Rapid epitaxy of 2-inch and high-quality α -Ga₂O₃ films by mist-CVD method

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Abstract: High thickness uniformity and large-scale films of α -Ga₂O₃ are crucial factors for the development of power devices. In this work, a high-quality 2-inch α -Ga₂O₃ epitaxial film on *c*-plane sapphire substrates was prepared by the mist-CVD method. The growth rate and phase control mechanisms were systematically investigated. The growth rate of the α -Ga₂O₃ films was limited by the evaporation of the microdroplets containing gallium acetylacetonate. By adjusting the substrate position (*z*) from 80 to 50 mm, the growth rate was increased from 307 nm/h to 1.45 μ m/h when the growth temperature was fixed at 520 °C. When the growth temperature exceeded 560 °C, ε -Ga₂O₃ was observed to form at the edges of 2-inch sapphire substrate. Phase control was achieved by adjusting the growth temperature. When the growth temperature was 540 °C and the substrate position was 50 mm, the full-width at half maximum (FWHM) of the rocking curves for the (0006) and (10-14) planes were 0.023° and 1.17°. The screw and edge dislocations were 2.3 × 10⁶ and 3.9 × 10¹⁰ cm⁻², respectively. Furthermore, the bandgaps and optical transmittance of α -Ga₂O₃ films grown under different conditions were characterized utilizing UV-visible and near-IR scanning spectra.

Key words: ultra-wide bandgap semiconductor; mist-chemical vapor deposition; epitaxy; alpha-gallium oxide

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1. Introduction

Gallium oxide, as an ultra-wide bandgap semiconductor, has become an excellent candidate for power electronic devices and solar-blind photodetectors by virtue of its large bandgap, high breakdown electric field and Baliga's figure of merit. Gallium oxide possesses six polymorphs, including α , β , γ , δ , ε , $\delta^{[1]}$. Among them, the thermodynamically stable phase, monoclinic β -Ga₂O₃, has been studied intensively in the last two decades. Compared with β -Ga₂O₃, metastable α -Ga₂O₃ has recently garnered extensive attention owing to its excellent properties. Meanwhile, α -Ga₂O₃ exhibits larger bandgap (~5.3 eV) and bigger Baliga's figure of merit (~6726 $\epsilon\mu$ Ec³) than that of β -Ga₂O₃. The α -Ga₂O₃ has higher symmetry which belongs to corundum structure, the space group is *R*-3m^[2], with unit cell parameters of a = 4.98 Å and c = 13.43 Å^[3]. The lattice mismatch of α -Ga₂O₃ and sapphire was only 4.8% (along the a-axis)^[3]. This makes it possible to obtain α -Ga₂O₃ films by heteroepitaxy on low-cost sapphire substrates. Additionally, the alteration of the bandgap of α -Ga₂O₃ could be achieved between 3.7 eV and 9.0 eV when alloyed with materials such as a-Al2O3, a-In2O3, a-Fe2O3, a- Cr_2O_3 , α -Rh₂O₃ and α -V₂O₃^[4, 5]. Furthermore, the problem of low solid solubility, as compared with β -Ga₂O₃, will not be encountered by α -Ga₂O₃^[6].

The α -Ga₂O₃ films can be obtained using various tech-

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niques, including metal-organic chemical vapor deposition (MOCVD)^[7, 8], halide vapor phase epitaxy (HVPE)^[9-11], and mist-chemical vapor deposition (mist-CVD)^[12-15] and so on. Among these techniques, the HVPE method has been used in the rapid growth of α -Ga₂O₃ films, and the growth rate could be reached to 10 μ m/h^[11]. Unfortunately, the quality of α -Ga₂O₃ films cannot be guaranteed to be high and the morphology was poor under high growth rates. The MOCVD method offers high-quality materials with a growth rate of 1 μ m/h^[7, 8]. However, the process cost is very high, which makes it unsuitable for mass production. Finally, mist-CVD is a non-vacuumbased, low-energy and economical approach for the growth of α -Ga₂O₃ films using safe and inexpensive source precursors. In addition, oxygen vacancies could be reduced by mist-CVD, which is one of the major issues with oxides preparation^[16]. Since 2008, corundum-structured α -Ga₂O₃ thin films have been successfully prepared applying mist-CVD^[12]. Moreover, the thick α -Ga₂O₃ films were obtained with a hot-wall type mist-CVD, and the growth rate was about 1 μ m/h according to recent research^[15]. However, the dominant factors affecting the growth rate are still unclear, and the low growth rate of a-Ga₂O₃ films grown by mist-CVD has limited the application of α -Ga₂O₃ films and devices. Additionally, the crystal quality and uniformity of large-scale α -Ga₂O₃ films were hardly guaranteed with high growth rate^[6, 12, 16-20]. Furthermore, other crystalline phases likely appeared in the epitaxy of α -Ga₂O₃ films^[13]. The dominant factors affecting the growth rate are still unclear.

