

Optical and electrical properties of Sb-doped β -Ga₂O₃ crystals grown by OFZ method

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Sb-doped β -Ga₂O₃ crystals were grown using the optical floating zone (OFZ) method. X-ray diffraction data and X-ray rocking curves were obtained, and the results revealed that the Sb-doped single crystals were of high quality. Raman spectra revealed that Sb substituted Ga mainly in the octahedral lattice. The carrier concentration of the Sb-doped single crystals increased from 9.55×10^{16} to 8.10×10^{18} cm⁻³, the electronic mobility depicted a decreasing trend from 153.1 to 108.7 cm² · V⁻¹ · s⁻¹, and the electrical resistivity varied from 0.603 to 0.017 Ω · cm with the increasing Sb doping concentration. The un-doped and Sb-doped β -Ga₂O₃ crystals exhibited good light transmittance in the visible region; however, the evident decrease in the infrared region was caused by increase in the carrier concentration. The Sb-doped β -Ga₂O₃ single crystals had high transmittance in the UV region as well, and the cutoff edge appeared at 258 nm.

Keywords: Sb-doped β -Ga₂O₃; crystal growth; optical properties; electrical properties.

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1. Introduction

In recent years, the β -Ga₂O₃ single crystal has attracted great interest in research due to its excellent optical and electrical properties^[1-3]. Properties of the β -Ga₂O₃ single crystal such as its huge bandgap (4.8 eV)^[4], high-breakdown electric field (8 MV/cm)^[5], and short absorption edge^[6] make them appropriate materials for deep ultraviolet (UV) electronic devices and high power, high voltage, and low loss power devices^[7-13]. In particular, β -Ga₂O₃ single crystals can be obtained by the melt method, which includes the edge-defined film-fed growth^[14], optical floating zone (OFZ)^[15], Czochralski^[16], and vertical Bridgman^[17] methods. Therefore, larger-size β -Ga₂O₃ single crystals can be obtained at a smaller cost than that of SiC and GaN crystals, which is beneficial for its large-scale applications.

β -Ga₂O₃ has a monoclinic structure with C2/m space group, and its structure is composed of deformed [GaO₄] tetrahedrons and [GaO₆] octahedrons^[18]. The [GaO₄] tetrahedrons and [GaO₆] octahedrons form a carrier chain, and the carriers can move freely^[19]. Therefore, its crystal cell contains two different Ga³⁺ sites: tetrahedral and octahedral. Electrical conductivity is the most important property to affect the devices applications of

β -Ga₂O₃ single crystals; however, the conductivity of the intrinsic β -Ga₂O₃ crystal is insufficient to achieve the requirements of device applications^[20]. Hence, it is urgent to search suitable doping elements for β -Ga₂O₃ single crystals to obtain adjustable conductivity. P-doped β -Ga₂O₃ crystals are difficult to fabricate and several researches have revealed the electrical properties of n-doped β -Ga₂O₃. For instance, group IV elements (Sn, Si, and Ge) served as suitable n-type dopants for β -Ga₂O₃ single crystals, and the carrier concentration in β -Ga₂O₃ can range from 2.5×10^{16} to 10^{19} cm⁻³^[21-24]. Our group has also reported that group V elements (Nb and Ta) served as suitable n-type dopants for β -Ga₂O₃ crystals, and the carrier concentration in the crystals reached as high as 3.0×10^{19} cm⁻³^[5,15]. Many literatures have reported that doped β -Ga₂O₃ epitaxial layers also have a good effect on improving the conductivity. Fikadu *et al.* presented that Ge-doped β -Ga₂O₃ epitaxial film was prepared by the metal organic chemical vapor deposition (MOCVD) method, and the carrier concentration can range from 2×10^{16} to 3×10^{20} cm⁻³^[25]. Akhil *et al.* reported that Sn-doped β -Ga₂O₃ epitaxial film was prepared by the molecular beam epitaxy (MBE) method, and the carrier concentration can range from 7×10^{16} to 2×10^{19} cm⁻³^[26].

