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Investigation of β-Ga_2O_3 thick films grown on c-plane sapphire via carbothermal reduction

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Abstract: We investigated the influence of the growth temperature, O_2 flow, molar ratio between Ga_2O_3 powder and graphite powder on the structure and morphology of the films grown on the c-plane sapphire (0001) substrates by a carbothermal reduction method. Experimental results for the heteroepitaxial growth of β-Ga_2O_3 illustrate that β-Ga_2O_3 growth by the carbothermal reduction method can be controlled. The optimal result was obtained at a growth temperature of 1050 °C. The fastest growth rate of β-Ga_2O_3 films was produced when the O_2 flow was 20 sccm. To guarantee that β-Ga_2O_3 films with both high-quality crystal and morphology properties, the ideal molar ratio between graphite powder and Ga_2O_3 powder should be set at 10 : 1.

Key words: β-Ga_2O_3 epitaxy; carbothermal reduction method; growth parameters

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1. Introduction

Gallium oxide (Ga_2O_3) has received extensive research attention recently as an ultra-wide bandgap material. Among all the five polymorphs α-, β-, ε-, δ-, γ-Ga_2O_3, β-Ga_2O_3 is the thermal stable phase[1]. Possessing numerous excellent properties including the wide band gap of 4.8–4.9 eV[2], the advanced breakdown voltage is expected to be 8 MV/cm[3], as well as high sensitivity in deep-UV[4], where β-Ga_2O_3 are widely used in many fields[5]. For instance, in the application of the Schottky barrier diode (SBD), higher breakdown voltage can be achieved by depositing high purity β-Ga_2O_3 films with low carrier density. Thus, the SBDs with higher quality can be achieved[6]. However, excellent performance power device applications require not only the quality but also the thickness of the epitaxial layer material[7]. It has been widely investigated how β-Ga_2O_3 films are grown by a variety of techniques, including molecular beam epitaxy (MBE), pulsed laser deposition (PLD), and halide vapor phase epitaxy (HVPE)[8–19] and etc. Nevertheless, these methods have some limitations. According to the numerous papers related to the epitaxy of different β-Ga_2O_3 polymorphs by the most common approaches mentioned above, the gas flows, the ratio of the raw materials used in the deposition and the growth temperature are the factors that can impact the quality of growth[20–23]. Recently, we demonstrate that the carbothermal reduction rapid growth method can be used to grow heteroepitaxial films of β-Ga_2O_3 on c-plane sapphire substrates[24]. It is a method which was widely used in synthesizing β-Ga_2O_3 nanostructure[25]. It is a simple, practical techniques for the synthesis and this method allowed for the rapid and inexpensive epitaxy[26, 27]. There are two stages in the carbothermal reduction reaction, in the first stage, suboxide Ga_2O was formed as the Ga_2O_3 powder react with graphite powder, and they further oxidation to Ga_2O_3 in the next process[28, 29]. In the second period, the Ga_2O vapor reacts further with CO to produce Ga and CO_2[24]. As the vapor pressure of Ga_2O is significantly higher than Ga[30], its desorption limits the growth rate due to the following three factors: (1) insufficient O-flux was provided to oxidize all formed suboxide[29], (2) high growth temperatures at which the thermally activated desorption of Ga_2O outperforms its oxidation even under O-rich growth conditions[2], and (3) β-Ga_2O_3 substrate orientations that provide only weak bonds to the adsorbed Ga_2O, resulting in a low activation energy for its desorption[28]. Hence, it is powerfully suggested to investigate the O_2 flux and growth temperature to enhance the first phase of the carbothermal reduction and impede the second period. In this work, we explored three main factors on the influence of high quality β-Ga_2O_3 films, including the lower part's furnace temperature, the O_2 flow, the molar ratio between Ga_2O_3 powder and graphite powder.

