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Exploring heteroepitaxial growth and electrical properties of α-Ga_2O_3 films on differently oriented sapphire substrates

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Abstract: This study explores the epitaxial relationship and electrical properties of α-Ga_2O_3 thin films deposited on a-plane, m-plane, and r-plane sapphire substrates. We characterize the thin films by X-ray diffraction and Raman spectroscopy, and elucidate thin film epitaxial relationships with the underlying sapphire substrates. The oxygen vacancy concentration of α-Ga_2O_3 thin films on m-plane and r-plane sapphire substrates are higher than α-Ga_2O_3 thin film on a-plane sapphire substrates. All three thin films have a high transmission of over 80% in the visible and near-ultraviolet regions, and their optical bandgaps stay around 5.02–5.16 eV. Hall measurements show that the α-Ga_2O_3 thin film grown on r-plane sapphire has the highest conductivity of 2.71 S/cm, which is at least 90 times higher than the film on a-plane sapphire. A similar orientation-dependence is seen in their activation energy as revealed by temperature-dependent conductivity measurements, with 0.266, 0.079, and 0.075 eV for the film on a-, m-, r-plane, respectively. The origin of the distinct transport behavior of films on differently oriented substrates is suggested to relate with the distinct evolution of oxygen vacancies at differently oriented substrates. This study provides insights for the substrate selection when growing α-Ga_2O_3 films with tunable transport properties.

Key words: gallium oxide; thin film epitaxy; orientation; oxygen vacancy; electrical properties

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1. Introduction

As an emerging third-generation semiconductor material, gallium oxide (Ga_2O_3) has superior properties such as wide band gap, high breakdown electric field, and strong radiation resistance, which can more effectively serve the emerging needs of power electronic device development, such as Schottky barrier diodes (SBDs)[1, 2], heterojunction diodes[3, 4], metal-oxide semiconductor field-effect transistors (MOSFETs)[5, 6], etc. compared with Si, SiC and GaN[5, 7–9]. It has a wide range of application prospects in the field of advanced information technology and new energy.

Ga_2O_3 has five crystalline phases: α, β, δ, γ and ε. Among them, the monoclinic structure β-Ga_2O_3 is a thermally stable phase at high temperature, and the other four phases are all metastable crystalline phases[10–16]. But, due to the lack of symmetry of monoclinic β-Ga_2O_3 compared with the hexagonal system, there is a problem of "twisting" in the heteroepitaxial, which is a major difficulty in the crystal quality of heteroepitaxial thin films[17]. Compared with β-Ga_2O_3, the corundom structure α-Ga_2O_3 is one of the metastable phases and has a wider band gap and higher breakdown field strength, making it more suitable for the fabrication of power electronic devices[18]. Both α-Ga_2O_3 (a = 4.98 Å, c = 13.43 Å)[19] and sapphire (a = 4.76 Å, c = 12.99 Å) (PDF: 46-1212) belong to a corundum structure, and the lattice mismatch between them is small. Therefore, sapphire is well suited as a substrate for epitaxial α-Ga_2O_3. We have mainly selected sapphire substrates with three different crystal orientations: a-plane ($11\bar{2}0$), m-plane ($10\bar{1}0$) and r-plane ($01\bar{1}2$). The study of the electrical properties of α-Ga_2O_3 thin films is an important basis for the fabrication of high-performance devices. However, the effect of substrate crystal orientation on the thin film properties remains contentious, including the crystalline quality and the electrical properties of the thin films[7, 20–23]. The anisotropy of the electrical properties of Ga_2O_3 may be follows from the anisotropy of the long-range electron–phonon interaction, as well as from the conduction band anisotropy[24]. On the other hand, the defects also play a crucial role in the electrical properties[25]. Therefore, there is a significant need to investigate the effect of substrate crystal orientation on the heteroepitaxial and electrical properties of the films.