It has been reported that the element Sb is often used as an effective dopant to improve the conductivity of semiconductor crystals^[27,28]. The Sb^{5+} ion not only has a similar radius (Sb^{5+} radius is 0.60 Å, and the six-coordination radius of Ga^{3+} is 0.62 Å), but also has more valence electrons than Ga^{3+} ^[29]. Thus, Sb^{5+} ions can substitute Ga^{3+} ions without causing new large-scale structural defects and supply extra electrons on the $\beta\text{-Ga}_2\text{O}_3$ lattice. These are the huge advantages of Sb^{5+} as a dopant. Hence, doping $\beta\text{-Ga}_2\text{O}_3$ with Sb^{5+} can improve the former's electrical conductivity. Therefore, Sb can be considered excellent effective dopant $\beta\text{-Ga}_2\text{O}_3$ crystals to improve the conductivity. There has been no research of Sb-doped $\beta\text{-Ga}_2\text{O}_3$ before. In this Letter, $\beta\text{-Ga}_2\text{O}_3$ single crystals doped with different Sb concentrations were grown by the OFZ method. The optical and electrical properties of the Sb-doped $\beta\text{-Ga}_2\text{O}_3$ were investigated. Our findings can provide new potential material for electronic devices based on $\beta\text{-Ga}_2\text{O}_3$ single crystals.

2. Experimental Procedure

Un-doped and Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystals were grown by the OFZ method using a Quantum Design IRF01-001-00 infrared (IR) image furnace (Made in Japan). High-purity powders of Ga_2O_3 (6N) and Sb_2O_5 (4N) were used as the raw materials. The raw materials were weighed precisely and mixed adequately, after which the mixture was pressed into rods. Then, the rods were sintered thoroughly at 1400°C for 10 h, and the (010) oriented $\beta\text{-Ga}_2\text{O}_3$ single crystals were employed as the seeds. Dry air at atmospheric pressure was used as the growth atmosphere, and the pull-down rate was 6 mm/h. The grown crystals were cut into the size of 6 mm × 8 mm × 1 mm and the 6 mm × 8 mm plane was parallel to the (100) plane, and both faces were polished into 0.5 mm thick wafers to prepare for testing optical and electrical properties.

The crystal structures were determined by X-ray diffraction (XRD) with a Rigaku D/max 2550. The X-ray rocking curve of the Sb-doped $\beta\text{-Ga}_2\text{O}_3$ was obtained via a Bruker D8 Discover X-ray diffractometer. The actual concentrations of Sb in the Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystals were characterized using a PerkinElmer Plasma-400 inductively coupled plasma atomic emission spectrometer (ICP-AES). The Raman spectra of the Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystals were obtained with a 633 nm laser beam as the excitation source (Horiba iHR550). The electrical properties were characterized at room temperature using the van der Pauw method. The room temperature transmittance spectra of the crystals were obtained by a Lambda 1050+ UV/visible (vis)/near-IR (NIR) spectrometer (PerkinElmer).

3. Results and Discussion

Figure 1 shows the photos of the as grown Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystals. The single crystals are 40–50 mm in length and approximately 6 mm in diameter. The as grown $\beta\text{-Ga}_2\text{O}_3$ crystal doped with 0.1% (molar fraction) Sb showed a light blue color, which deepened with the increase of Sb concentration; the crystal

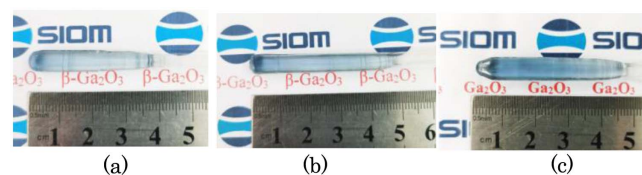


Fig. 1. Photos of the as grown Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystals. (a) 0.1% Sb; (b) 1.0% Sb; (c) 2.0% Sb.

doped with 2.0% Sb showed a dark blue color. It has been reported that the phenomenon of darkening of the color is due to infrared absorption by free carriers^[30].

Figure 2 shows the XRD patterns of the database (JCPDF: 41-1103) and Sb-doped $\beta\text{-Ga}_2\text{O}_3$ single crystals. Compared with the measured diffraction peaks of JCPDF (41-1103), the pattern showed a perfect match without any other diffraction peaks, indicating that the crystal structure did not change after Sb-doping and that the Sb-doped single crystals still belonged to the $C2/m$ space group. The intensity of several XRD peaks was not coincident with the PDF (41-1103), which is due to the residual microcrystals changing the intensity after the crystals were ground into powder. Figure 3 shows the X-ray rocking curve for the (400) plane of 1.0% Sb-doped $\beta\text{-Ga}_2\text{O}_3$ crystal. The full width at half-maximum (FWHM) value was 101 arcsec, which meant that Sb-doped $\beta\text{-Ga}_2\text{O}_3$ single crystals grown by the OFZ method were of good quality.