2. Experiment

Pure Ga_2O_3 powder with a purity level of 99.999% was mixed with graphite powder used as the precursors, meanwhile, argon was used as protective gas. The mixtures were put into a corundum crucible. The substrate was c-plane sapphire. Carbothermal reduction was the method used to deposit Ga_2O_3 films. The deposition time was 2 h and the gas pressure for the samples treated in this research was fixed at 3.0×10^3 Pa. Since the facility was a vertical dual temperature zone furnace, the diagram was shown in Fig. 1. There were two settings for the lower and upper parts of the furnace, respectively. A lower part setting temperature of the furnace was the primary factor affecting the quality of the films. The growth reaction mechanism could be explained as follows[24]:

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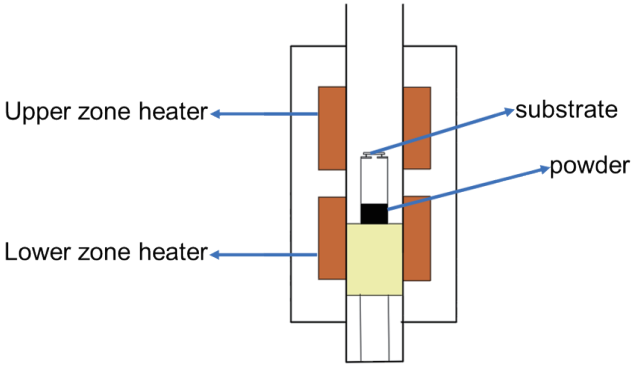

Fig. 1. (Color online) The simple diagram of the home-made vertical cylindrical dual temperature zone furnace.

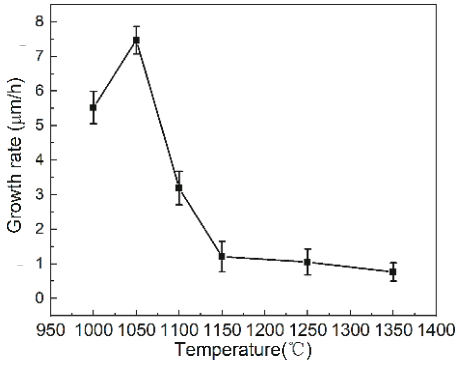

Fig. 2. The growth rate of β-Ga_2O_3 films grown at different growth temperatures ranged from 1000 to 1350 °C.

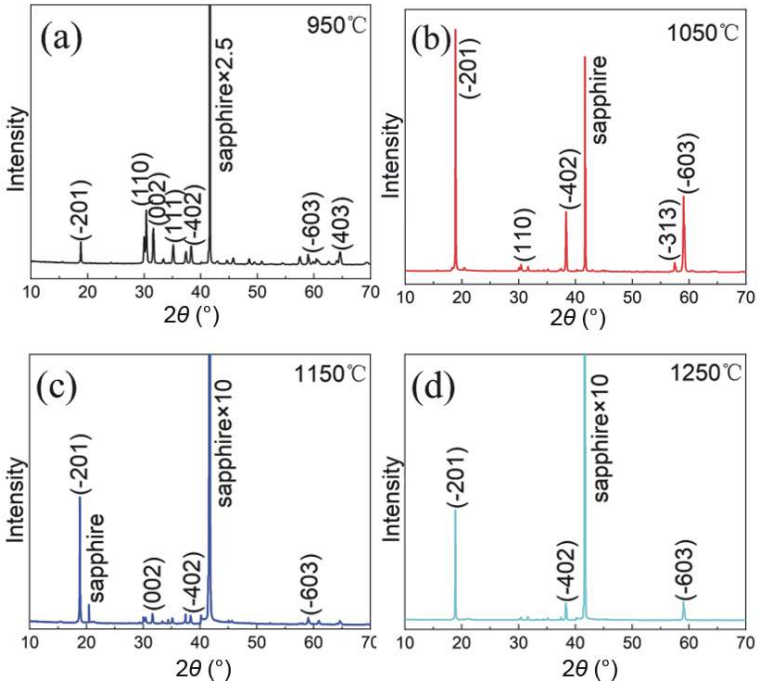

Fig. 3. (Color online) Typical XRD θ–2θ scan of the β-Ga_2O_3 films grown on c-plane sapphire substrate at different temperatures of (a) 950 °C, (b) 1050 °C, (c) 1150 °C, (d) 1250 °C.

$$2C(s) + Ga_2O_3(s) \rightarrow Ga_2O(g) + 2CO(g), \quad (1)$$

$$Ga_2O(g) + O_2(g) \rightarrow Ga_2O_3(s). \quad (2)$$

The surface morphologies of the as-grown samples were examined using scanning electron microscopy (SEM, FEI Nova Nano 450). The Ga_2O_3 thick films were investigated by X-ray diffraction (XRD, Bruker D8 Advance), and standard XRD powder profiles (θ–2θ) were measured. We present the factors that determine the crystal phases and orientations based on the experiments results.

