REVIEWS

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Application of halide vapor phase epitaxy for the growth of ultra-wide band gap Ga_2O_3

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Abstract: Halide vapor phase epitaxy (HVPE) is widely used in the semiconductor industry for the growth of Si, GaAs, GaN, etc. HVPE is a non-organic chemical vapor deposition (CVD) technique, characterized by high quality growth of epitaxial layers with fast growth rate, which is versatile for the fabrication of both substrates and devices with wide applications. In this paper, we review the usage of HVPE for the growth and device applications of Ga_2O_3, with detailed discussions on a variety of technological aspects of HVPE. It is concluded that HVPE is a promising candidate for the epitaxy of large-area Ga_2O_3 substrates and for the fabrication of high power β-Ga_2O_3 devices.

Key words: halide vapor phase epitaxy; Ga_2O_3; Schottky barrier diodes; epitaxy growth

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1. Introduction

Gallium oxides (Ga_2O_3) has five different crystal structures, namely, α-, β-, γ-, ε-, and δ-phases[1]. Among them, monoclinic β-Ga_2O_3 structure is the thermodynamically most stable under atmospheric pressure. As a new ultra-wide bandgap semiconductor, β-Ga_2O_3 has a band gap as large as 4.8–4.9 eV[2–5] and a critical breakdown electric field of 8 MV/cm, much higher than SiC and GaN. It also has excellent chemical, mechanical and thermal stabilities at elevated temperatures. As a result, β-Ga_2O_3 is considered a suitable and promising ultra-wide bandgap semiconductor for a variety of applications. Current interests of β-Ga_2O_3 focus on three areas: (1) UV emission/detection devices for disinfection purposes and solar-blind sensor[6], (2) substrates for high-brightness vertically-structured and flip-chip GaN-LEDs[7, 8], and (3) high-power devices because the large band gap of β-Ga_2O_3 is advantageous for the realization of high-efficiency transistors[9–11]. At present, although the research on Ga_2O_3 is still at the initial stage, Ga_2O_3, which is suitable for large-scale production and has excellent chemical properties and thermal stability, will become one of the candidate materials for power electronic devices in the future.

One of the great advantages of β-Ga_2O_3 over GaN and SiC lies on the fact that high-quality single crystal wafers can be produced from melt by Czochralski (CZ)[12], floating-zone (FZ)[13, 14] and edge-defined film-fed growth (EFG)[15]. Up to date, EFG-grown 4-inch β-Ga_2O_3 wafers are commercially available. However, problems such as large size and easy cracking of Ga_2O_3 crystal are still not solved. The melt growth method enables easy growth of single crystal with less crystal defects and large diameter with relatively low cost. However, the EFG growth of β-Ga_2O_3 from melt requires the use of very expensive noble-metal crucibles (such as Ir). Besides, the melt methods require effective and precise control of the growth conditions, such as temperature distribution, oxidation atmosphere, and air pressure. At the same time, Ga_2O_3 is easy to decompose at high temperature to generate GaO, Ga_2O, Ga and etc., which will volatilize and corrode the precious metal crucible seriously. The melt growth method can also produce multiple crystals, double crystals, Mosaic structure, screw dislocation, cracking and other problems, such that it is difficult to grow large-area and high quality Ga_2O_3 crystals. In addition, there are also many difficulties for the control of crystal orientation, cutting and polishing processes of Ga_2O_3 crystals for device epitaxy because of the easy cleavage of Ga_2O_3.

One of the solutions for the above-mentioned problems is the realization of heteroepitaxial β-Ga_2O_3 film on large-area foreign substrates, such as sapphire. Thin film growth of β-Ga_2O_3 has also been realized by using molecular beam epitaxy (MBE)[9, 10, 16], pulsed laser deposition (PLD)[4], and metal-organic vapor phase epitaxy (MOVPE)[17]. However, it is usually difficult to grow high-purity β-Ga_2O_3 at high growth rates by these methods. To ensure sufficiently high device breakdown voltage, thick Ga_2O_3 layers need to be grown with controllable thickness, electrical conductivity in a cost-effective production manner. Therefore, the growth of β-Ga_2O_3 by halide vapor phase epitaxy was proposed, similar to the growth of high-purity GaN, ZnO and AlN by HVPE[18–20].

