

Homoepitaxial growth of (100) Si-doped β -Ga₂O₃ films via MOCVD

Wenbo Tang^{1,2}, Xueli Han^{3,4}, Xiaodong Zhang^{1,2}, Botong Li^{1,2}, Yongjian Ma^{1,2}, Li Zhang², Tiwei Chen^{1,2}, Xin Zhou², Chunxu Bian², Yu Hu^{1,2}, Duanyang Chen³, Hongji Qi^{3,4,†}, Zhongming Zeng^{1,2}, and Baoshun Zhang^{1,2,†}

¹School of Nano-Tech and Nano-Bionics, University of Science and Technology of China, Hefei 230026, China

²Nanofabrication facility, Suzhou Institute of Nano-Tech and Nano-Bionics, Chinese Academy of Sciences, Suzhou 215123, China

³Research Center of Laser Crystal, Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences, Shanghai 201800, China

⁴Hangzhou Institute of Optics and Fine Mechanics, Hangzhou 311421, China

Abstract: Homoepitaxial growth of Si-doped β -Ga₂O₃ films on semi-insulating (100) β -Ga₂O₃ substrates by metalorganic chemical vapor deposition (MOCVD) is studied in this work. By appropriately optimizing the growth conditions, an increasing diffusion length of Ga adatoms is realized, suppressing 3D island growth patterns prevalent in (100) β -Ga₂O₃ films and optimizing the surface morphology with [010] oriented stripe features. The slightly Si-doped β -Ga₂O₃ film shows smooth and flat surface morphology with a root-mean-square roughness of 1.3 nm. Rocking curves of the (400) diffraction peak also demonstrate the high crystal quality of the Si-doped films. According to the capacitance–voltage characteristics, the effective net doping concentrations of the films are $5.41 \times 10^{15} - 1.74 \times 10^{20} \text{ cm}^{-3}$. Hall measurements demonstrate a high electron mobility value of $51 \text{ cm}^2/(\text{V}\cdot\text{s})$, corresponding to a carrier concentration of $7.19 \times 10^{18} \text{ cm}^{-3}$ and a high activation efficiency of up to 61.5%. Transmission line model (TLM) measurement shows excellent Ohmic contacts and a low specific contact resistance of $1.29 \times 10^{-4} \Omega\cdot\text{cm}^2$ for the Si-doped film, which is comparable to the Si-implanted film with a concentration of $5.0 \times 10^{19} \text{ cm}^{-3}$, confirming the effective Si doping in the MOCVD epitaxy.

Key words: homoepitaxial growth; MOCVD; Si-doping films; high activation efficiency; Ohmic contacts

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1. Introduction

As an ultrawide bandgap semiconductor material for next-generation high power electronics, β -Ga₂O₃ has attracted enormous attention due to its high breakdown field strength of 8 MV/cm, a large Baliga's figure of merit of 3444, and the availability of high-quality melt-grown native substrates^[1–3]. Many β -Ga₂O₃ electronic devices such as Schottky barrier diodes^[4–6], MOSFETs^[7, 8], FinFETs^[9–11], and CAVETs^[12, 13] have already been successfully demonstrated, which reveal their great potential in high-power electronics. To further improve their performance, it is essential to obtain epitaxial β -Ga₂O₃ films with high crystal quality. To date, the homoepitaxy of β -Ga₂O₃ films is still confronted with various challenges such as twins and stacking faults, despite the perfect crystalline match between the film and the substrate due to immature research status^[14–17]. Various epitaxial growth techniques for β -Ga₂O₃ films have been demonstrated over the past few years, such as halide vapor phase epitaxy (HVPE)^[18, 19], molecular beam epitaxy (MBE)^[20, 21], low-pressure chemical vapor deposition (LPCVD)^[22, 23], metalorganic

chemical vapor deposition (MOCVD)^[24–26], and carbothermal reduction rapid growth method^[27]. With the available (100), (010), (001), and (201) orientated substrates^[28], the homoepitaxy of β -Ga₂O₃ films exhibits a great development prospect. Owing to the anisotropic characteristics, the crystal orientation of β -Ga₂O₃ substrates has an enormous impact on the quality of the epilayers. Among them, (100) oriented substrates can be easily prepared, but its epitaxy is still challenging due to the preferred cleavage plane with the lowest surface energy^[29]. 2D island growth mode usually plays a dominant role due to the limited diffusion length of the (100) surface adatoms^[30], which causes deterioration of the crystal quality. Many efforts have been made on the homoepitaxial growth of (100) β -Ga₂O₃ thin films^[20, 31, 32]. In addition, the high concentration n-type doping in β -Ga₂O₃ still remains challenging, and Si-ion implantation is usually used in the Ohmic contact region nowadays^[33–35]. Therefore, the homoepitaxial growth of (100) Si-doped β -Ga₂O₃ thin films with high concentration is important for β -Ga₂O₃ devices.