3. Results and discussion

3.1. Effects of temperature of lower part of furnace on film

Fig. 2 displays the influence on the growth rate of the Ga_2O_3 films regarding temperatures of the lower part of the furnace. The growth rate shows a tendency of accelerated growth rate from 5.43 μm/h at 1000 °C and reaches 7.47 μm/h at 1050 °C. However, in the range of 1050 to 1150 °C, the growth rate is sharply reduced to 1.14 μm/h, equivalents to the thickness of 2.28 μm. When temperatures were set at 1150 and 1250 °C, it appears that the rate of growth of the epitaxy films remained relatively stable, measuring 1.14 and 1.006 μm/h, respectively. This phenomenon can be explained by too high temperatures leading to the desorption of Ga_2O outperforming its oxidation under O-rich growth[22].

A comparison of X-ray diffraction (XRD) patterns for films grown at different temperatures are depicted in Fig. 3. Peaks of 18.9°, 38.4°, and 59.2° correspond to the diffraction of (–201), (–402), and (–603) planes of β-Ga_2O_3, respectively[31–33]. Fig. 3(a) representing the setting temperature of the lower part of the furnace at 950 °C, no obviously dominated peaks can be seen which means the film is anisotropic growth. Meanwhile, in Fig. 3(b), the intensities of other diffraction peaks that belong to β-Ga_2O_3 are lower, which indicate that the films are pure β-Ga_2O_3 with (–201) plane orientation. However, comparing the diffraction peaks that are indexed to the {–201} plane family, the films obtained at 1150 and 1250 °C show considerably lower intensity, as shown in Figs. 3(c) and 3(d).

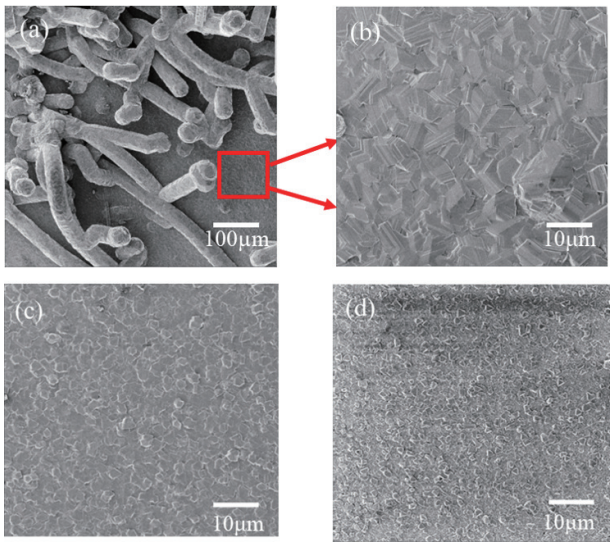

Fig. 4. The top view of SEM images of β-Ga_2O_3 film deposited at different growth temperatures. (a) Large area of film grown at 950 °C. Small area of film grown at (b) 950 °C, (c) 1050 °C, (d) 1350 °C.

According to Fig. 4(a), the film grown at 950 °C exhibits a special microrod like structure, and not able to be measured in terms of thickness. Columns made up the majority of the film. Fig. 4(b) depicts the small area of the film with no obvious columns covering it. This is the evidence of the generation of crystal structures in this region at this temperature. An example of a film produced at 1050 °C is shown in Fig. 4(c), this is a surface that exhibits a high degree of uniformity. The top view of the SEM image of the film showed flat morphology, with almost no visible protrusions across the entire image area for the films obtained under 1050 and 1350 °C. However, the film grown at 1350 °C has fewer grains than other films, as shown in Fig. 4(d). In conclusion, this result confirms that high temperatures can contribute to the desorption of Ga_2O at a high rate. In this case, lower temperatures caused a decrease in the diffusion length of Ga_2O[34]. Moreover, as the diffusion energies of the deposited atoms are different according to the varied temperatures[35]. When the temperature was lower, the growth of the crystal was restrained due to the atoms not having sufficient energy to move to the substrate during the crystallization process. Whereas, with the temperature of 1050 °C, the atoms acquire enough energy to move and bond with adjacent nucleation sites. However, when the temperature rises further, the epitaxy speed decreases sharply owing to the migration of the atoms being too fast to form the regular bonds of a single crystal[32].