The main methods currently used to grow Ga_2O_3 films are radio frequency magnetron sputtering (RFMS)[25, 26], metal organic chemical vapor deposition (MOCVD)[14, 27–29], molecular beam epitaxy (MBE)[7, 8]. Compared to the above methods, pulsed laser deposition (PLD) has the following outstanding

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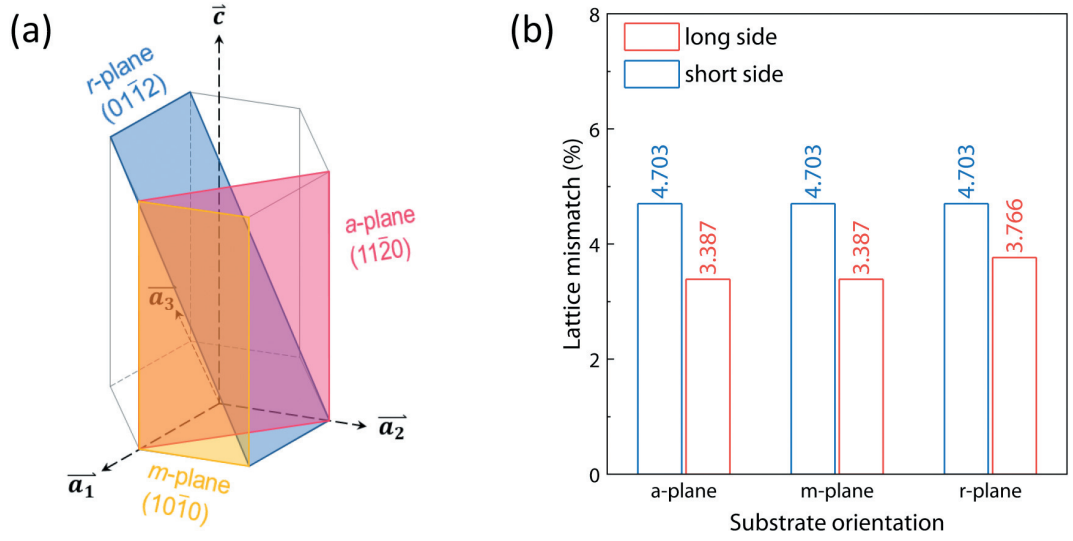

Fig. 1. (Color online) (a) Schematic diagram of various crystal planes in the corundom structure. (b) The lattice mismatch between different crystal planes α-Ga_2O_3 films and corresponding sapphire substrates.

advantages: fast film growth, easy doping, easy preparation of films with the same stoichiometric ratio as the target, easy adjustment of the growth process[10, 11, 13, 30].

In this work, we have systematically investigated the epitaxial relationships and the differences in electrical properties of epitaxial α-Ga_2O_3 films on a-plane, m-plane, and r-plane sapphire substrates using the PLD technique. We found that the epitaxial films have the same corundum structure and the growth orientation as the substrate, respectively. The conductivity shows an increasing trend from a-plane to m-plane to r-plane, which may be related to oxygen vacancy.

2. Experimental

The 1% Sn-doped α-Ga_2O_3 thin films were deposited with the same growth condition on a-plane, m-plane, and r-plane sapphire substrates by PLD (SKY Technology Development Co., Ltd. Chinese Academy of Sciences), at about 800 °C, the oxygen pressure was fixed at 0.1 mTorr. The epitaxial growth relationship and crystalline structure of α-Ga_2O_3 film were analyzed by X-ray diffraction (XRD, D8 Discover). The structure of the α-Ga_2O_3 thin films was also confirmed by Raman spectroscopy (Renishaw inVia Reflex) at room temperature using a laser beam of 532 nm. Film transmission in the range of 200–800 nm was measured by a UV-visible spectrophotometer (UV-vis, Lambda 1050). The elemental composition and chemical state were characterized by X-ray photoelectron spectroscopy (XPS, Kratos AXIS SUPRA) using Mg Kα ($h\nu$ = 1253.6 eV) as the excitation source. The electrode spacing and effective electrode area were controlled using a direct-write optical lithography machine (MicroWriter ML3) and a lift-off process. The electrical properties at room temperature and varies temperatures were measured in the Hall measurement system (HL-5500PC) with a 0.5 T magnet, and in a semiconductor parameter analyzer (Keithley 4200-SCS), respectively.