In this work, (100) β -Ga₂O₃ films with Si-doping were grown on semi-insulating (100) substrates by MOCVD, which is a preferred growth method for semiconductor device technologies at the industrial level. The suppression of the 2D island growth mode was demonstrated through the optimization of the growth conditions, such as VI/III ratio, temperature and chamber pressure. A promotion of the surface mor-

Correspondence to: H J Qi, qhj@siom.ac.cn; B S Zhang, bszhang2006@sinano.ac.cn

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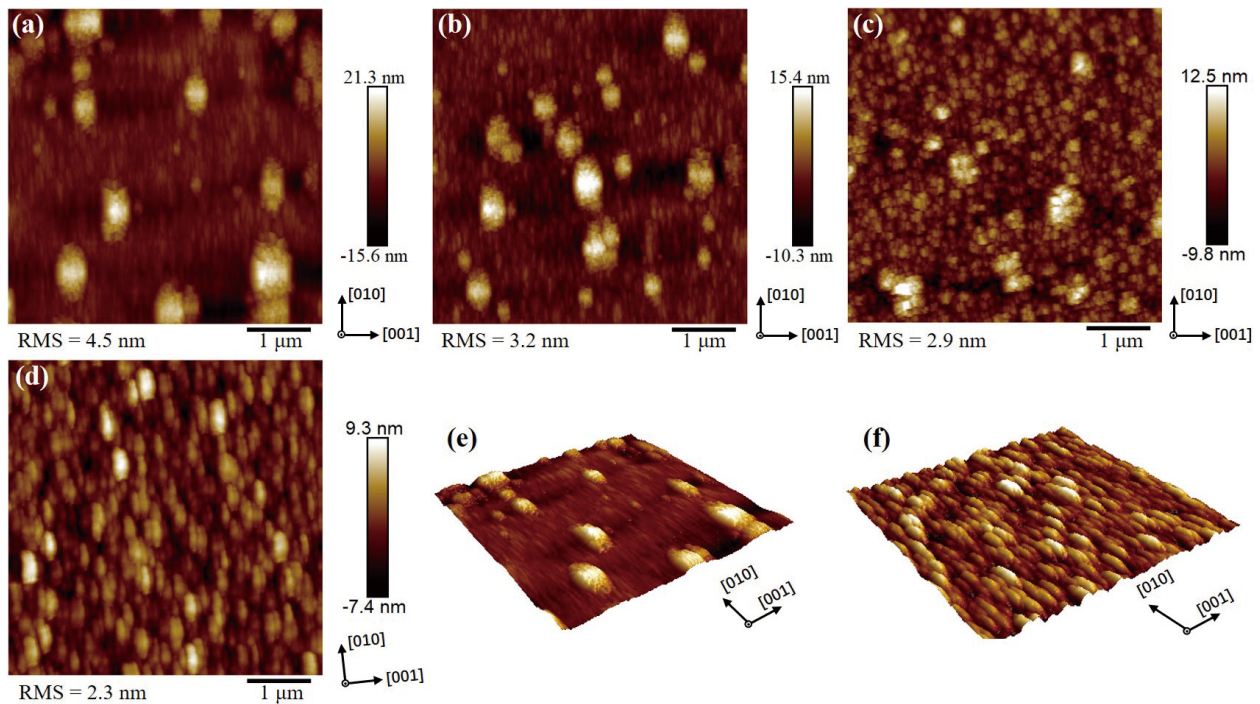


Fig. 1. (Color online) Surface morphologies of β -Ga₂O₃ films grown in different conditions. AFM topography images of sample (a) A₁, (b) A₂, (c) A₃ and (d) A₄. 3D AFM images of sample (e) A₁ and (f) A₄.

phology of the epitaxial films from Si doping were confirmed, which proved the facilitation of the growth kinetics. The obtained (100) β -Ga₂O₃ films exhibited a smooth surface with high crystal quality, even for the heavy doped films. Excellent Ohmic contacts and high current density were achieved.