3.2. Effects of O_2 flow on film quality

In Fig. 5, the growth rates of films grown under different O_2 flow conditions are summarized. Increasing the O_2 flow from 5 to 20 sccm resulted in an increased growth rates from 3.1225 to 7.4745 μm/h. In the case of O_2 flow greater than 20 sccm, the rate of Ga_2O_3 films epitaxy decreased significantly. Growth rate increased when more O_2 flow was used since more Ga_2O vapor can fully react with O_2. O_2 fluxes of 50 and 100 sccm contributed to a reduction in film thickness as a result of more graphite powder reacting with O_2, less Ga_2O_3 powder was able to react adequately.

XRD patterns of β-Ga_2O_3 crystallized during deposition on c-plane sapphire under O_2 flow ranged from 5 to 50 sccm and are shown in Fig. 6. Diffraction peaks of (−201), (−402), (−603) appeared for deposition O_2 flow between 5 and 100 sccm. However, Fig. 6(a) shows the film grown at 50 sccm is not oriented properly in any plane. In Figs. 6(c) and 6(d), the intensity of the (−201) peaks are lower compared to those peaks obtained at O_2 flow of 20 sccm (Fig. 6(b)), this indicates that the β-Ga_2O_3 films have inferior [−201]-oriented crystallinity when 20 sccm O_2 is used in the experimental process. In other words, the film grown at 20 sccm O_2 has the greatest crystallization considering the relationship of parallel between β-Ga_2O_3 (−201) and c-plane sapphire (0001). The extension mode should to be domain matching epitaxy because the oxygen atoms in the (−201) equivalent plane of β-Ga_2O_3 have the same arrangement as the oxygen atoms of the c-plane (0001) plane[36, 37]. However, the too high oxygen pressures cause the poor property of crystalline of the films[38].

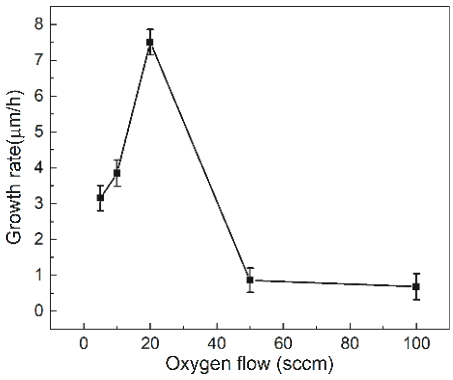

Fig. 5. The growth rate of β-Ga_2O_3 films grown at different O_2 flow ranged from 5 to 100 sccm.

According to the top view SEM of the films, the typical growth pattern of grains have excellent uniformity of the film grown at 50 sccm O_2 flow, as shown in Fig. 7(a). It should be noted, however, that the film obtained under 5 sccm O_2 did not reveal a very flat surface (Fig. 7(c)). It is due to the decrease of the O_2 flow, the growth rate increased. Poor surface morphology is not guaranteed by a low O_2 component. A haphazard pattern of surface islands of Ga_2O_3 has been observed in this case. With an O_2 flow of 20 sccm, the fastest growth rate was achieved, and good surface morphology was ensured with an appropriate O_2 fraction (Fig. 7(b)).

3.3. Effects of the graphite powder on film quality

To determine the effect of the molar ratio between graphite powder and Ga_2O_3 powder on the quality of Ga_2O_3 films, the graphite powder was adjusted in weight. It can be concluded from Fig. 8 that the growth rate was 25 μm/h when the molar ratio was 5 : 1. By doubling the graphite powder usage, the deposition rate declined to 12 μm/h, meanwhile, the thickness decreased to 25.32 from 50.23 μm. In order to confirm this tendency, fourfold graphite powder was used, the film growth rate decreased by nearly half to 7.4745 μm/h. This phenomenon can be explained by the fact that the newly introduced O_2 will react with the excess graphite powder, where there is insufficient O_2 to react with Ga_2O in the second period reaction.

In Fig. 9, XRD patterns of β-Ga_2O_3 films crystallized during deposition on c-plane sapphire are illustrated. The peak at 18.9° belongs to (−201), the other (−201) plane family