3. Results and discussion

The schematic diagram of different crystal planes in the corundom structure are shown in Fig. 1(a). The lattice mismatch (δ) is calculated according to the following equation: $\delta = (a_e - a_s)/a_s$. Here, a_e and a_s indicate the lattice constants of epitaxial thin films and substrates, respectively. The lattice mismatches between different crystal planes α-Ga_2O_3 films and corresponding sapphire substrates are shown in Fig. 1(b). It is easy to find that the lattice mismatch between r-plane sapphire and r-plane α-Ga_2O_3 of the long side is larger than that of the a-plane and m-plane, which may be the reason why it is more difficult to epitaxy Ga_2O_3 on r-plane sapphire.

We determine the crystalline phase of Ga_2O_3 using XRD and Raman scattering. As shown in Fig. 2(a), the (11$\bar{2}$0) and (22$\bar{4}$0) diffraction peaks of α-Ga_2O_3 are accompanied by (11$\bar{2}$0) and (22$\bar{4}$0) diffraction peaks of a-plane sapphire, the (30$\bar{3}$0) diffraction peak of α-Ga_2O_3 are accompanied by (30$\bar{3}$0) diffraction peak of m-plane sapphire, and the (01$\bar{1}$2), (02$\bar{2}$4) and (03$\bar{3}$6) diffraction peaks of α-Ga_2O_3 are accompanied by (01$\bar{1}$2), (02$\bar{2}$4) and (03$\bar{3}$6) diffraction peaks of r-plane sapphire. The structure of α-Ga_2O_3 thin films is also confirmed by Raman scattering. The XRD θ–2θ scan patterns of the above three samples all show that the crystal orientation of the α-Ga_2O_3 films is almost consistent with the sapphire substrate.

XRD ϕ-scan mode measurements are used to further determine the in-plane epitaxial growth relationship of α-Ga_2O_3 film relative to sapphire substrates. As shown in Fig. 2(b), the characteristic (30$\bar{3}$0) of a-plane α-Ga_2O_3 thin film and (30$\bar{3}$0) of a-plane sapphire substrate appear at the same rotational angle of ϕ, suggesting that the α-Ga_2O_3 thin film has the same corundom structure as the sapphire substrate. The (02$\bar{2}$4) of the m-plane α-Ga_2O_3 thin film and sapphire substrate, and the (01$\bar{1}$2) of r-plane α-Ga_2O_3 thin film and sapphire substrate show similar results to those above.

Fig. 2(c) shows the Raman shift of α-Ga_2O_3 thin films grown on a-, m-, r-plane sapphire substrates. The Raman peaks belonging to α-Ga_2O_3 thin film are mainly located at 430.4, 575.8 cm^{-1}, which is consistent with reported values[31, 32]. The Raman results are consistent with the XRD results. The XRD rocking curves of a-plane α-Ga_2O_3 (11$\bar{2}$0) peak, m-plane α-Ga_2O_3 (30$\bar{3}$0) peak, and r-plane α-Ga_2O_3 (02$\bar{2}$4) peak are shown in Fig. 2(d). The fullwidth at half maxi-