2. Experiments

The (100) β -Ga₂O₃ epitaxial films were grown via MOCVD on semi-insulating β -Ga₂O₃ substrates with no intentional off-cut produced by Hangzhou Fujia Gallium Technology Co., Ltd. Before the epi-growth, the substrates were immersed in piranha solution and then hydrofluoric acid to remove the possible Si contaminants on the substrate surface and followed with in-situ annealing for 15 min at 800 °C under an O₂ atmosphere. Triethylgallium (TEGa), high-purity oxygen gas (O₂), and silane (SiH₄) were used as Ga and O precursors and Si dopants, respectively. High-purity nitrogen (N₂) was used as the carrier gas. The growth temperature was set at 800–900 °C with the pressure kept at 10 kPa. The growth rates of the epitaxial films were in the range of 4–7 nm/min. Atomic force microscopy (AFM), high-resolution X-ray diffraction (HRXRD), scanning electron microscopy (SEM), time-of-flight secondary ion mass spectroscopy (ToF-SIMS), and Hall measurement were applied to characterize the material properties of the (100) β -Ga₂O₃ films. A Keysight B1505A Power Device Analyzer was used to measure the electrical characteristics of the devices based on the epi-films.

3. Results and discussion

Compared to (010) and (001) β -Ga₂O₃, the homoepitaxy of (100) β -Ga₂O₃ films is up against more issues owing to the low surface energy and the facile cleavage plane^[36]. Slow growth rate^[28] and 2D island growth mode^[37] are commonly seen in the (100) β -Ga₂O₃ films growth process. As the absorption and diffusion length of Ga adatoms on the (100) surface

define low incorporation efficiency, random nucleation and island formation will easily occur. And the lack of energetically favorable lattice sites induces more formation of 3D islands by the encounter of Ga adatoms^[30], resulting in the roughening of the surface. To improve the surface morphology and get a smooth film, suppressing the formation of 3D islands is essential. Usually, an O-rich regime is adopted in the growth environment, and the decrease of the O/Ga ratio can bring an increasing diffusion length of Ga adatoms^[31]. In the experiments, the TEGa molar flow rate was 68.2 μ mol/min. The β -Ga₂O₃ films grown in different conditions with unintentional doping were labeled as sample A₁, A₂, A₃ and A₄, respectively. Fig. 1(a) shows the AFM image of sample A₁ grown at 10 kPa and 850 °C with the O/Ga ratio of 1308, showing a root-mean-square (RMS) roughness of 4.5 nm in a $5 \times 5 \mu\text{m}^2$ scanning area. Scattered islands on the surface contribute to the large roughness and deteriorate the crystal quality. Similar morphology was reported in Ref. [36]. In addition, the islands, 0.3–1.5 μm long and 0.2–0.7 μm wide, with a height of no more than 27 nm, show a fixed orientation along the [010] direction, which is consistent with the stripes in the region without islands. The aligned islands also indicate strong anisotropic characteristics in growth. To suppress the formation of islands, a slightly smaller O/Ga ratio of 1250 was implemented. As can be seen from Fig. 1(b), islands get smaller with an increasing diffusion length of Ga adatoms, accompanied by a smaller RMS roughness of 3.2 nm. To further eliminate the islands, the growth temperature was decreased from 850 to 800 °C. It is found that the big islands disappear and small grains appear instead. The grains have the same arrangement oriented along the [010] direction and some of them even stack as hills. As exhibited in Fig. 1(c), the film surface was filled with aligned grains and had a smaller RMS roughness of 2.9 nm. As the pressure drops to 8 kPa, the length of the grains extends along the [010] direction with