In this work, the large-scale α -Ga₂O₃ films with high qual-

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ity uniformity were rapidly prepared by mist-CVD. The growth mechanism of rapid preparation of α -Ga₂O₃ films by mist-CVD was investigated. The growth rates of the α -Ga₂O₃ films were strongly dependent on the position of the substrate in the furnace. The evaporation of the microdroplets was decreased as the position of substrate decreased. The growth rate was calculated to be 1.45 μ m/h at z = 50 mm, which was much faster than previous reports^[6, 12, 16–20]. By adjusting the growth temperature and the position of substrates, 2-inch α -Ga₂O₃ films with pure phases were obtained. Accordingly, the full-width at half maximum (FWHM) of rocking curves for the (0006) and (10–14) planes are as low as 0.023° and 1.17°, respectively. The rapid preparation of large-scale and high-quality α -Ga₂O₃ films has been obtained.

2. Experiment

The 2-inch α -Ga₂O₃ films were grown on c-plane sapphire substrates using a vertical hot-wall mist-CVD system. Gallium acetylacetonate (Ga(acac)₃) was dissolved in deionized (DI) water, and 1 vol% hydrochloric acid (HCI) was used as the cosolvent. The solution (0.05 mol/L) was atomized into micron-sized droplets using a 1.7 MHz ultrasonic transducer. High-purity argon (99.999%) and high-purity oxygen (99.999%) were used as the carrier gas and diluting gas, respectively, and the flow rates were maintained at 1500 and 150 sccm. The gallium source was delivered from the bottom of the reactor into the mist-CVD system. Pure α -Ga₂O₃ films were obtained by controlling temperature between 500 and 580 °C. In the vertical hot-wall mist-CVD system, the position of the substrate can be adjusted by moving the susceptor. The vertical position (z) was defined by the reference origin z = 0 mm at the heater entrance and the regulated range of the substrate position (z) was 0-150 mm.

The quality of the a-Ga₂O₃ films was characterized using high-resolution X-ray diffraction (HRXRD, Bruker-AXS D5005HR) and X-ray diffraction (XRD, D8ADVANCE). The surface morphology and thickness were characterized using scanning electron microscopy (SEM, Nova NanoSEM 450). The optical transmission spectra were characterized using a UV-visible near-IR scanning spectrophotometer (Cary 5000).

3. Results and discussion

Fig. 1 shows the vertical hot-wall mist-CVD system. The vertical position (z) was defined by the reference origin z =0 mm at the heater entrance. In the vertical hot-wall mist-CVD system, the guality and morphology of the epitaxial films were obviously depended on the substrate position (z). In our experiments, the surface of the sapphire substrate was covered by solid particles at T = 520 °C and z = 150 mm, indicating that the precursors had been fully transformed into solid particles. As the position of the substrate decreased, the solid particles were gradually reduced and the surface of the sapphire substrate became smooth. Ga₂O₃ films with smooth surfaces and great transparencies were successfully prepared at z = 80 mm. It was believed that as the microdroplets were traveling in furnace, the sizes of microdroplets became smaller or were even transformed into solid particles due to evaporation^[16]. Therefore, the position of the substrate was important for the surface topography and crystallization quality of the film.



Fig. 1. (Color online) Schematic of a vertical hot-wall mist-CVD.