HVPE is widely used in the III–V semiconductor industry, especially for the preparation of free-standing GaN substrates in recent years. As a non-organic chemical vapor deposition technique, HVPE method is not only characterized by a fast growth rate and the high quality of thick epitaxial layers than other methods, but also characterized by the versatility for the fabrication of both substrates and devices. In this paper, the growth and device application of HVPE method for Ga_2O_3 are reviewed in details.

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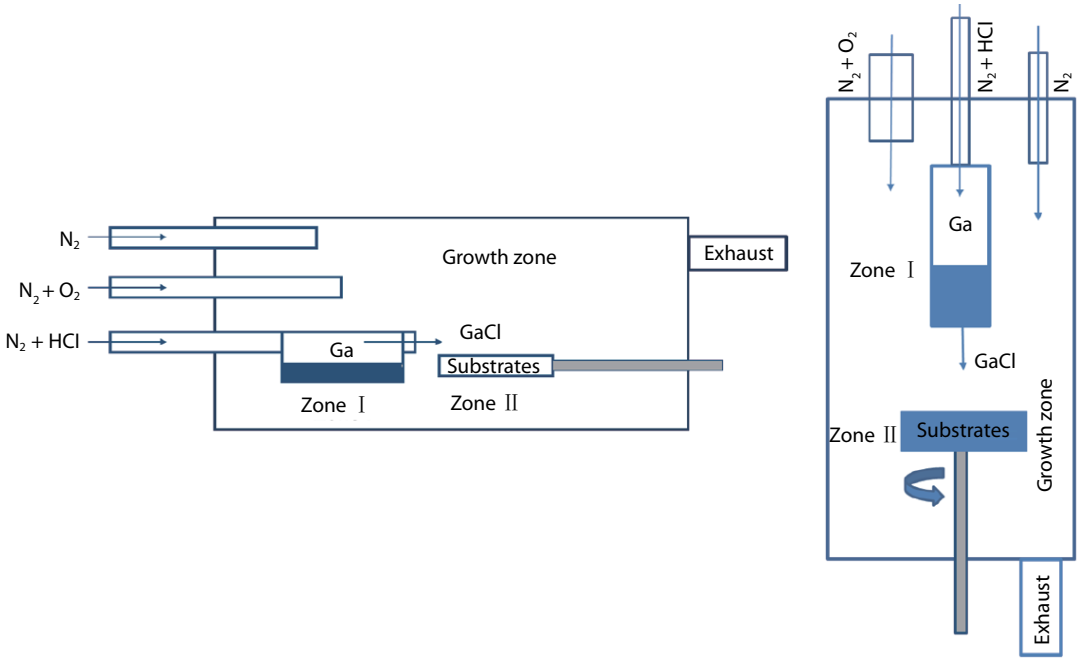

Fig. 1. (Color online) Schematic diagram of (a) horizontal HVPE and (b) vertical HVPE.

2. Halide vapor phase system for Ga_2O_3 growth

HVPE growth of β-Ga_2O_3 thin films is generally carried out at ~1050 °C by using gallium chloride (GaCl) and O_2 as the precursors. N_2, He or Ar can be used as the carrier gas. Nomura[21] has reported thermodynamic aspects of the growth of β-Ga_2O_3 by HVPE. GaCl and O_2 were determined to be the appropriate precursors for the HVPE growth of β-Ga_2O_3. The theoretical estimations agreed well with their experimental results on the homoepitaxial growth, indicating that the HVPE growth of β-Ga_2O_3 can be thermodynamically controlled[21].

In the HVPE system, HCl is more commonly used than chlorine gas due to the purity and operability. The GaCl was formed upstream in the reactor by the reaction between Ga metal and HCl gas. The main chemical reactions are as follows.

$$\mathrm{Ga(s)+HCl(g)\rightarrow GaCl(g)+H_2(g)},$$

$$\mathrm{GaCl(g)+O_2(g)\rightarrow Ga_2O_3(s)+Cl_2(g)}.$$

Gallium chloride has a variety of different valence states, e.g. GaCl, $GaCl_3$, etc. GaCl is stable at high temperatures, and will decompose into $GaCl_3$ below a certain temperature. This feature enables temperature adjustment of the reaction chamber to realize GaCl generation, transportation and deposition of Ga_2O_3. As a result, a typical HVPE growth system contains two reaction chambers at different temperatures, as shown in Fig. 1.