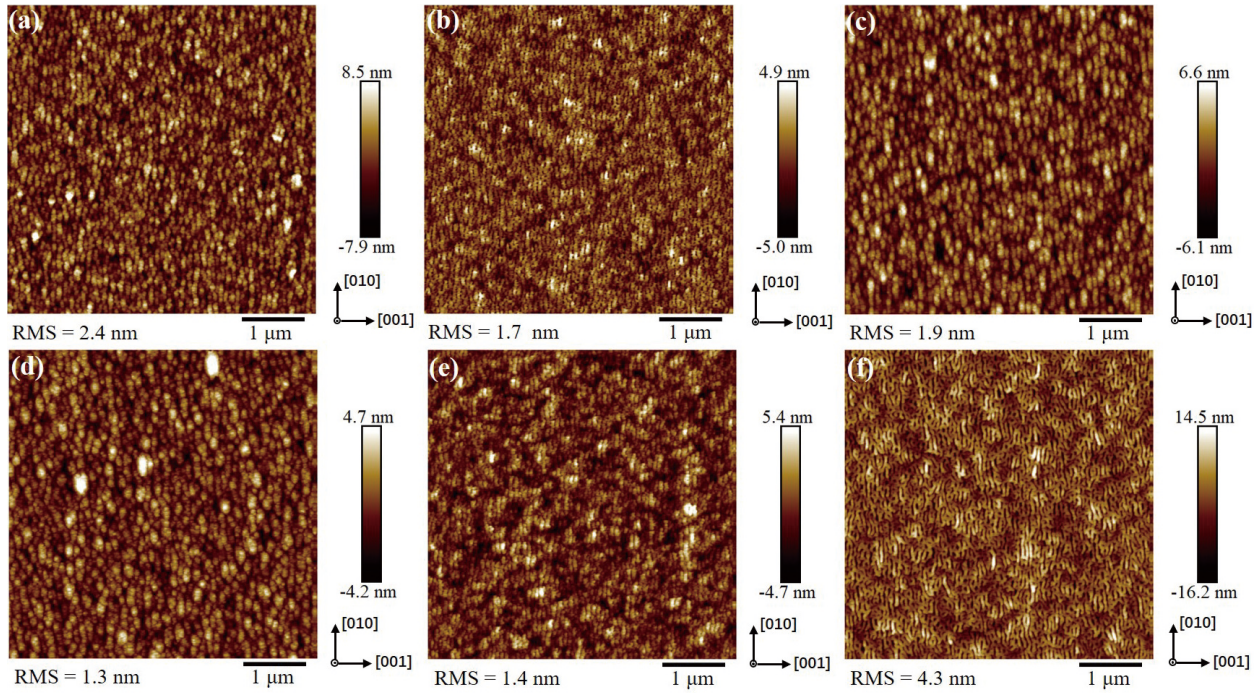


Fig. 2. (Color online) AFM images of the Si-doped β -Ga₂O₃ films grown with different flow rate of SiH₄. (a) B₂, 0.5 sccm. (b) B₃, 1.2 sccm. (c) B₄, 3 sccm. (d) B₅, 6 sccm. (e) B₆, 10 sccm. (f) B₇, 20 sccm.

granular features, which implies that lateral growth along the [010] direction gets stronger. The granular surface morphology is shown in Fig. 1(d), with a length of ~ 500 nm, a width of ~ 120 nm, and a height of ~ 6 nm. The transition from island morphology to granular morphology attributes to the relatively low RMS roughness of 2.3 nm. Figs. 1(e) and 1(f) are the 3D AFM images corresponding to Figs. 1(a) and 1(d), respectively. It can be intuitively seen that oriented 3D islands disperse on the film surface in Fig. 1(e) and oriented granules align on the film surface in Fig. 1(f). The feature of the [010] orientation confirms the anisotropic growth of β -Ga₂O₃. Similar morphology was also reported in the homoepitaxy of (201) β -Ga₂O₃ films^[38, 39].

