10–14) plane of the α -Ga₂O₃ epilayer and α -Al₂O₃ substrate; (c) and (d) AFM images of 250 nm and 2.5 μ m-thick α -Ga₂O₃ epilayer, respectively.

when a threading dislocation with a screw component intersects the growing front surface, in particular, in the initial nucleation stage.¹⁸ The dislocation causes a surface displacement equal to the component of the Burgers vector normal to the surface. The inset of Fig. 1(d) shows that, by increasing the epitaxial thickness, lateral growth facilitates the spiral hillocks to be developed into micrometer-size single domains, as shown in the inset of Fig. 1(d). Within each individual domain, the atomic steps are straight and elongated along the [11–20] and no surface depressions within the terraces are observed. Existing theories developed for dislocation mediated surface morphologies have been employed to interpret the formation of pinned steps, spiral growth hillocks, and surface depression.¹⁸ The density of spiral hillocks is consistent with the density of mixed dislocations, while the absence of surface depressions at terraces is only correlated with pure edge dislocations because pure edge dislocations do not have a component of the Burgers vector perpendicular to the surface.

The specific threading dislocation geometry in a film will lead to a distortion of only specific crystallographic planes.¹⁹ Hence, to examine the contributions of tilt and twist structures, X-ray rocking curves (XRCs) for different planes under both symmetric and skew symmetric geometries were performed on 2.5 μ m-thick α -Ga₂O₃, as shown in Fig. 2(a). The full-width at half maximum (FWHM) of the rocking curve for (0006) planes ($\Psi = 0^\circ$) is only about 54 arc sec, indicative of a very small tilt angle of the (0001) plane. For the ($hk.l$) reflections with inclination angles approaching $\Psi = 90^\circ$, the FWHM increases gradually up to 1260 arc sec. The dramatic broadening of the asymmetric scans is an indicative of a large density of pure edge dislocations with a Burgers vector $\mathbf{b}_e = \frac{1}{3}\langle 2\bar{1}\bar{1}0 \rangle$.¹⁴ Similar to the heteroepitaxy of GaN and ZnO on sapphire substrates, the mosaic model has been proven to be an effective approach to quantitatively analyze both tilt and twist rotations.²⁰ Figure 2(b) summarizes the broadening feature $W(\Psi)$ as a function of inclination angle (Ψ), which is defined as the angle between the scattering vector and the sample surface normal as illustrated in the inset of Fig. 2(b). Assuming that the tilt and twist

FIG. 2. (a) X-ray rocking curves (XRCs) of different planes under symmetric and skew symmetric geometries; (b) FWHM (W) as a function of inclination angle. The line is a least-squares fit using a model developed by Srikant *et al.* The inset of the schematic is reprinted with permission from Srikant *et al.*, J. Appl. Phys. **82**(9), 4286–4295 (1997). Copyright 1997 AIP Publishing.²⁰

distributions are independent, the angular component of two distributions can be convoluted to determine the resultant misorientation distribution. Taking each microstructure as a rigid body, $W(\Psi)$ can be determined by using rotation matrices²⁰

$$W(\Psi) = \left\{ [W^{\text{tilt}}(\Psi)]^n + [W^{\text{twist}}(\Psi)]^n \right\}^{1/n}, \quad (1)$$

$$W^{\text{tilt}}(\Psi) = \cos^{-1} [\cos^2(\Psi) \cos(W_{\text{out}}) + \sin^2(\Psi)],$$

$$W^{\text{twist}}(\Psi) = \cos^{-1} [\sin^2(\Psi) \cos(W_{\text{in}}) + \cos^2(\Psi)],$$

where $W^{\text{tilt}}(\Psi)$ and $W^{\text{twist}}(\Psi)$ are the broadening features resulting from mosaic tilt and twist at a given Ψ , respectively, while W_{out} and W_{in} are the out-of-plane tilt and in-plane twist angles, respectively. The constant $n = 1 + (1 - m)^2$ depends on the fraction (m) of the Lorentzian character in the XRD rocking curves in terms of the pseudoVoigt (PV) function, $PV(x) = (1 - m)G(x) + mL(x)$, where $G(x)$ and $L(x)$ represent the Gaussian and Lorentz functions, respectively. The fittings to the rocking curves in Fig. 2(a) give rise to a small deviation and an average value (m) of 0.78. Based on Eq. (1), a good fitting of $W(\Psi)$ as a function of Ψ is obtained in Fig. 2(b), giving rise to the in-plane twist angle (W_{in}) of 0.39° for the $2.5 \mu\text{m}$ -thick $\alpha\text{-Ga}_2\text{O}_3$. Assuming that dislocations are randomly distributed, the dislocation densities can be estimated from the modified relationships developed by Kaganer *et al.* as²¹