The actual concentration of Sb in the Sb-doped crystals and in the polycrystalline rods, tested using ICP-AES, is listed in Table 1. Evidently, the concentration of Sb in the crystals and in the polycrystalline rods was much lower than that in the raw materials. Sb_2O_5 has a low melting point, and it evaporates easily at high temperature. So, most of Sb has been volatilized during the preparation of polycrystalline rods by high-temperature sintering. The effective segregation coefficient for the three samples was about 0.71–0.77. This is due to the impurity segregation throughout the growth process.

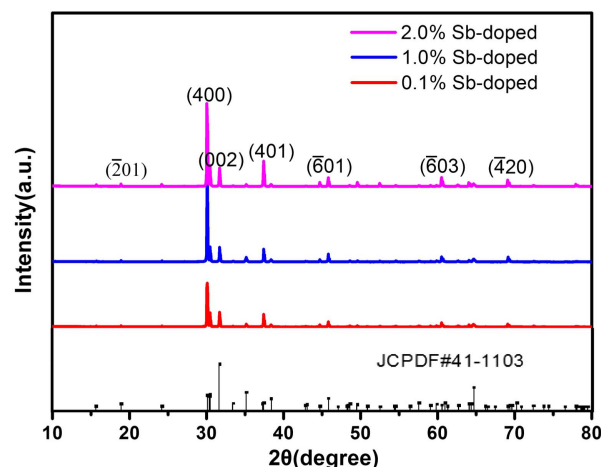


Fig. 2. XRD patterns of the database (PDF: 41-1103) and Sb-doped $\beta\text{-Ga}_2\text{O}_3$ single crystals.

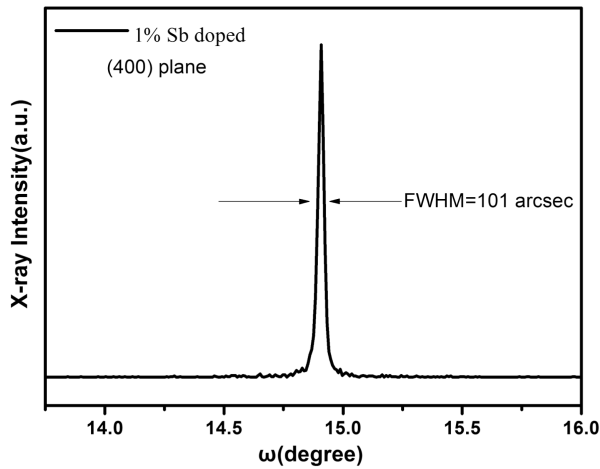


Fig. 3. X-ray rocking curve for the (400) plane of 1.0% Sb-doped β -Ga₂O₃ single crystal.

Table 1. ICP-AES Data of Sb-Doped β -Ga₂O₃ Crystals with Different Sb Doping Concentrations.

	Sample 1	Sample 2	Sample 3
Nominal composition Sb (%)	0.1	1.0	2.0
Rod [ICP-AES (%)]	0.0142	0.0206	0.0309
Crystal [ICP-AES (%)]	0.0101	0.0157	0.0237
Effective segregation coefficient	0.71	0.76	0.77

Room temperature Raman spectra of Sb-doped β -Ga₂O₃ crystals between 100 and 1000 cm⁻¹ are shown in Fig. 4. According to the previous reports^[19,31], the Raman active modes of β -Ga₂O₃ could be divided into three parts: (I), (II), and (III). (I) is attributed to the vibration and translation of the

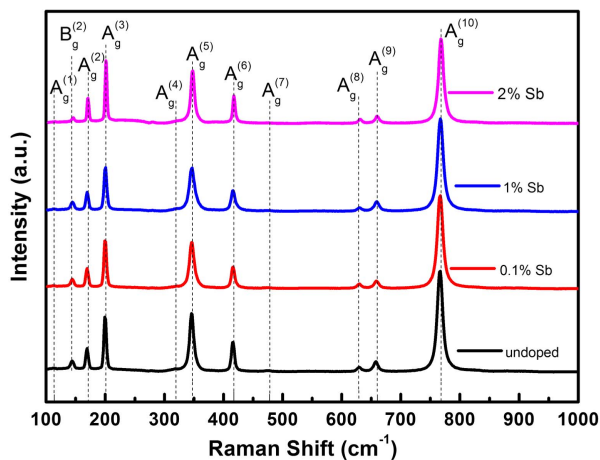


Fig. 4. Room temperature Raman spectra of Sb-doped β -Ga₂O₃ single crystals.