Subsequently, the homoepitaxial growth of (100) β -Ga₂O₃ films with different Si-doping concentration was investigated. The samples are labeled as B₁, B₂, B₃, B₄, B₅, B₆ and B₇, with the flow rate of SiH₄ in the growth of 0, 0.5, 1.2, 3, 6, 10, and 20 sccm, respectively. The growth rate of the films was almost unchanged with the introduction of SiH₄. Fig. 2 shows the surface morphologies of the films. Sample B₁ is undoped and has a similar surface morphology to sample A₄, therefore its AFM image is not shown here. It can be seen that the introduction of Si atoms brings a decrease of RMS roughness to the epitaxial films. With 0.5 sccm SiH₄ flow rate, the morphology transforms from granules into narrow and continuous stripes with a width of ~ 80 nm, as shown in Fig. 2(a). The RMS roughness of sample B₂ is 2.4 nm and stays almost unchanged. Thinner continuous stripes with a width of ~ 60 nm were found for sample B₃, the films grown with 1.2 sccm SiH₄, resulting in a decreased RMS roughness of 1.7 nm, shown in Fig. 2(b). By gradually further increasing the flow rate of SiH₄, similar morphologies with oriented stripes appeared on the film surface. Figs. 2(c)–2(e) display the surface morphologies of sample B₄, B₅, and B₆, whose RMS roughness is 1.9, 1.3, and 1.4 nm, respectively. The incorporation of Si atoms can suppress random nucleation and island forma-

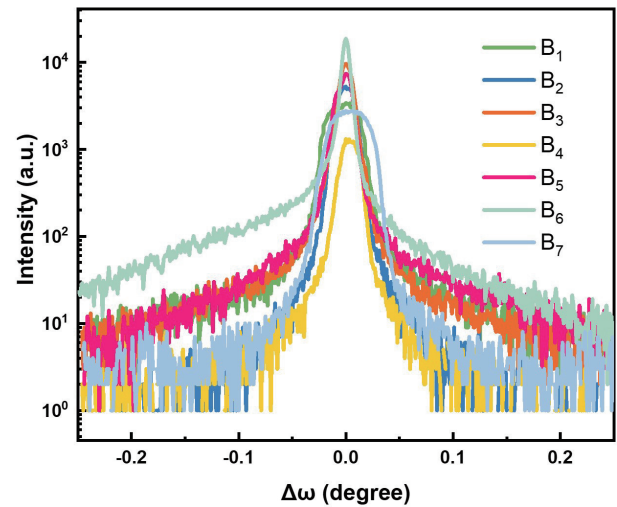


Fig. 3. (Color online) The HRXRD rocking curves of the (400) diffraction peak for the homoepitaxial films.

tion during (100) β -Ga₂O₃ growth to a certain extent through the preferred bonding species of Si adatoms to the O atoms, where a similar mechanism was reported in the (Al_xGa_{1-x})₂O₃ growth^[30]. As a result, a smooth and flat surface was obtained through light Si doping. The coalescence of the original 2D islands was promoted by the distributed Si nucleation sites on the growth surface, facilitating the step-flow growth mode^[16]. However, when the doping amount exceeds a certain degree, such as 20 sccm SiH₄, the stripes start to lose their orientation and reveal an undulating surface with increasing RMS roughness. Fig. 2(f) depicts the surface morphology of sample B₇, which has curly stripes and an RMS roughness of 4.3 nm. The weak anisotropic growth may contribute to the above morphology change.

The effect of Si incorporation on the crystal quality of the epitaxial films was studied through HRXRD. Rocking curves of the (400) diffraction peak of β -Ga₂O₃ were shown in Fig. 3. All

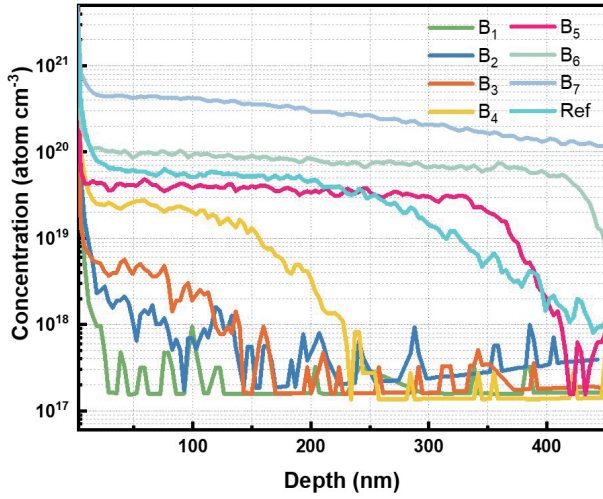


Fig. 4. (Color online) ToF-SIMS profiles of Si for the epitaxial films.