Figs. 2(a)–2(d) show the cross-sectional SEM images of the a-Ga₂O₃ films on sapphire. At the same epitaxy temperature of 520 °C, the thickness of the films increased as the position of the substrate decreased, as shown in Fig. 3. The thickness of the Ga₂O₃ film was 307 nm at T = 520 °C and z = 80 mm, as shown in Fig. 2(a). When the position of the substrate was decreased to 50 mm, the thickness of Ga₂O₃ films increased to 2.9 μ m accordingly at the same temperature and carrier gas rate, as shown in Fig. 2(d).

Fig. 3 shows the growth rates of the films deposited at varying substrate positions (z = 50-80 mm). The growth rate of films was increased with the position of substrate decreased and the growth rate could reach 1.45 μ m/h at T = 520 °C and z = 50 mm, which was the fastest in the α -Ga₂O₃ epitaxy by mist-CVD method^[6, 12, 16-20].

The growth mechanism of the mist-CVD system was discussed based on the principles of the growth rate. The microdroplets must be glided over the substrate surface by Leidenfrost motion during epitaxy, which was the key factor in the preparation of the α -Ga₂O₃ films^[21]. As the microdroplets were transported in the furnace, the size of microdroplets could be decreased due to evaporation^[14]. With the evaporation of microdroplets, sufficiently small microdroplets would be interacted with the substrate by Leidenfrost motion and this released precursors for epitaxy. When effectively used, small microdroplets were employed to successfully prepare α -Ga₂O₃ films with smooth surfaces. As the small microdroplets were further evaporated, some of them were transformed into precipitates or even solid particles. The growth rate of the α -Ga₂O₃ films decreased because of the depletion of the microdroplets. Hence, the microdroplets should not be allowed to fully evaporate before approaching the substrate surface. The proper position of the substrate was a key factor in the successful preparation of α -Ga₂O₃ films.

The suitability of growth temperature for obtaining a-Ga₂O₃ films was studied when the position of the substrates (z) was fixed at 50 mm. Fig. 4 shows the X-ray diffraction (XRD) spectra of the α -Ga₂O₃ films grown at temperatures ranging from 500 to 580 °C. The diffraction peaks at 38.94°, 40.34° and 41.76° correspond to the (004) plane of ε -Ga₂O₃, (0006)



Fig. 2. Cross-sectional SEM images of the Ga_2O_3 films on a sapphire epitaxy at 520 °C with different substrate positions.



Fig. 3. Growth rates of the Ga₂O₃ films at different substrate positions.



Fig. 4. (Color online) XRD patterns of Ga₂O₃ film grown at 500-580 °C.



Fig. 5. (Color online) The crystal phases distributions of Ga_2O_3 films at (a) T = 560 °C and z = 50 mm, (b) T = 540 °C and z = 50 mm.

plane of the sapphire substrate and (0006) plane of α -Ga₂O₃, respectively. Apart from the peak of the sapphire substrate at 41.76°, only one peak of the (0006) plane of α -Ga₂O₃ at 40.34° could be observed at the growth temperatures of 500-540 °C, indicating the preparation of pure α -Ga₂O₃ films on sapphire substrates. The improvement of the crystal quality of α -Ga₂O₃ was demonstrated by the increase of the peak at 40.34° as the temperature increased. The diffraction peak of the ε -Ga₂O₃ (004) plane at 38.94° was observed when the growth temperature exceeded 560 °C, which demonstrates the appearance of the second phase of ε -Ga₂O₃. According to First-principle calculations, the expansion and formation energy relationships in the five forms of Ga₂O₃ are as follows: $\beta < \epsilon < \alpha < \delta < \gamma^{[22]}$. The expansion and formation energy of α -Ga₂O₃ are higher than those of β -Ga₂O₃, considering that α -Ga₂O₃ was generally more unstable than β -Ga₂O₃. However, α -Ga₂O₃ is more stable than β -Ga₂O₃ at a high hydrostatic pressure of greater than 40 GPa^[23]. The sapphire substrate and α -Ga₂O₃ are classified into the corundum crystal structure, and the lattice mismatch is 4.81%^[3]. Despite the fact that α -Ga₂O₃ has a higher formation energy than ϵ -Ga₂O₃ and β -Ga₂O₃, α -Ga₂O₃ could be stabilized under the misfit strain created by lattice mismatch. The mismatch strain at the interface is quickly released as the growth temperature rises, leading to the acceleration of ϵ -Ga₂O₃ growth^[13].