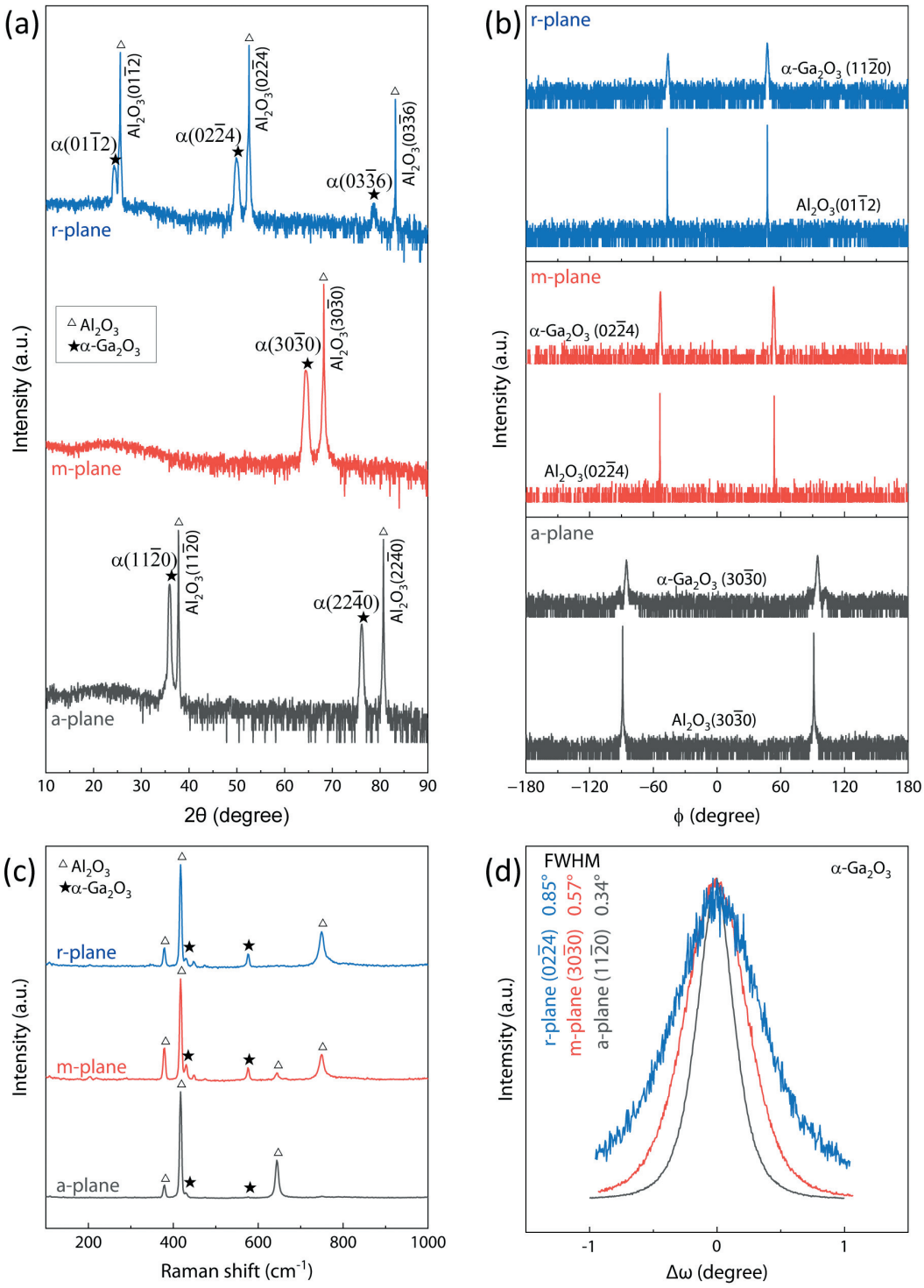

Fig. 2. (Color online) (a) XRD θ–2θ full scans of α-Ga_2O_3 thin films grown on a-plane, m-plane, and r-plane sapphire substrates. (b) XRD ϕ-scans of (30$\bar{3}$0) plane, (02$\bar{2}$4) plane, and (11$\bar{2}$0) plane of corresponding α-Ga_2O_3 films and the underlying sapphire, respectively. (c) Raman scattering spectra of α-Ga_2O_3 thin films grown on a-plane, m-plane, and r-plane sapphire substrates. (d) XRD rocking curves around the α-Ga_2O_3 (11$\bar{2}$0) peak grown on a-plane sapphire, α-Ga_2O_3 (30$\bar{3}$0) peak grown on m-plane sapphire, and α-Ga_2O_3 (02$\bar{2}$4) peak grown on r-plane sapphire.

mum (FWHM) of the ω-scan rocking curves of (11$\bar{2}$0) peak, (30$\bar{3}$0) peak, and (02$\bar{2}$4) peak are 0.34°, 0.57°, and 0.85°, respectively. This indicates that the crystalline quality of a-plane α-Ga_2O_3 is greater at the a-plane than m-plane and r-plane, which is affected by multiple factors, including the different crystalline quality of the substrate with different orientations, the lattice mismatch, and the nucleation kinetics for different orientations[17, 33, 34].