$$D_e = \frac{W_{\text{in}}^2}{(2\pi \ln 2)b_e^2}, \quad D_s = \frac{W_{\text{out}}^2}{(2\pi \ln 2)b_s^2}, \quad (2)$$

where D_e (D_s) and b_e (b_s) are dislocation densities and lengths of Burgers Vectors for edge (screw) types, respectively. From the Burgers vectors $\mathbf{b}_e = \frac{1}{3}\langle 2\bar{1}10 \rangle$ and $\mathbf{b}_s = \langle 0001 \rangle$, an estimation of the screw and edge dislocation densities in this $2.5 \mu\text{m}$ -thick sample is 1.6×10^6 and $5.0 \times 10^9 \text{ cm}^{-2}$, respectively, which is comparable to other results reported in the $\alpha\text{-Ga}_2\text{O}_3$ epilayers grown by mist-CVD and HVPE techniques.^{14,16}

Figures 3(a) and 3(b) show the XRC of (0006) and (10–14) planes for a series of samples with different thicknesses, respectively. The XRC width for the (0006) plane is almost independent of the thickness with an averaged value of 49 arc sec, while the width for the (10–14) plane decreases remarkably with the increasing thickness. Following the same approach described above, the screw and edge dislocations in

FIG. 3. (a) and (b) XRCs of (0006) and (10–14) planes for $\alpha\text{-Ga}_2\text{O}_3$ epilayers with different thicknesses, respectively, and (c) calculated edge and screw dislocation densities as functions of film thickness. The dashed line is a least-squares fit to the relationship of $D_e = C/h$.

different thick layers have been calculated and are summarized in Fig. 3(c). When the film thickness increases from 80 nm to $8 \mu\text{m}$, the screw dislocation density remains constant at around $1.8 \times 10^6 \text{ cm}^{-2}$, while the edge dislocation density decreases gradually from 1.0×10^{11} to $2.7 \times 10^9 \text{ cm}^{-2}$. The edge dislocation density exhibits an inversely proportional dependence to the film thickness, as described by $D_e = C/h$, where the constant C is the line dislocation density and is extracted to be $1.57 \times 10^6 \text{ cm}^{-1}$ from the excellent fitting in Fig. 3(c). Cross-sectional TEM analysis was carried out to directly quantify the nature and density of threading dislocations in a $4.5 \mu\text{m}$ -thick $\alpha\text{-Ga}_2\text{O}_3$ epilayer. Figures 4(a)–4(c) exhibit the two-beam bright field TEM images with different diffraction vectors, respectively. The $\mathbf{g} \cdot \mathbf{b}$ criterion in TEM is generally applied for the two-beam condition to evaluate the alignment of the strain field of the dislocation with

FIG. 4. (a)–(c) Two beam bright field TEM images with diffraction vectors of $\mathbf{g} = (0006)$, (-2110) , and $1/3(-2116)$, respectively. (d) Magnified image of (c) marked by the red rectangle and (e) the cross-sectional HRTEM image focusing on the interface of the $\alpha\text{-Ga}_2\text{O}_3$ epilayer and sapphire substrate.

Burgers vector $\mathbf{b}$ with respect to the diffraction vector $\mathbf{g}$ to produce an image contrast. Pure edge dislocations and pure screw dislocations should not be observable under $\mathbf{g} = [0002]$ and $\mathbf{g} = [-2110]$ imaging conditions, respectively, while edge, screw, and mixed dislocations are present simultaneously in a combined case with $\mathbf{g} = 1/3[-2116]$. Figure 4(a) shows that no recognizable pure screw dislocations are observed and the contrast may be a result of mixed screw/edge thread dislocations, while under $\mathbf{g} = [-2110]$, pure edge thread dislocations can be distinguished. By comparing three two-beam images, the contrast induced by dislocations is almost the same in Figs. 4(b) and 4(c), suggesting that edge dislocations with the Burgers vector of $\mathbf{b}_e = \frac{1}{3}\langle 2\bar{1}\bar{1}0 \rangle$ have a high density at the film-substrate interface and extend along the film normal.

Quantitative analysis of spatial distribution of edge dislocations has been observed from Fig. 4(b). The solid triangles in Fig. 3(c) are the edge dislocation densities statistically estimated by TEM as a function of depth, which is quite consistent with the XRD results. A similar dependence of the edge and screw dislocations on the film thickness has been observed in different lattice-mismatched epitaxial systems. The cross-sectional HRTEM image in Fig. 4(e) shows obvious periodic distortion at the interface with a clear contrast along the $[1-100]$ direction, and the average line density of such distorted structures is estimated to be $1.12 \times 10^6 \text{ cm}^{-1}$. These are misfit dislocations, which are formed due to the relaxation of strain induced by the lattice mismatch, similar to the observation by Kaneko *et al.*¹⁴ Given the in-plane lattice misfit of $f = 4.81\%$, the spacing of the edge component of the misfit segment ($R_{\text{ave}} = b_e/f$) and the line density of misfit dislocations at the interface are calculated to be 10.3 nm and $1.16 \times 10^6 \text{ cm}^{-1}$, respectively. The line density of misfit dislocations is quite consistent with that of edge dislocations ($1.57 \times 10^6 \text{ cm}^{-1}$) derived from Fig. 3(c), which indicates that misfit dislocations are the origin of edge dislocations with a glide plane coinciding with the interface. As observed in Fig. 4(e), the misfit dislocations can easily glide along the interface to form an almost periodic array with an average spacing of 8.9 nm, thus minimizing the elastic energy induced by the lattice mismatch.