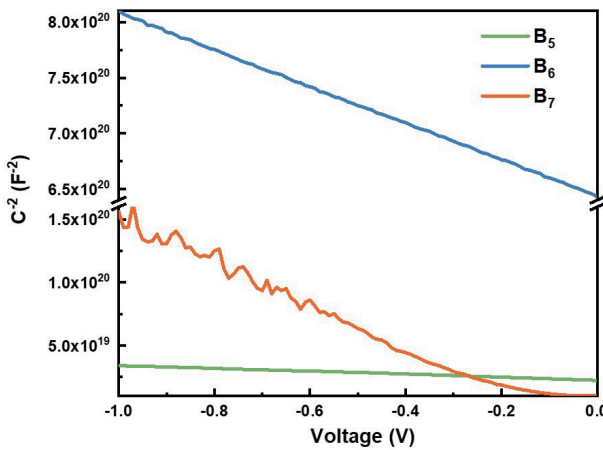


Fig. 5. (Color online) Plot of $1/C^2-V$ for the epitaxial β -Ga₂O₃ films with high doping levels.

the films show good crystal quality with the full-width at high maximum (FWHM) of the (400) peak below 60 arcsec, except for the highly doped sample B₇, whose SiH₄ flow rate is 20 sccm. It should be noted that all the prepared films have a thickness of ~400 nm, most of the diffraction signal may come from the substrate as a result. However, if the Si doping leads to a deterioration in crystal quality, a large FWHM should be present, such as for sample B₇, which has a FWHM of 119 arcsec. This suggests new defects are generated in the epitaxial films, and the excess incorporation of Si atoms deteriorates the crystal quality of the β -Ga₂O₃ films. Therefore, to ensure the high crystal quality of the film, the flow rate of SiH₄ in the growth should not exceed 20 sccm.

ToF-SIMS was applied to identify the concentration of Si atoms in the grown β -Ga₂O₃ films. As depicted in Fig. 4, the concentrations of Si atoms of all the films are in the range of $1.57 \times 10^{17} - 2.83 \times 10^{20} \text{ cm}^{-3}$. Note that the detection limit of Si is $1.57 \times 10^{17} \text{ cm}^{-3}$, and a Si-ion implanted film with a concentration of $5.0 \times 10^{19} \text{ cm}^{-3}$ is used as the standard sample. For sample B₄, B₅, B₆ and B₇, their Si concentrations are 2.63×10^{19} , 3.46×10^{19} , 7.73×10^{19} , and $2.83 \times 10^{20} \text{ cm}^{-3}$, respectively. Such high doping levels with high crystal quality could be achieved only by Si-ion implantation previously^[8, 13, 35, 40–42]. While ion implantation usually results in large lattice damage and requires high-temperature activation^[40], which may cause adverse effects on the subsequent process^[43, 44]. As a

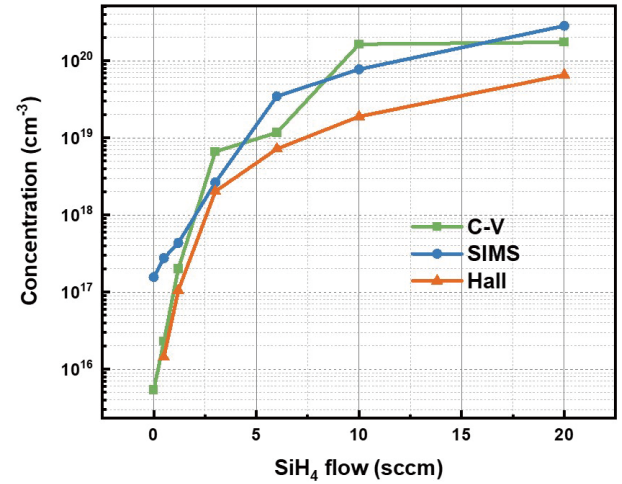


Fig. 6. (Color online) Comparison of the concentrations extracted from C-V, SIMS, and Hall measurements.

result, the technical route of Si doping in the process of epitaxy can bring more convenience and solutions to semiconductor processing technology.