Based on the above test results, it is not difficult to deduce the growth behaviors of α-Ga_2O_3 thin films grown on differently-oriented sapphire substrates. As shown in Fig. 3, the unit cell arrangement of α-Ga_2O_3 thin films is almost the same as that of the corresponding sapphire substrates.

Then, we probed the defect density and distribution of differently-oriented α-Ga_2O_3 films using XPS. Fig. 4 shows the O 1*s* core level spectra of various plane α-Ga_2O_3 thin films. We calibrate the data with a standard binding energy of 284.8 eV for the C 1s peak[35]. The XPS O 1*s* core level spectra of the films

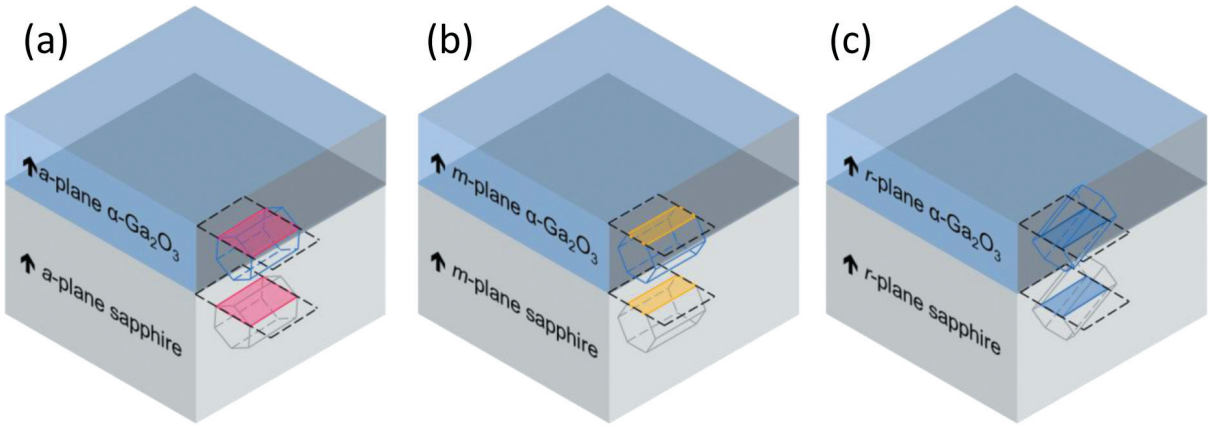

Fig. 3. (Color online) Schematic of growth relation of α-Ga_2O_3 grown on (a) a-plane, (b) m-plane, and (c) r-plane sapphire substrates by PLD.

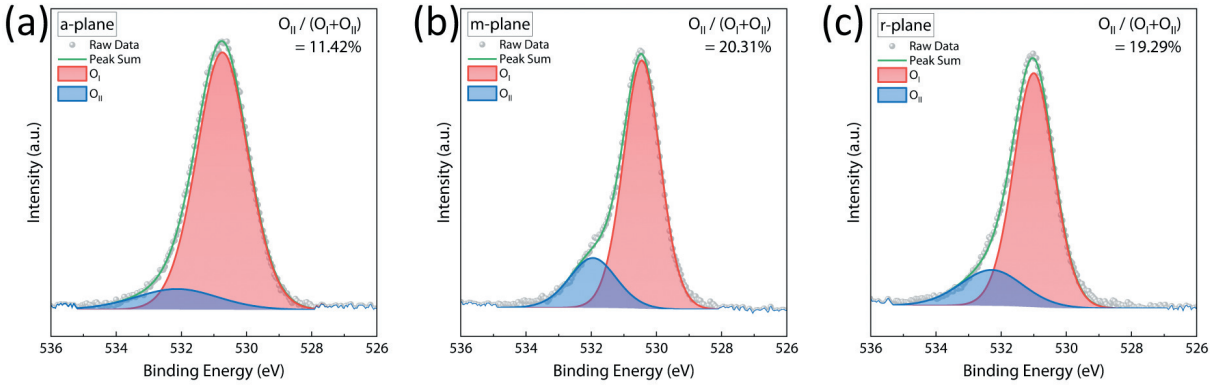

Fig. 4. (Color online) XPS spectra of O 1*s* peak of α-Ga_2O_3 films grown on (a) a-plane, (b) m-plane, and (c) r-plane sapphire substrates.

