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Fabrication and Testing of Substrates Made from Bulk Gallium Oxide Crystals by the Cleavage Method

D.A. Bauman[1,*], D.I. Panov[1], V.A. Spiridonov[1], V.V. Lundin[2], S.N. Rodin[2], N.D. Prasolov[2], A.L. Kolesnikova[1,3]

[1] Institute of Advanced Data Transfer Systems, ITMO University, Kronverkskiy pr., 49, lit. A, St. Petersburg, 197101, Russia
[2] Ioffe Physical-Technical Institute, Russian Academy of Sciences, St. Petersburg, 194021, Russia
[3] Institute for Problems in Mechanical Engineering, V.O., Bolshoj pr., 61, Russian Academy of Sciences, St. Petersburg, 199178, Russia

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Abstract

The paper proposes a method for fabricating gallium oxide substrates from bulk β-Ga_2O_3 crystals by the cleavage method. Layers of β-Ga_2O_3, β-$(Al_xGa_{1-x})_2O_3$ and structures of β-Ga_2O_3/β-$(Al_xGa_{1-x})_2O_3$ are grown on the prepared substrates by the MOCVD method. The surface morphology of the layers and growth regimes are analyzed. The fundamental possibility of using gallium oxide substrates obtained by the cleavage method for the subsequent epitaxy is shown.

Keywords: Gallium oxide; Bulk crystal; Substrates; MOCVD; Surface morphology

1. INTRODUCTION

Over the past ten years gallium oxide has attracted the attention of researchers as a new wide-gap semiconductor, significantly exceeding the band gap (E_g = 4.85 eV for β-Ga_2O_3 [1], E_g = 5.3 eV for α-Ga_2O_3 [2]) of such materials as Si, SiC, GaN (E_g = 1.12, 3.3 and 3.4 eV, respectively), which have become traditional for the modern semiconductor industry. Of the semiconductors used today in epitaxial technologies, only AlN and AlGaN have larger band width (E_g = 6.2 eV), and among bulk materials, AlN and diamond have E_g = 5.5 eV. The most stable β form of gallium oxide also has other advantages that are important for power and optoelectronics: high breakdown voltage (V_{br} = 8 MV/cm [3]), and significant hardness [4]. Today, methods for creating gallium oxide of *n*-type conductivity are well studied [5], and in recent years there have been works on *p* doping of β-Ga_2O_3 [6]. Thus, at the present time, we have come close to the use of gallium oxide in the industrial production of semiconductor devices, primarily power electronics, and solar-blind photodetectors.

For the manufacture of a high-quality electronic semiconductor device and power electronic devices in particular, where a high breakdown voltage is crucial, it is important to ensure the manufacture of low-defect epitaxial layers. The most effective way to solve this problem is to use homoepitaxy: to grow device structures on "native" substrates. And here one more advantage of gallium oxide manifests itself — the possibility of manufacturing bulk crystals by relatively cheap methods of pulling from the melt (the Czochralski, Stepanov or edged defined film fed growth, Bridgman, floating zone methods [7]).

This paper presents the results of developing a method for growing high-quality (low-defect) bulk β-Ga_2O_3 crystals, a method for preparing substrates from the obtained crystals, as well as the results of growing β-Ga_2O_3 and β-

* Corresponding author: D.A. Bauman, e-mail: dabauman@itmo.ru

$(Al_xGa_{1-x})_2O_3$ layers on the fabricated substrates by the MOCVD method. The main goal of experiments on growing epitaxial layers in this work was to test the obtained gallium oxide substrates and to demonstrate the fundamental possibility of using substrates for subsequent epitaxy of β-Ga_2O_3 and β-$(Al_xGa_{1-x})_2O_3$ layers.

2. EXPERIMENTS

Bulk crystals of β-Ga_2O_3 were grown on an industrial installation "Nika-3" (manufactured by EZAN, Russia), designed for growing crystals by the Czochralski method. An iridium crucible was used to form the melt. The height of the crucible was 26 mm, the inner diameter was 40 mm, the wall thickness was about 2 mm. The crucible was placed in a thermal zone of zirconium dioxide doped with amorphous silicon. A water-cooled inductor was located around the zone, providing induction heating of the crucible. The seed crystal was attached to the upper rod using a special tool. Fragments of previously grown Ga_2O_3 crystals in the form of narrow strips (bars) were used as a seed. The starting material for the melt formation was Ga_2O_3 powder with a purity of 99.999%. The crystal pulling speed in all experiments was in the range of 0.1 to 0.2 mm/min, and the rotation speed was in the range of 5 to 10 rpm.