The distribution of crystal phases in Ga₂O₃ films at different temperatures was also investigated, as shown in Figs. 5(a)– 5(b). The α -Ga₂O₃ phase is represented by the green areas, while the red areas represent a mixture of α and ε -Ga₂O₃ phases. At T = 560 °C and z = 50 mm, a secondary ε -Ga₂O₃ phase was observed at the edge areas of the substrate. Therefore, the temperature distribution in the furnace was not uniform. The temperature of the edge areas was higher than that of the central areas on the substrate, resulting in the formation of a mixture of α and ε -Ga₂O₃ phases. At T = 540 °C and z = 50 mm, only the α -Ga₂O₃ phase was observed with 4 Journal of Semiconductors doi: 10.1088/1674-4926/44/6/062803



Fig. 6. (Color online) SEM images of Ga_2O_3 films grown at (a) 500 °C, (b) 520 °C, (c) 540 °C, (d) 560 °C, (e) 580 °C, and (f) photograph of 2-inch α -Ga₂O₃ films prepared at 540 °C.



Fig. 7. (Color online) (a) Optical transmittance spectra of α -Ga₂O₃ films grown at 500 to 580°C. The inset is the partial optical transmittance spectra at the range of wavelength between 200 and 300 nm. (b) The optical bandgaps of α -Ga₂O₃ films grown at 500 to 580 °C.

no additional phases, as shown in Fig. 5(b).

The top-view SEM images of α -Ga₂O₃ grown at various temperatures are presented in Figs. 6(a)-6(e). The evolution of the surface morphology of α -Ga₂O₃ films grown at 500 to 580 °C was observed. As shown in Figs. 6(a)-6(c), a smooth surface morphology of the α -Ga₂O₃ film grown at 500 °C was observed. When the growth temperature was increased to 520 °C, some random three-dimensional (3D) grains were observed. As the temperature increased, the grain-size increased correspondingly. Although some 3D grains were observed, the phase of α -Ga₂O₃ films was maintained as α phase, and the quality of the α -Ga₂O₃ films increased with increasing temperature from 500 to 540 °C. Some irregular surface areas of the films grown at 560 °C or higher temperatures are shown in Figs. 6(d)-6(e), which indicates the occurrence of a phase transition. As shown in the inset of Fig. 6(e), the surface was dominated by hexagonal structures and such areas were dominated by polycrystalline ε -Ga₂O₃, as revealed by the X-ray diffraction (XRD) $2\theta/\omega$ spectrum (not shown).

The optical transmittances of the α -Ga₂O₃ films exceeded 80% in the visible and UV regions, with a sharp cut-off at around 220 nm. As the crystal quality was improved, the trans-

mittance of the α -Ga₂O₃ films also increased. The transmittance of the α -Ga₂O₃ film prepared at 540 °C was greater than 90%. A blue shift was exhibited by the transmittance when the crystal quality increased, as shown in the inset of Fig. 7. Fig. 7(b) depicts the optical bandgap of the films grown at 500 to 580 °C. The optical bandgap of α -Ga₂O₃ could be calculated using Tauc's formula,

$$(\alpha h v)^2 = B(h v - E_{\rm q}), \tag{1}$$

where *h*, *v*, and *B* are Planck's constant, the light frequency, and a constant, respectively^[23]. The bandgap of a-Ga₂O₃ films could be obtained by extrapolating the linear portion of the plot of $(ahv)^2$ versus energy (hv). As the quality was increased, the bandgap of the samples was increased from 5.20 to 5.36 eV. The optical bandgap of the best quality was calculated to be 5.36 eV, which was prepared at 540 °C. When the growth temperature exceeded 540 °C, a second phases of ε -Ga₂O₃ appeared. Affected by the ε -Ga₂O₃ phases, in which the optical bandgap was about 4.9 eV^[24], the optical bandgap of the mixture of ε and α phases slightly reduced.



Fig. 8. (Color online) (a) XRD ω scan of samples grown at 500–560 °C for α -Ga₂O₃ (0006) planes. (b) The locations of P1-5 on 2-inch α -Ga₂O₃ film grown at 540 °C. (c) The rocking curves of α -Ga₂O₃ (0006) planes recorded from five different points (P1–P5). (d) The rocking curves of α -Ga₂O₃ (0006) and (10-14) planes, corresponds to the point of P1.