tetrahedrons–octahedrons chains and appears below 300 cm⁻¹; (II) is associated with the deformation of [GaO₆] octahedrons and appears between 300 and 600 cm⁻¹; (III) is due to the stretching and bending of [GaO₄] tetrahedrons and appears above 600 cm⁻¹. Eleven main Raman peaks of Sb-doped crystals were observed. A_g(1):114cm⁻¹, B_g(2):144cm⁻¹, A_g(2):169 cm⁻¹, A_g(3):200 cm⁻¹, A_g(4):317 cm⁻¹, A_g(5):344 cm⁻¹, A_g(6):416 cm⁻¹, A_g(7):472 cm⁻¹, A_g(8):629 cm⁻¹, A_g(9):654 cm⁻¹, and A_g(10):767 cm⁻¹ were observed, respectively. As shown in Fig. 5, the intensity of part (II) peaks decreases with the increasing of Sb concentration. Based on the analysis of Raman spectra and ion radius, it indicates that Sb⁵⁺ substitutes Ga³⁺ mainly at the octahedral sites.

Room temperature electrical properties of the un-doped and Sb-doped β -Ga₂O₃ single crystals are listed in Table 2. Ohmic contacts were obtained by sputtering 10 nm Ti/90 nm Al layer on the four corner surfaces of the wafers. All crystals revealed n-type conduction, and the carrier concentration of the un-doped β -Ga₂O₃ single crystal was 9.55×10^{16} cm⁻³ due to the raw materials impurities, which was similar to the previous report^[5]. The carrier concentration in the crystal was found to increase with Sb concentration by approximately two orders of magnitude, from 9.55×10^{16} cm⁻³ to 8.10×10^{18} cm⁻³. The carrier concentration of 0.1% Sb-doped β -Ga₂O₃ and 1% Sb-doped β -Ga₂O₃ crystals is smaller than the Sb concentration in the crystals, while the carrier concentration (8.10×10^{18} cm⁻³) of 2% Sb-doped β -Ga₂O₃ is close to the Sb concentration in the crystal (0.0237% is equivalent to 8.96×10^{18} cm⁻³). As a double electron donor, the activation efficiency of Sb⁵⁺ is much lower than 200%, even in 2% Sb-doped β -Ga₂O₃ crystals. The electronic mobility decreased with Sb concentration, from 153.1 to 108.7 cm² · V⁻¹ · s⁻¹. The value of the electrical resistivity in the β -Ga₂O₃ crystals changed from 0.603 to 0.017 Ω · cm. Based on the above results, Sb can be considered a suitable n-type dopant for β -Ga₂O₃ crystals to improve its conductivity.

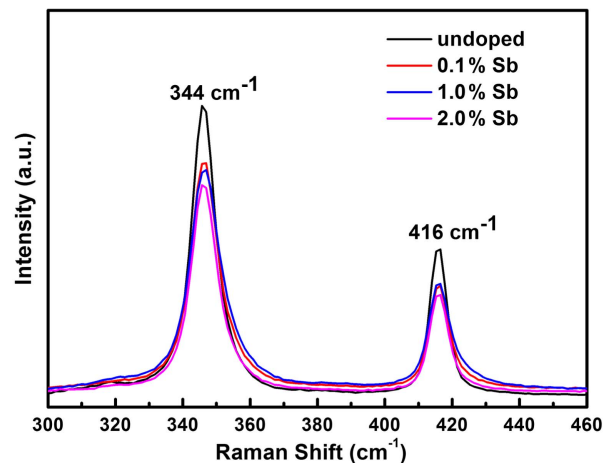
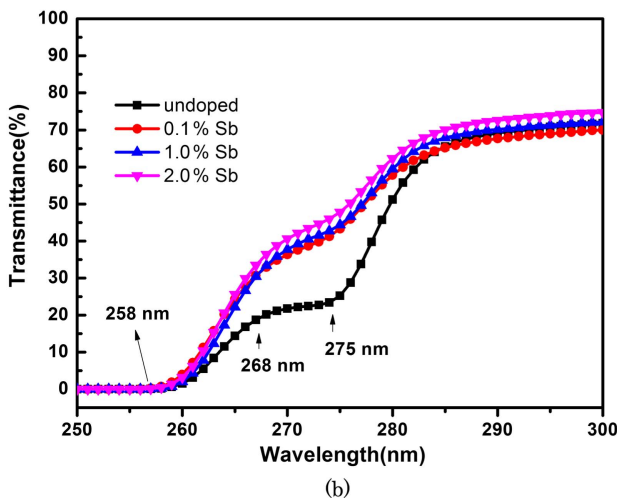
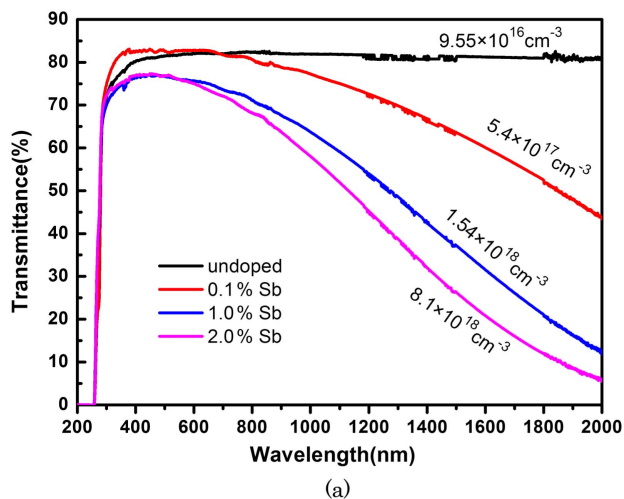