The standard approach to the manufacture of substrates from bulk crystalline material (boules) requires cutting, subsequent mechanical or chemical-mechanical polishing and complex cleaning. However, in the case of a β-Ga_2O_3 single crystal, another method for fabricating substrates is available [8,9]. This method uses the nature of the cleavage of β-Ga_2O_3 crystals: thin plates easily peel off from the bulk crystal along the (100) cleavage plane. Moreover, the surface of the obtained plates (substrates) is quite smooth and does not require additional machining. The procedure for making the substrate is as follows. The ends of the boule are sawn perpendicular to the direction of growth (direction [010]). At the end of the boule (on the cut), an incision is made about 0.6 mm thick and 3–5 mm long. The cut must be oriented strictly parallel to the (100) plane. The orientation is determined by the orientation of the initial seed crystal. Next, a metal wedge is inserted into the slot and pressure is applied with a slight turn of the wedge. In this way, substrates were obtained in the form of plane-parallel plates with dimensions of about 10×15 mm and a thickness of about 1 mm.

The epitaxial growth of β-Ga_2O_3 and β-$(Al_xGa_{1-x})_2O_3$ layers on the prepared substrates was performed on an Epiquip VP-50 MOCVD setup upgraded for oxide growth. This setup has a horizontal reactor with induction heating. The epitaxial growth was carried out at a temperature of 750 °C and a pressure of 100 mbar. The carrier gas flow (nitrogen) was 4.5 slm (liter per minute under standard conditions), oxygen flow was 1 slm, trimethylgallium (TMGa) and trimethylaluminum (TMAl) flows were 21 μmol/min and 6 μmol/min, respectively. The growth rate of the epitaxial layers was about 750 nm/h.

3. RESULTS AND DISCUSSION

The surface roughness of the obtained substrates was studied using atomic force microscopy (AFM). Images of the

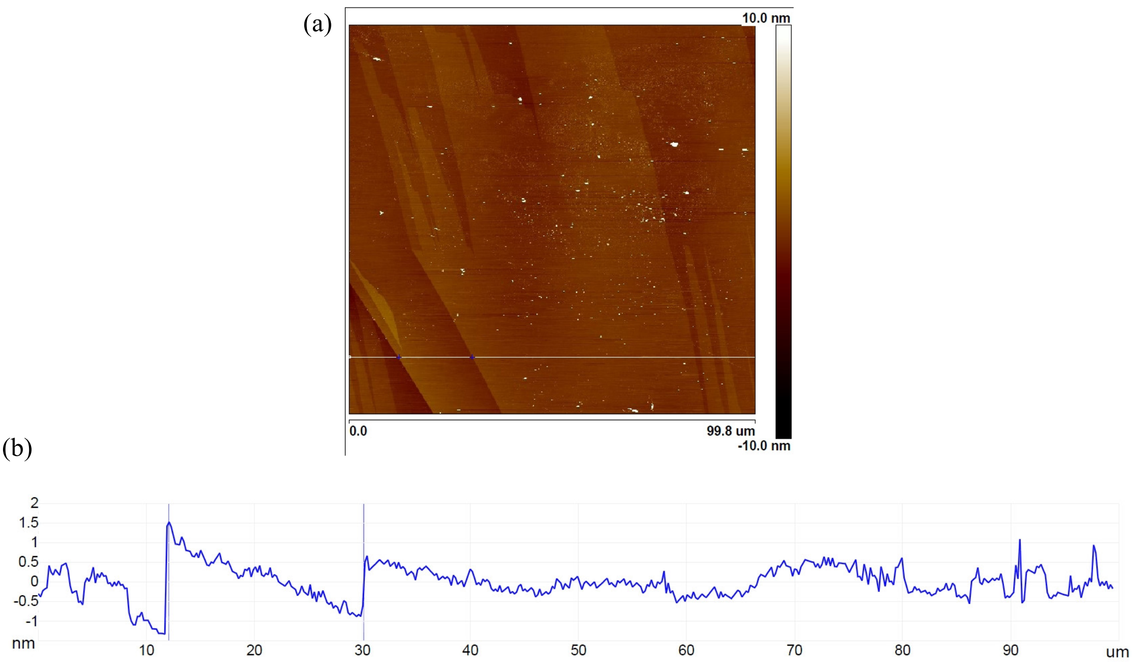

Fig. 1. AFM image of the substrate surface: a) surface map, b) roughness profile along the marked line.