As a cost-effective growth technique, HVPE supports production of high quality crystals with a faster growth rate. The fastest growth rate reported to date was ~250 μm/h for the growth of homoepitaxial layer on β-Ga_2O_3 (001) substrate[22]. However, the as-grown Ga_2O_3 films suffer from large surface roughness due to the high growth rate, and an additional polishing procedure is required prior to device processing. Besides, the Ga_2O_3films show the presence of Cl-induced impurities and defects because of the use of GaCl as the Ga source.

To realize n-type doping of β-Ga_2O_3 thin films grown by HVPE, either $SiCl_4$ or $SnCl_4$ can be used as the efficient dopant source[23]. Besides, semi-insulating Ga_2O_3 substrates can be achieved by using Fe or Mg dopants[24, 25]. In the HVPE system, Fe-doped Ga_2O_3 can achieved by HCl flowing through the metal Fe to form $FeCl_2$ as the dopants. Of course, organic iron compound can be used as the dopants.

3. HVPE for the growth of polymorph Ga_2O_3

Note that the β-Ga_2O_3 structure is the most stable phase of Ga_2O_3, among the five different phases (α, β, γ, ε, and δ)[1, 2]. So, HVPE growth for β-Ga_2O_3 have already been studied. However, the interests in other Ga_2O_3 phases have also arisen in recent years, particularly the metastable rhombohedral α- and hexagonal ε-Ga_2O_3 phases, both of which have been observed to grow epitaxially on oriented substrates. The α- and ε-phases are of particular interest because of their higher symmetry and simpler epitaxial relationships with c-plane sapphire. As a result, α, β, ε-Ga_2O_3 have all been obtained by HVPE. All reported films containing only α- and ε-Ga_2O_3 were grown at temperatures of $\leqslant$ 800 °C, and the growth temperature of β-Ga_2O_3 is generally above 850 °C.

3.1. β-Ga_2O_3 HVPE growth

The first HVPE growth of β-Ga_2O_3 has been reported by Matsumoto *et al.*[26]. Small flakes or needle-like β-Ga_2O_3 were synthesized by the reaction between GaCl gas and O_2 at 1100–1150 °C. Foreign substrates, such as sapphire[27–32], MgO[27, 33] and etc, have been used for the growth of β-Ga_2O_3. The substrate symmetry would affect the formation of in-plane rotational domains, which results in the growth of β-Ga_2O_3 thin-films with highly textured surface and single out-of-plane orientations. For example, (-201) oriented β-Ga_2O_3 layers on sapphire (0001) consist of six in-plane rotational domains with six-fold symmetry in accordance with the substrate symmetry[16, 27–29, 31]. Similarly, (100) and (102) oriented β-Ga_2O_3 layers are obtained on MgO (100) and (110) with four- and two-fold symmetric in-plane rotational domains, respect-

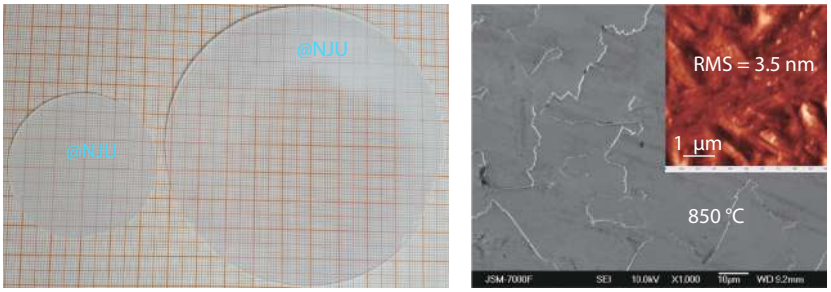

Fig. 2. (Color online) Optical photograph (unpublished) and SEM images of HVPE grown β-Ga_2O_3 at 850 °C.