Typically, for large mismatch films (e.g., mismatch strains in excess of $\sim 2\%$), the edge dislocation densities are quite high near the film substrate interface, often on the order of 10^{10} – 10^{11} cm^{-2} . In the initial growth stage, the lattice misfit easily leads to three-dimensional nuclei that are misoriented with respect to each other by a small twist angle. Once two such nuclei grow into large lateral domains, the small misorientation gives rise to a low-angle domain boundary with edge dislocations approximately parallel to the domain planes. If the lattice misfit is less than 10%, the observed inverse relationship between the film thickness and dislocation density can be well described by the glide model.²² Similar to the case of GaN epitaxy on sapphire, if only lateral coalescence reactions are considered, the overall dislocation density is determined by second-order kinetics with a relationship of $dD(h) = -KD^2 dh$, where K is a kinetic reaction coefficient related to the dislocation annihilation and fusion reactions.⁴ This kinetic approach considers the reactions between threading dislocations in relation to their densities and relative motions that correspond to the densities and kinetic energy of the molecules in chemical reactions. There is a thermodynamic driving force to diminish the dislocation density, and high densities of dislocations facilitate the development of a kinetic approach for dislocation reduction. The screw dislocation densities (D_s) in $\alpha\text{-Ga}_2\text{O}_3$ epilayers are quite low ($\sim 10^6 \text{ cm}^{-2}$),

suggesting that these isolated screw dislocations have low reaction rates and low possibilities for annihilation or coalescence.⁴ As a result, the small value of D_s leads to small $dD_s(h)/dh$, which explains that the density of the screw dislocation is almost independent of the epilayer thickness.

For edge dislocations with Burgers vectors parallel to the interface, the solution of the above equation predicts an inverse linear relationship between D_e and thickness h , as given by²²

$$D_e(h) = [f/16b_e(1-v)\ln(1/4f)]/h, \quad (3)$$

where the Poisson ratio ν is about 0.3, the misfit f is about 4.81%, and the length of $\mathbf{b}_e = \frac{1}{3}\langle 2\bar{1}\bar{1}0 \rangle$ is 0.497 nm. As a result, the coefficient of $f/16b_e(1-v)\ln(1/4f)$ in Eq. (3) turns out to be $1.42 \times 10^6 \text{ cm}^{-1}$, very close to the estimated line density of misfit dislocations. This strongly suggests that the glide model is valid to explain the dislocation evolution in the $\alpha\text{-Ga}_2\text{O}_3$ epilayer on sapphire substrates. Dislocation mediated surface morphology and two-beam cross-sectional TEM images give clear evidence that edge dislocations are a result of the coalescence of spiral hillocks with a misorientation along the c -axis. If the space between neighboring dislocations is smaller than a minimum value, in particular, near the interface, the combination of attractive glide force and line tension associated with the misfit dislocations will drive the glide of the threading dislocation toward one another and coalesce at a particular glide plane. However, as the film grows thicker, the misfit-induced strain relaxes quickly, and most of the parallel edge dislocations along the c -axis become stable with increasing spacing separation. The low reaction probabilities lead to a slow reduction rate of edge dislocations. In addition, the bending and fusion of partial edge dislocations could also be formed as a result of the reaction with screw dislocations, as shown in Fig. 4(d). However, $\alpha\text{-Ga}_2\text{O}_3$ grown on sapphire undergoes a semicoherent growth and arrays of parallel edge dislocations form low angle tilt boundaries, which prevents the generation of screw dislocations and suppresses the reaction of mixed dislocations.

In summary, the dependence of threading dislocations on the thickness of $\alpha\text{-Ga}_2\text{O}_3$ heteroepitaxial layers on sapphire grown by the Mist-CVD technique has been revealed by means of HRXRD and TEM. Despite the low screw dislocation density of around 10^6 cm^{-2} , independent of the epilayer thickness, the edge dislocations generated by the lattice misfit propagate along the c -axis and the low reaction probabilities result in a slow reduction in density. The inverse relationship between edge dislocation density and the film thickness is a result of dislocation glides and coalescence, which can be explained by the dislocation glide model. To reduce the threading dislocation density, the bending of dislocations toward the lateral direction on inclined facets is essential to minimize the misfit-induced elastic strain energy within a thin layer.

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