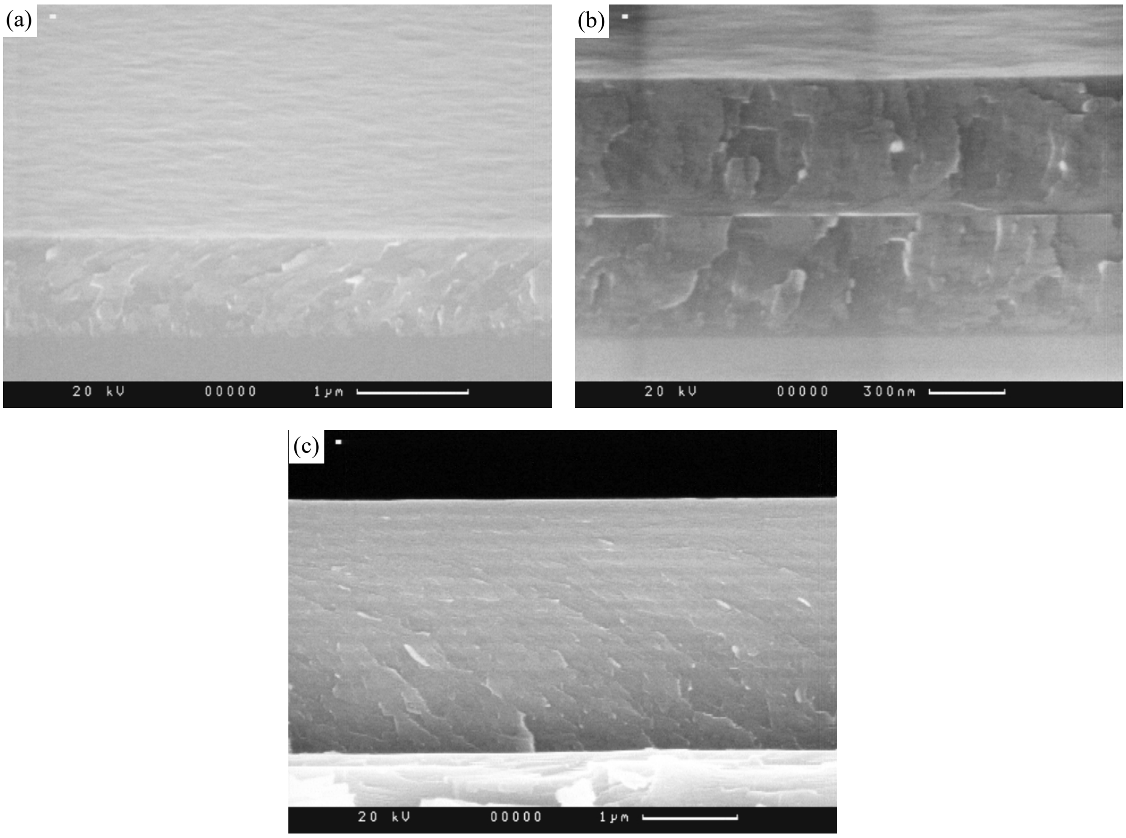

Fig. 2. Images of layers on the cleavage of the structures obtained using a scanning electron microscope: (a) β-Ga_2O_3 layer, about 0.9 μm thick, (b) β-Ga_2O_3 layers (bottom) and β-$(Al_xGa_{1-x})_2O_3$ (top), about 0.5 μm each, (c) a system of 11 pairs of β-Ga_2O_3/β-$(Al_xGa_{1-x})_2O_3$ layers, each pair is approximately 0.16 μm thick, as well as a lower β-Ga_2O_3 layer adjacent to the substrate, about 0.9 μm thick.

substrate surface obtained using the AFM facility (NT-MDT, Russia) are shown in Figure 1. The height of the steps on the surface does not exceed 3–4 nm, which is comparable with the relief of commercially produced substrates and is sufficient for the epitaxial growth process.

At the same time, the growth surface remains oriented parallel to the (100) plane, while the system of steps on the surface is not regular, as on standard substrates, which are usually misoriented by 0.2–0.3° relative to the crystal plane. This, as will be shown below, leads to an island mechanism of layer growth, instead of layer-by-layer growth of steps (the so-called step flow growth).

Layers of β-Ga_2O_3 and of β-Ga_2O_3/β-$(Al_xGa_{1-x})_2O_3$, as well as a system of eleven pairs of alternating layers of β-Ga_2O_3/β-$(Al_xGa_{1-x})_2O_3$ were grown on the obtained substrates. The aluminum content in the epitaxial layers was determined by the EDX method (Energy-dispersive X-ray spectroscopy) and amounted to 4 at.% for a single layer β-$(Al_xGa_{1-x})_2O_3$ and 2 at.% for the layer system. The images of the layers on the cleavage of the structures, obtained using a scanning electron microscope, are shown in Figure 2.

The surface morphology of the grown β-Ga_2O_3 layer is shown in Figure 3. It is clearly seen that the layer has an island surface, which is typical for layers grown on non-misoriented substrates. The surface of gallium oxide layers obtained in Ref. [10] for substrate misorientation angles less than 0.1° has a similar shape.

Thus, we can conclude that gallium oxide substrates obtained by the cleavage method make it possible to grow continuous layers of gallium oxide and a solid solution of β-$(Al_xGa_{1-x})_2O_3$ on their surface, but in the island growth mode. To implement growth in the step-flow growth mode, the substrate surface must be misoriented.