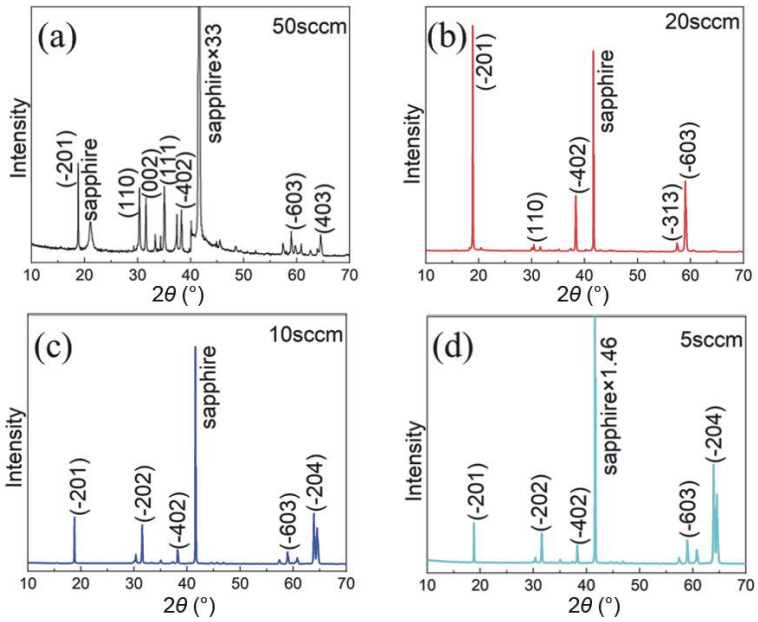

Fig. 6. (Color online) Typical XRD θ–2θ scan of the β-Ga_2O_3 films grown on c-plane sapphire substrate at different O_2 flow. (a) 50 sccm. (b) 20 sccm. (c) 10 sccm. (d) 5 sccm.

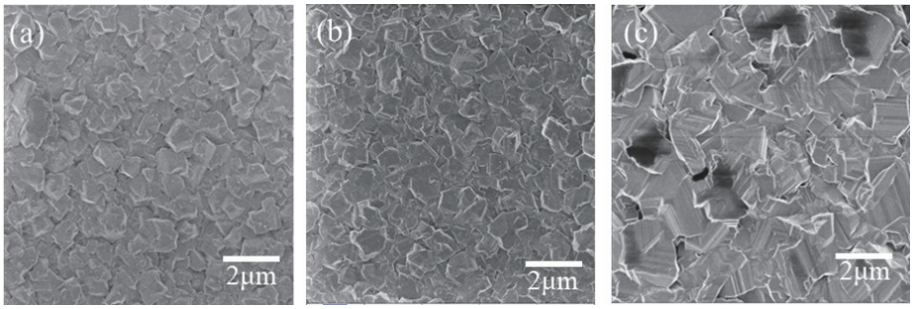

Fig. 7. The top view of SEM images of β-Ga_2O_3 film deposited at different O_2 flow. (a) 50 sccm. (b) 20 sccm. (c) 5 sccm.

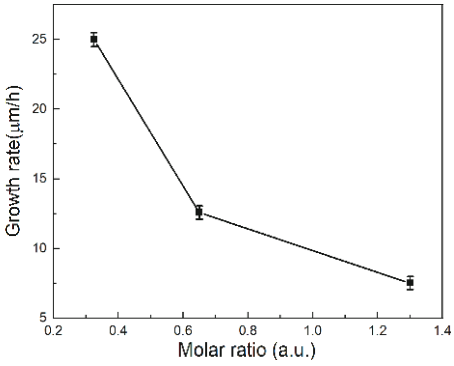

Fig. 8. The growth rate of β-Ga_2O_3 films grown at different molar ratio between graphite and Ga_2O_3 powder.

peaks appear with considerable intensities. However, the diffraction signal at 2θ = 30.3° and 57.6° is assigned to (110) and (–313) in Fig. 9(a). It was found in Fig. 9(c) that the film grown at the molar ratio of 5 : 1 possessing the (310) and (–601) planes. Compared to the {–201} peak of β-Ga_2O_3, these peaks have lower intensity. Nonetheless, at the molar ratio of 10 : 1, no obvious subpeaks are seen in Fig. 9(b). Meanwhile, the intensity of the diffraction peak gets its maximum at a molar ratio of 10 : 1. Hence, the ideal molar ratio between graphite powder and Ga_2O_3 powder is 10 : 1 for obtaining good β-Ga_2O_3 films.

In Fig. 10, we show SEM images of β-Ga_2O_3 films that were deposited on c-plane sapphire substrates. With a 20 : 1 molar ratio between graphite powder and high purity Ga_2O_3 powder, β-Ga_2O_3 films with good uniformity were obtained (see Fig. 10(a)). When the molar ratio was 5 : 1, β-Ga_2O_3 islands developed were observed to be more vertical to the substrates (Fig. 10(c)). This may be a result of too high a growth rate. It is impossible for these islands to become regular at a high growth rate. On the other hand, as shown by the SEM images, the size of Ga_2O_3 islands was larger when the molar ratio between graphite powder and Ga_2O_3 powder was 10 : 1 (Fig. 10(b)). In addition, the islands did not have sharp edges. Accordingly, the crystalline quality of β-Ga_2O_3 films can be enhanced by properly balancing the molar ratio between two types of powders.