Fig. 5. Room temperature Raman spectra of Sb-doped β -Ga₂O₃ single crystals in the 300–460 cm⁻¹ wavenumber range.

Table 2. Room-Temperature Hall Data of the Un-Doped and Sb-Doped β -Ga₂O₃ Crystals.

Samples	Carrier Type	Carrier		
		Concentration (cm ⁻³)	Mobility (cm ² · V ⁻¹ · s ⁻¹)	Resistivity (Ω · cm)
β -Ga ₂ O ₃	n	9.55×10^{16}	153.1	0.603
β -Ga ₂ O ₃ :Sb (0.1%)	n	5.40×10^{17}	134.7	0.064
β -Ga ₂ O ₃ :Sb (1%)	n	1.54×10^{18}	126.4	0.037
β -Ga ₂ O ₃ :Sb (2%)	n	8.10×10^{18}	108.7	0.017

Figure 6 shows the room temperature optical transmittance spectra of Sb-doped β -Ga₂O₃ crystals with different Sb concentrations. Figure 6(a) shows that the transmittance of the crystals

**Fig. 6.** Optical transmittance of un-doped and Sb-doped β -Ga₂O₃ crystals. (a) 200–2000 nm (including their carrier concentrations), (b) 250–300 nm.

in the range of visible wavelength is between 70% and 80%, which indicates that the Sb-doped β -Ga₂O₃ crystals exhibit good light transmittance in the visible region. The obvious decrease in transmittance of the Sb-doped crystals in the IR region was caused by the increase in carrier concentration. According to the previous report^[19], IR absorption is associated with plasma frequency, which is determined by conductive electrons. Sb-doped β -Ga₂O₃ had good transmittance in the UV region as well, and the cutoff absorption edge appeared at 258 nm, as shown in Fig. 6(b). The transmission spectra in the UV range had a shoulder from 268 nm to 275 nm as a result of the transition from the valence band perturbed by Ga³⁺ vacancies to the conduction band^[32]. The shoulder tended to become smooth with the increase of Sb concentration, which indicated that Sb⁵⁺ ions substituted Ga³⁺ vacancies, the reduction of which reduced the disturbance.

4. Conclusion

In summary, Sb-doped β -Ga₂O₃ single crystals were successfully obtained for the first time, to the best of our knowledge, by the OFZ method. XRD showed that the crystal structure did not change after doping, and the Sb-doped single crystals still belonged to the C2/m space group. Room temperature Raman spectra revealed that Sb⁵⁺ substituted Ga³⁺ ions mainly in the octahedral site. The carrier concentration of the Sb-doped single crystals increased from 9.55×10^{16} to 8.10×10^{18} cm⁻³; the electronic mobility decreased from 153.1 to 108.7 cm² · V⁻¹ · s⁻¹, and the electrical resistivity varied from 0.603 to 0.017 Ω · cm with the increasing Sb doping concentration. Sb⁵⁺ can be considered as a suitable n-type dopant for doping to improve the conductivity of the β -Ga₂O₃ crystals. The transmittance diminished in the NIR range with the increase in Sb concentration, which could be attributed to the increase in the carrier concentration. Our study can promote further research in the field of devices based on β -Ga₂O₃ crystals.

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