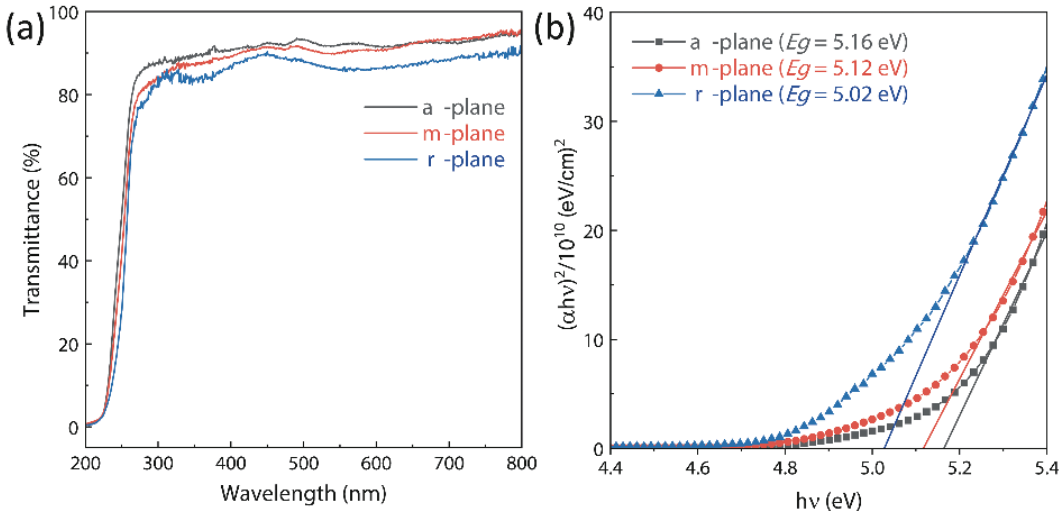

Fig. 5. (Color online) (a) Transmittance spectra and (b) Tauc plot of the a-plane, m-plane, and r-plane α-Ga_2O_3 thin films grown on various plane sapphire substrates.

are divided into two individual peaks and well fitted with the sum Gaussian functions. The positions of the two peaks correspond to lattice oxygen of Ga_2O_3 (O_I) and oxygen vacancies (O_{II}), respectively[26]. We can use $O_{II}/(O_I+O_{II})$ to reflect the concentration of oxygen vacancies. The oxygen vacancy concentration of the a-plane, m-plane, and r-plane α-Ga_2O_3 are 11.42%, 20.31%, and 19.29%, respectively.

Furthermore, the UV-vis transmission spectra for as grown α-Ga_2O_3 epitaxial films on differently-oriented sapphires are illustrated in Fig. 5(a). Both films have high transparency (>80%) in the visible and near-UV regions. As shown in Fig. 5(b), the optical band gap (E_g) of the α-Ga_2O_3 films can be evaluated using the plot of $(\alpha h\nu)^2$ vs $h\nu$, where α is the absorption coefficient and $h\nu$ is the photon energy. a-plane, m-plane, and r-plane α-Ga_2O_3 films have an estimated bandgap of 5.16, 5.12, and 5.02 eV, respectively.

In order to explore the electrical transport properties of differently-oriented α-Ga_2O_3 thin films, Hall measurement is performed in a conventional van der Pauw geometry. Table 1 shows the results of Hall measurements. The conductivity (σ)

Table 1. Film thicknesses, conductivities, mobilities, and carrier concentrations for α-Ga_2O_3 thin films grown on a-, c- and r-plane sapphire substrates.