4. CONCLUSION

The paper describes the procedure for fabricating substrates from bulk gallium oxide crystals by cleavage along the (100) cleavage plane. The surface roughness of the obtained substrates and the height of the steps are quite low. This made it possible to grow layers of β-Ga_2O_3 and β-$(Al_xGa_{1-x})_2O_3$ on the obtained substrates by the MOCVD method with an aluminum content of 2 at.% up to 4 at.% As far as the authors know, this is the first experiment in Russia on the epitaxial growth of gallium oxide layers on bulk gallium oxide substrates made in Russia. However, due to the absence of misorientation of the substrates and the system of regular steps, the island growth of the layers is predominantly realized.

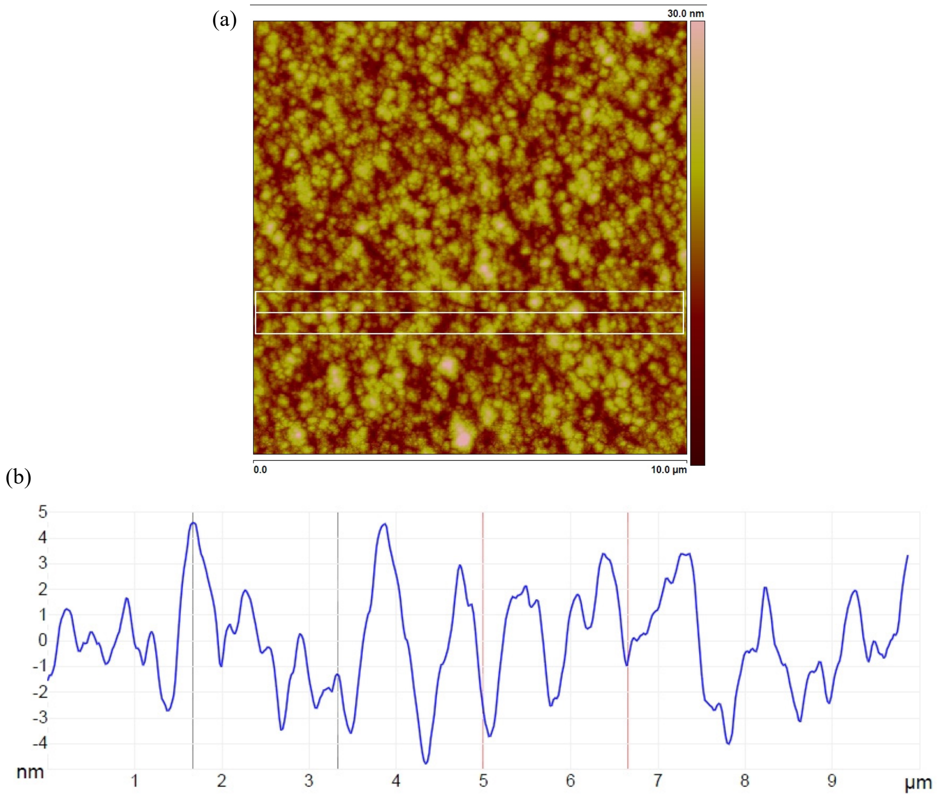

Fig. 3. AFM image of a surface fragment of an epitaxial gallium oxide layer grown on a bulk gallium oxide substrate: (a) surface map, (b) profile along the marked line.

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Изготовление и испытание подложек, получаемых из объемных кристаллов оксида галлия методом скалывания

Д.А. Бауман[1], Д.Ю. Панов[1], В.А. Спиридонов[1], В.В. Лундин[2], С.Н. Родин[2], Н.Д. Прасолов[2], А.Л. Колесникова[1,3]

[1] Институт перспективных систем передачи данных Университета ИТМО, Кронверкский пр., д. 49, лит А, Санкт-Петербург, 197101, Россия
[2] Физико-технический институт им. А.Ф. Иоффе РАН, Санкт-Петербург, 194021, Россия
[3] Институт проблем машиноведения РАН, В.О., Большой пр., д. 61, Санкт-Петербург, 199178, Россия

Аннотация. В работе предложен метод изготовления подложек оксида галлия из объёмных кристаллов β-Ga_2O_3 методом скола. На изготовленных подложках методом MOCVD выращены слои β-Ga_2O_3, β-$(Al_xGa_{1-x})_2O_3$ и структуры β-Ga_2O_3/β-$(Al_xGa_{1-x})_2O_3$. Проанализирована морфология поверхности слоёв и режимы роста. Показана принципиальная возможность использования подложек оксида галлия, полученных методом скола, для последующей эпитаксии.

Ключевые слова: оксид галлия; объёмные кристаллы; подложки; MOCVD; морфология поверхности