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Single Crystal β-Ga_2O_3 Substrates

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Abstract

Single crystal β-Ga_2O_3 substrates containing no twin boundaries with sizes up to 4 inches in diameter were fabricated. Intentional n-type doping was shown to be possible by using Si or Sn as a dopant. An etch pit observation revealed that the dislocation density was on the order of 10^3 cm^{-3}.

1. Introduction

Gallium oxide (Ga_2O_3) is a semiconductor material that has both a large band gap energy and electrical conductivity. It has been attracting attention because it has high potential for power device applications[1-4)] and high-brightness LED applications[5)]. β-Ga_2O_3 can be grown from a melt source; therefore, its growth rate is high. This means it has a lower production cost compared with other wide band gap semiconductor materials such as silicon carbide, gallium nitride, aluminum nitride, and diamond, whose growth rates are relatively low because they can be grown from only diluted vapor sources.

In this presentation, we report on single crystal β-Ga_2O_3 substrates made from large and high-quality bulk crystals grown with an edge-defined film-fed growth (EFG) process[6)].

2. Experimental methods

β-Ga_2O_3 bulk crystals were grown using the EFG process. A schematic drawing of this process is shown in Fig 1. 5N-grade Ga_2O_3 powder was used as the source material. The source powder was put in a crucible made of Ir together with an iridium die. The growth pressure was atmospheric pressure, and the growth atmosphere was a mixture of 98% nitrogen and 2% oxygen gas. The growth rate was adjusted to 15 mm/h.

The EFG-grown β-Ga_2O_3 bulk crystals were processed into forms of semiconductor substrates. The crystals were annealed in a nitrogen ambient at 1450°C for 6 hours to reduce residual stress in the crystal and to achieve full activation of donors before the substrate fabrication process.

3. Results and discussion

The EFG-grown β-Ga_2O_3 bulk crystal was cut perpendicular to the [010] direction and was shaped into a flat hexagonal cylinder with a length of 55 mm, width of 60 mm, and thickness of 18 mm. Figure 2 shows a picture of the

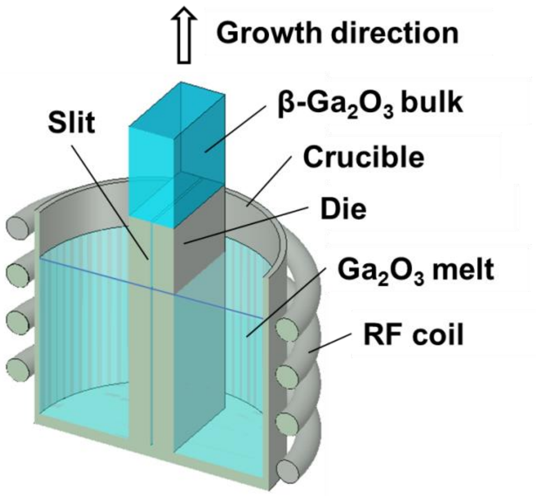

Fig. 1 Schematic drawing of EFG process. Ga_2O_3 melt goes up to top of die through slit by capillary action. Crystal growth occurs on top of die.

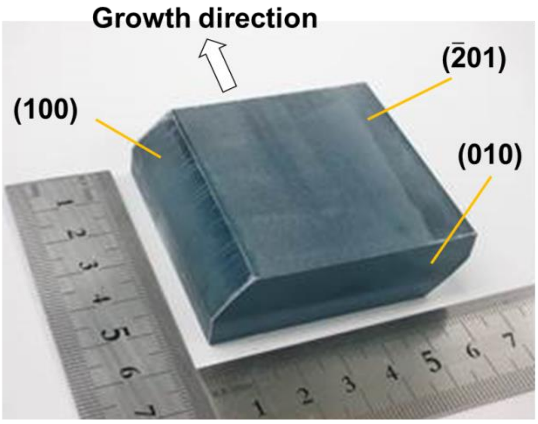

Fig. 2 Photograph of EFG-grown β-Ga_2O_3 bulk crystal. Sample was cut perpendicular to [010] direction and shaped into flat hexagonal cylinder with length of 60 mm, width of 60 mm, and thickness of 18 mm. Principal plane was ($\bar{2}01$), and cross section was (010). (100) planes appeared as facets formed during growth.

samples after cutting. The principal plane was $(\bar{2}01)$, and the cross section was (010). The (100) planes appeared as facets formed during growth.

Figure 3 shows pictures of the processed β-Ga_2O_3 substrates. The substrates were 10 × 15 mm (10 × 15), 2 inches in diameter, and 4 inches in diameter. The surface orientation was $(\bar{2}01)$, (010), or (001) for the 10 × 15 substrates but only $(\bar{2}01)$ for 2-inch and 4-inch substrates. It should be emphasized that the crystals did not contain any twin boundaries.