A Ti (50 nm)/Au (100 nm) contact was deposited on the film surface as the Ohmic electrode, followed by 475 °C annealing in N₂ atmosphere for 1 min. And a Ni (50 nm)/Au (100 nm) contact was deposited as the Schottky electrode subsequently. Capacitance-voltage (C-V) characteristics of the epitaxial β -Ga₂O₃ films were measured, and the effective net doping concentration ($N_D - N_A$) can be extracted from the slope of the $1/C^2-V$ plots in Fig. 5. The equation is given as

$$\left(\frac{1}{C}\right)^2 = \frac{2(V_{bi} + V_R)}{e\epsilon_s(N_D - N_A)}, \quad (1)$$

where C is the capacitance per unit area, e is the electron charge, V_{bi} is the built-in potential difference, V_R is the applied voltage, and ϵ_s is the dielectric constant of β -Ga₂O₃. The values of $N_D - N_A$ of the prepared films are 5.41×10^{15} , 2.30×10^{16} , 2.01×10^{17} , 6.65×10^{18} , 1.17×10^{19} , 1.64×10^{20} , and $1.74 \times 10^{20} \text{ cm}^{-3}$, corresponding to the samples B₁, B₂, B₃, B₄, B₅, B₆ and B₇, respectively. The $1/C^2-V$ plots of the lightly doped films are not shown here. The small changes of the $N_D - N_A$ between the films grown with 10 and 20 sccm SiH₄ further confirm the excessive Si-doping in B₇. Hall measurement was also used to acquire the effective carrier concentration of the epitaxial films. A high electron mobility value of 51 cm²/(V·s) at room temperature was achieved for sample B₅ with a carrier concentration of $7.19 \times 10^{18} \text{ cm}^{-3}$. And a high activation efficiency of about 61.5% is obtained for sample B₅ meanwhile. Fig. 6 compares the concentrations extracted from C-V, SIMS, and Hall measurements. The extracted curves have a similar trend, which means the efficiency of Si incorporation gets smaller when the SiH₄ flow rate increases.

Current density-voltage ($J-V$) characteristics of the epitaxial β -Ga₂O₃ films were measured. Fig. 7(a) shows the $J-V$ characteristics acquired on transmission line model (TLM)^[45] patterns of the epitaxial β -Ga₂O₃ films with different Si-doping concentrations in a linear plot. It is clear that the grown films with high doping exhibit good Ohmic contacts. With the proper increase of the flow rate, the resistivity of the film decreases, and B₆ gets the minimum. The significant increase

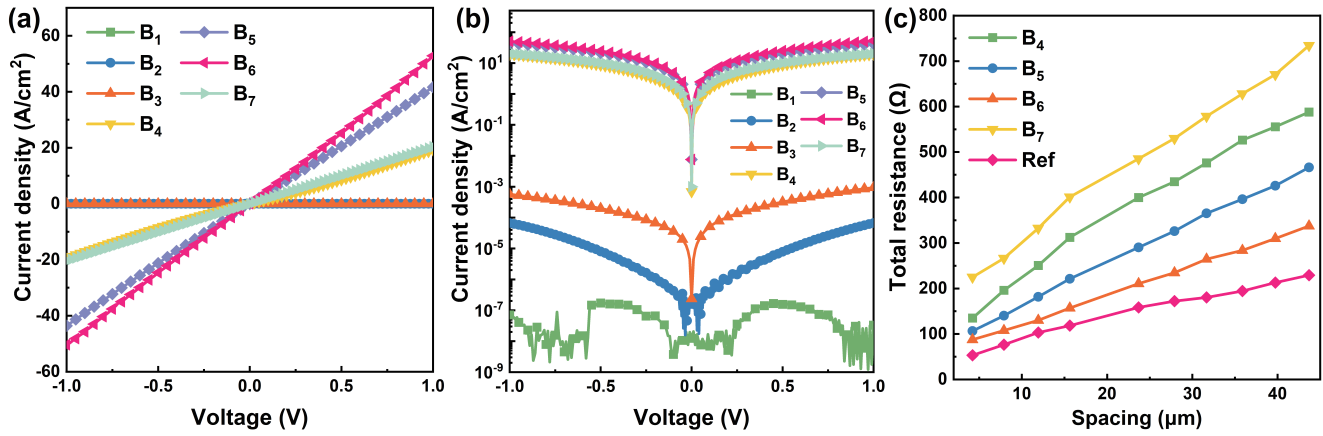


Fig. 7. (Color online) J - V characteristics of the β -Ga₂O₃ films acquired on TLM patterns in (a) a linear plot and (b) a semi-log plot. (c) Linear fit of total resistance as a function of pad spacing.