Substrate	Thickness (nm)	σ (S/cm)	μ (cm^2/(V·s))	n (10^{17} cm^{-3})
a-sapphire	302	Exceed test limit (<0.03)	–	–
m-sapphire	280	0.29	12.2	1.45
r-sapphire	273	2.71	1.35	125

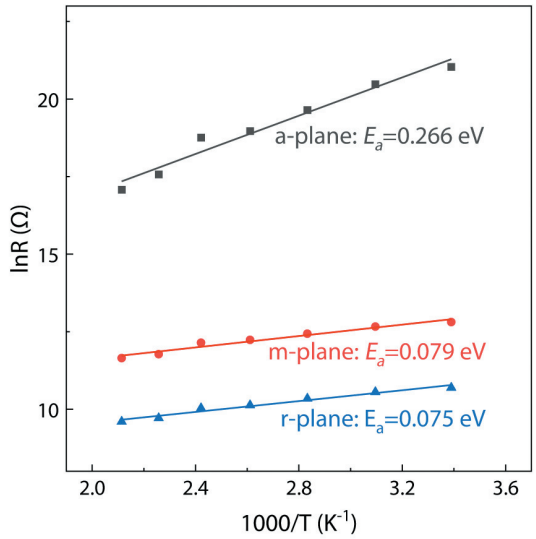

Fig. 6. (Color online) Fitting plots of temperature-dependent resistance deriving the activation energy for a-plane, m-plane, and r-plane α-Ga_2O_3 thin films.

of the α-Ga_2O_3 thin film epitaxial on a-plane sapphire exceeds the measurement capacity of the Hall tester. For α-Ga_2O_3 thin film epitaxial on m-plane sapphire, Hall measurements indicate that the film is n-type conductive with a high conductivity of 0.29 S/cm, a mobility (μ) of 12.2 cm^2/(V·s) and a carrier concentration (n) of 1.45×10^{17} cm^{-3}. While the α-Ga_2O_3 thin film epitaxial on r-plane sapphire has a higher conductivity of 2.71 S/cm, a mobility of 1.35 cm^2/(V·s) and a high carrier concentration of 1.25×10^{19} cm^{-3}.

The temperature dependence of the resistance in the range from 295 to 473 K for the Sn-doped α-Ga_2O_3 thin films are shown in Fig. 6. The activation energy (E_a) is derived from the linear fit of the ln(R)–T plot using $R \propto \exp(E_a/k_BT)$, where R is the resistance, k_B is the Boltzmann constant, and T is the temperature[10, 36]. The estimated activation energy for α-Ga_2O_3 epitaxial on a-plane, m-plane and r-plane sapphire substrates are 0.266, 0.079, and 0.075 eV, respectively. The value of activation energy is used to quantify the thermal ionization of defect states[28].

The α-Ga_2O_3 thin films on m-plane and r-plane sapphire substrates are far more conducting than α-Ga_2O_3 thin film on a-plane sapphire substrate under similar thin film growth conditions. It is interesting that the oxygen vacancy concentration of α-Ga_2O_3 thin films on m-plane and r-plane sapphire substrates are also higher than α-Ga_2O_3 thin film on a-plane sapphire substrates. It is speculated that the conductivity behavior of α-Ga_2O_3 thin films may be related to the oxygen vacancy concentration, and the formation energy could strongly depend on the lattice orientation, thus leading to drastical differences in the dopant activation and carrier transport[29, 37, 38]. Further studies on the mechanism of the effect of anisotropy and crystallinity on electrical conductivity are expected.

4. Conclusions

We have investigated the Sn-doped α-Ga_2O_3 thin films deposited on a-plane, m-plane, and r-plane sapphire substrates by PLD. Due to the same corundum structure, the out-of-plane and in-plane alignment of α-Ga_2O_3 films is almost identical to that of the corresponding oriented sapphire substrate. The FWHMs of ω-scan rocking curves of the a-plane, m-plane, and r-plane α-Ga_2O_3 thin films are 0.34°, 0.57°, and 0.85°, respectively. The r-plane α-Ga_2O_3 thin film has the highest conductivity, the m-plane α-Ga_2O_3 thin film the second, and the a-plane α-Ga_2O_3 thin film dramatically more insulating. A similar trend is also observed in their activation energy. This could be understood by that the orientation difference leads to the concentration variation of oxygen defects in differently oriented α-Ga_2O_3 thin films, further causing differences in the electrical properties of the thin films.

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