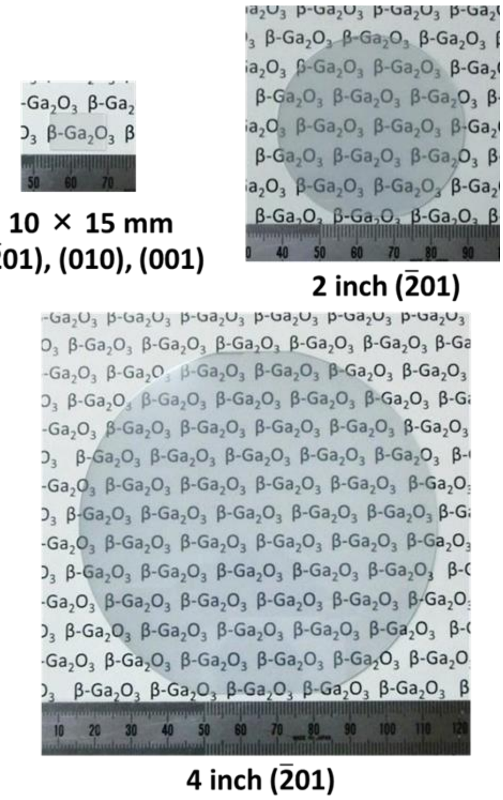

Fig. 3 Photographs of processed β-Ga_2O_3 substrates. Substrates were 10 × 15 mm, 2 inches in diameter, and 4 inches in diameter. Surface orientation was $(\bar{2}01)$, (010), or (001) for 10 × 15 substrates but only $(\bar{2}01)$ for 2-inch and 4-inch substrates.

We examined what residual impurity elements were included in the Ga_2O_3 source powder and the EFG-grown crystals by taking GDMS and SIMS measurements. The major residual impurity elements unintentionally contained in the crystal were found to be Si and Ir. Since Si was the element with the second largest concentration in the source power, it is estimated that the Si came from the source powder. Meanwhile, the Ir probably came from the crucible as it was made of Ir.

Next, we investigated intentional n-type doping by using SnO_2 and SiO_2 in the EFG process. N_d-N_a could be controlled in a range between 10^{17} and 10^{19} cm^{-3} by changing the dopant content. For Sn, the upper limit of N_d-N_a was about 2×10^{19} cm^{-3}. N_d-N_a saturated and did not exceed the upper limit when the SnO_2 content was further increased. A possible origin of the saturation phenomena is the high vapor pressure of the SnO_2. SnO_2 has a high vapor pressure at the melting point of β-Ga_2O_3, and this prevents the Sn concentration from being high. Meanwhile, no apparent upper limit was observed in the Si doping, although the quality of the grown crystal deteriorated rapidly when the Si concentration exceeded 1×10^{19} cm^{-3}. The grown crystals with high Si concentrations had voids and cracks. The lower limit of N_d-N_a was about 2×10^{17} cm^{-3}. The lower limit was simply governed by the residual Si concentration, which was determined by the purity of the source material.

The dislocation density was estimated by investigating the density of etch pits. Three samples were prepared: a 2-inch $(\bar{2}01)$ substrate, a 10 × 15 (010) substrate, and a 10 × 15 (001) substrate. The region of the 10-μm depth under the sample surface was first removed by ICPRIE using boron tri-chloride to eliminate the influence of the damaged layer generated by polishing. Then, the substrates were dipped in H_3PO_4 heated at 130°C for two hours to form etch pits. We observed two kinds of pits: small ones and line-shaped ones. Most of the pits were small. The density of the small pits was between 1×10^3 and 1×10^4 cm^{-2}. The TEM observation clarified that dislocations existed under the small pits. The number density of the line-shaped pits was one order of magnitude smaller than that of the small pits. The origin of the line-shaped pits has not been clarified yet. For (010) substrates, two types of etch pit were observed with a pit density of major and minor types on the order of 10^3 and 10^2 cm^{-2}, respectively. For (001) substrates, only one type of etch pit was observed with a pit density on the order of 10^3 cm^{-2}. From these results, the dislocation density in the bulk β-Ga_2O_3 crystal was estimated to be on the order of 10^3 cm^{-2}.

4. Conclusion

Single crystal β-Ga_2O_3 substrates containing no twin boundaries with sizes up to 4 inches in diameter were fabricated. Residual impurities, doping controllability, and crystal defects were investigated, and the results show that β-Ga_2O_3 single substrates can be made with high enough quality for semiconductor device applications.

Acknowledgements

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