Table 1. Summary of the electronic characteristics extracted from the TLM measurements corresponding to different contact lengths on the basis of linear TLM theory.

Sample number	R_c (Ω -mm)	R_{SH} ($\Omega/\square$)	L_T (μ m)	ρ_c (10^{-4} Ω -cm ²)
B ₄	7.67	2466.93	3.11	2.38
B ₅	6.90	1324.57	5.21	3.59
B ₆	3.73	1084.55	3.44	1.29
B ₇	11.18	1929.83	5.79	6.48
Ref	2.81	751.48	3.74	1.05

in resistivity of B₇ compared to B₅ is due to the deteriorating film quality caused by excessive doping. Fig. 7(b) shows the J - V characteristics of the epitaxial β -Ga₂O₃ films in a semi-log plot. The current density of the high Si-doped film is greater than 10 A/cm². The relationship between the total resistance (R_T) and the pad spacing determined from the J - V curves of samples with different Si doping amounts is shown in Fig. 7(c), and the data were fitted linearly. Based on the TLM measurements, the contact resistivities, the sheet resistances, the transfer lengths, and the specific contact resistances are extracted, as presented in Table 1. From the fits, the $R_c = 3.73$ Ω , $L_T = 3.44$ μ m and $R_{SH} = 1084.55$ $\Omega/\square$ of sample B₆ were extracted. The specific contact resistance $\rho_c = 1.29 \times 10^{-4}$ Ω -cm² is obtained by this analysis. These excellent electrical conductivities are comparable to the sample Ref in the table, which is the Si-implanted film with a concentration of 5.0×10^{19} cm⁻³, confirming the effective Si doping in the MOCVD epitaxy. The low specific contact resistance and high current density indicate that apart from ion implantation, Si-doped epitaxy can also achieve excellent Ohmic contacts.

4. Conclusion

Homoeptaxial growth of (100) β -Ga₂O₃ films and Si doping with different concentration through MOCVD were studied in this work. By adjusting the VI/III ratio, chamber temperature and pressure, the diffusion length of Ga adsorption atoms was increased, inhibiting the 2D island growth mode and resulting in smooth surface morphology. The gradient Si-doping films indicate that the introduction of Si atoms facilitates the growth kinetics and promotes the coalescence of original 2D islands, which is beneficial to the formation of a smooth surface. Apart from the over-doped sample, most of

the epitaxial β -Ga₂O₃ films with Si-doping exhibit good crystal quality, with (400) FWHM less than 60 arc sec. Based on the C - V characteristics, an effective net doping concentration from 5.41×10^{15} to 1.74×10^{20} cm⁻³ was realized. And a high Hall mobility of 51 cm²/(V·s) was also achieved for the sample with a carrier concentration of 7.19×10^{18} cm⁻³ and a high activation efficiency of about 61.5%. The epitaxial film demonstrates a low specific contact resistance of 1.29×10^{-4} Ω -cm², which is comparable to the Si-implanted film, indicating the effective Si doping in the MOCVD epitaxy of β -Ga₂O₃ films can meet the demand of both a high concentration doping and high crystal quality, which provides a solution for the future development of β -Ga₂O₃ power devices apart from Si-ion implantation.

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Wenbo Tang is a PhD student at University of Science and Technology of China and Suzhou Institute of Nano-Tech and Nano-Bionics of Chinese Academy of Sciences under the supervision of Prof. Baoshun Zhang. His research focuses on the homoepitaxy and power electronics of β -Ga₂O₃.



Hongji Qi received his BS degree from Zhengzhou University in 2000 and his PhD degree from Shanghai Institute of Optics and Fine Mechanics, Chinese Academy of Sciences in 2005. After that, he stayed at the institute as an assistant researcher and became a full researcher in 2011. His research interests include nonlinear crystal materials, novel semiconductor materials and scintillation crystal materials.



Baoshun Zhang received his BS degree from Changchun University of Science and Technology in 1994 and PhD degree from the Institute of Semiconductors, Chinese Academy of Sciences in 2003. Then he joined in Hong Kong University of Science and Technology. Currently, he is a researcher at Suzhou Institute of Nano-Tech and Nano-Bionics, Chinese Academy of Sciences, and his research interests include semiconductor material growth and device technology research.