4. Conclusions

In summary, the heteroepitaxy of β-Ga_2O_3/c-plane sapphire was investigated. The monoclinic films with the (–201) preferred orientation can be obtained indicating carbothermal reduction is a promising growth technology for growing high quality β-Ga_2O_3 films. According to the results of SEM and XRD, the growth rate, the orientation, and the morphology of the surface were strongly influenced by the temperature of the lower part of the furnace, the O_2 flow, and the molar ratio between Ga_2O_3 powder and graphite powder.

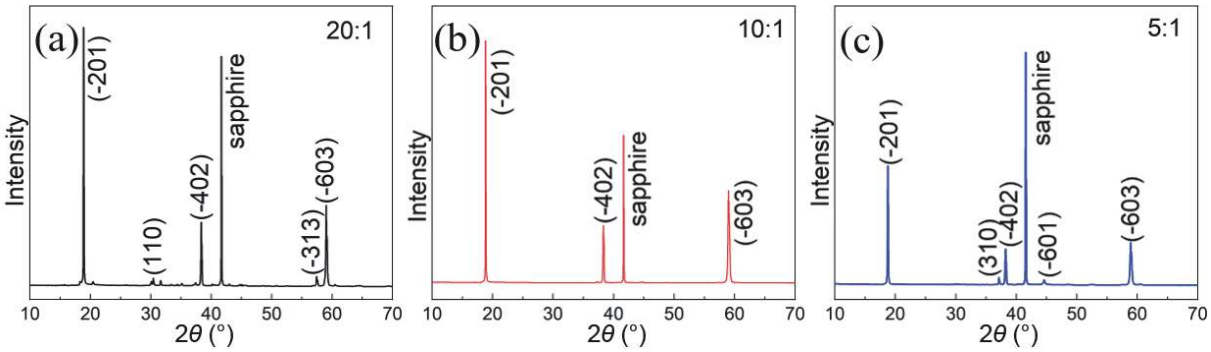

Fig. 9. (Color online) Typical XRD θ–2θ scan of the β-Ga_2O_3 films grown on c-plane sapphire substrate at different molar ratio between graphite powder and Ga_2O_3 powder. (a) 20 : 1. (b) 10 : 1. (c) 5 : 1.

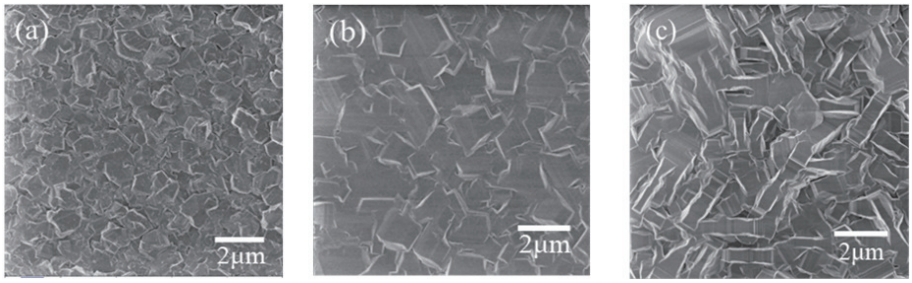

Fig. 10. The top view of SEM images of β-Ga_2O_3 film deposited at different molar ratio between graphite powder and Ga_2O_3 powder. (a) 20 : 1. (b) 10 : 1. (c) 5 : 1.

Higher growth rate, the best crystallinity and surface morphology can be realized by the growth temperature of 1050 °C. In addition, our results reveal that an increasing O_2 flux helps maintain the quality of morphology. However, the results of XRD indicate that the 20 sccm O_2 flow can ensure good crystalline property. As the molar ratio between Ga_2O_3 powder and graphite powder declined, the growth rate increased. The highest deposition rate of 25 μm/h has been achieved with sacrificing the crystalline quality of the β-Ga_2O_3 layers. The best quality of crystalline was achieved when the molar ratio was 10 : 1. These results indicate promising properties for the future development of the carbothermal reduction method.

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