The quality of the α -Ga₂O₃ films was significantly impacted by the presence of ϵ -Ga₂O₃ phases. Therefore, the growth temperature should be maintained below 560 °C to prepare the pure α -Ga₂O₃ films. A large number of threading dislocations and lattice distortions would appear in the α -Ga₂O₃ film, owing to the lattice and thermal mismatch with sapphire. Fig. 8(a) shows the FWHM of the rocking curves of the α -Ga₂O₃ (0006) planes under different growth conditions. The FWHM of the rocking curves for the (0006) planes decreased with the increasing temperature between 500 to 540 °C. The highest crystal quality of the α -Ga₂O₃ film was obtained at 540 °C, and the FWHM of the scan rocking curves for the (0006) plane was 0.023°. Affected by the ε -Ga₂O₃ phases, the FWHM deteriorated to 0.028° for the film growth at 560 °C. Fig. 8(c) shows the rocking curves of five different points (P1-P5) distributed on the 2-inch substrate, as shown in Fig. 8(b), grown at 540 °C. The FWHMs of the XRCs for the (0006) plane are consistent, ranging from 73 to 89 arcsec. The 2-inch α -Ga₂O₃ films with high uniformity and quality were obtained at T = 540 °C and z = 50 mm. The dislocation densities of the as-grown film prepared at 540 °C were evaluated. The FWHM of the rocking curves for the (10–14) plane under skew-symmetric scan configuration was measured to be 1.17°, as illustrated in Fig. 8(d). The dislocation densities can be approximately calculated using the Eqs. (2) and (3),

$$D_s = \beta_{(0006)}^2 / 4.35 b_s^2, \tag{2}$$

$$D_e = \beta_{(10-14)}^2 / 4.35 b_e^2, \tag{3}$$

where $D_e(D_s)$ and $b_e(b_s)$ are the dislocation densities and lengths of Burgers vectors for edge (screw) types, respectively. $\beta_{(0006)}$ and $\beta_{(10-14)}$ are the FWHM of ω scan for α -Ga₂O₃ (0006) plane and α -Ga₂O₃ (10–14) plane^[13, 25]. The screw and edge dislocation densities in the film prepared at 540 °C were estimated to be 2.3 × 10⁶ and 3.9 × 10¹⁰ cm⁻², respectively.

4. Conclusion

In this work, high-quality 2-inch a-Ga₂O₃ films on sapphire substrates were prepared by mist-CVD. The growth rates were systematically explored, and it was found that the growth rate of the a-Ga₂O₃ increased as the position of the substrate decreased. The growth rate was improved to 1.45 μ m/h, which was faster than previous reports in the mist-CVD method. It is believed that the position of the substrate was the dominant factor affecting the growth rate because the source material microdroplets decreased or even depleted while traveling in the furnace. To obtain high-quality pure a-Ga₂O₃ films, the growth temperatures were adjusted in the range of 500 to 580 °C. When the growth temperature

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perature exceeded 560 °C, *ε*-Ga₂O₃ appeared at the edge of 2-inch sapphire substrate. The α -Ga₂O₃ films which were prepared at 540 °C exhibited the best crystal quality. The FWHM of the (0006) and (10–14) planes were 0.023° (82.8 arcsec) and 1.17°, respectively. The corresponding densities of the screw and edge dislocations were estimated to be 2.3 imes 10^{6} cm⁻² and 3.9×10^{10} cm⁻², respectively. As the quality was improved, the optical transmittances of the α -Ga₂O₃ films were improved. The transmittance of the α -Ga₂O₃ films with the best crystal guality was greater than 90% at the wavelength of 285 nm. Meanwhile, with the crystal quality increased, a blue shift was exhibited in the spectra and the bandgaps of the films were increased from 5.20 to 5.36 eV. Affected by the ε -Ga₂O₃ phases, with an optical bandgap was about 4.9 eV, the optical bandgap of the mixture of the ε and α phases was slightly reduced. Large-scale high-quality α - Ga_2O_3 films contribute to the development of α -Ga₂O₃ materials and devices.

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