ively[27, 30]. Y Oshima *et al.*[22] reported that the rotational domains could be suppressed in β-Ga_2O_3 grown on off-angled (0001) sapphire substrates, which shows no in-plane rotational symmetry. The growth rate can reach over 250 μm/h. However, the fast growth rate also leads to thin films with rough surface. In fact, even at low growth rates, the surface roughness of β-Ga_2O_3 layers grown by HVPE is relatively large. In 2017, Xiu *et al.* from Nanjing University, had successfully grown 2 and 4 inch β-Ga_2O_3 films on sapphire substrate by HVPE[32]. Fig. 2 shows the optical photograph and the SEM images of HVPE grown β-Ga_2O_3 layers[32]. The β-Ga_2O_3 films are transparent with roughened surface at micrometer scale. For β-Ga_2O_3 film grown at 850 °C by HVPE, the root mean square (RMS) value by atomic force microscopy is larger than 3 nm (shown in inset of Fig. 2). And the RMS value of β-Ga_2O_3 films becomes larger with increasing the growth temperature.

3.2. α-Ga_2O_3 HVPE growth

α-Ga_2O_3 is also a very promising wide-bandgap semiconductor. α-Ga_2O_3 has been reported to have a large bandgap energy (E_g = 5.3 eV)[34] and its electrical conductivity can be well-controlled[35–36]. Moreover, α-Ga_2O_3 has been proposed to fabricate novel functional materials through the formation of solid solutions with other corundum-structured oxides, such as α-Al_2O_3[37].

Although trigonal corundum-structured α-Ga_2O_3 exists in a meta-stable phase compared to the β-Ga_2O_3 and cannot be obtained from the melt method, α-Ga_2O_3 has been hetero-epitaxially grown on sapphire substrates by HVPE[38], which is the first report about the successful high-speed growth of high-purity (0001) α-Ga_2O_3 by HVPE.

α-Ga_2O_3 with the same corundum structure is the best lattice-matched polymorph with sapphire. The lattice mismatches between α-Ga_2O_3 and sapphire along the *a*- and *c*-axes are ~4.5 and ~3.3%, respectively. Although the lattice mismatch along the *c*-axis is considerably large, the pseudomorphic growth of α-Ga_2O_3 on α-Al_2O_3 can be stabilized because they share a common corundum structure. Once the first layer of α-Ga_2O_3 is formed, the structural similarity will promote the pseudomorphic growth.

It has been discovered that the deposition temperature was within the range of 525 to 650 °C[38] and the growth rate can reach approximately 150 μm/h, over two orders of magnitude larger than those of conventional mist CVD or MBE. It means that HVPE is an excellent candidate for growing thick α-Ga_2O_3 films or even free-standing α-Ga_2O_3 wafers. The films are twin-free and have no detectable formation of β-Ga_2O_3. The optical bandgap is estimated to be 5.16 eV. The epitaxial relationships are also determined to be [10-10]α-Ga_2O_3||[10-10] sapphire and (0001)α-Ga_2O_3|| (0001) sapphire.

3.3. ε-Ga_2O_3 HVPE growth

Epitaxial growths of the metastable ε-Ga_2O_3 phases have also been reported recently. Oshima[39] reported the epitaxial growth of ε-Ga_2O_3 by HVPE for the first time. The pure phase ε-Ga_2O_3 films are grown on (0001) GaN, (0001) AlN, and (-201) β-Ga_2O_3 by HVPE at 550 °C, although some minor misoriented domains are observed. The ε-Ga_2O_3 is thermally stable up to approximately 700 °C. The optical bandgap of ε-Ga_2O_3 is determined for the first time to be 4.9 eV.

Yao *et al.*[40] also reported metastable α- and ε-phases grown by HVPE between 650 °C and 850 °C. The epitaxial relationship was revealed to be [-1100] ε-Ga_2O_3||[11-20] α-Ga_2O_3||[11-20] α-Al_2O_3, as shown in Fig. 3.

4. HVPE growth of Ga_2O_3 for nitride epitaxy and device application

Currently, HVPE growth of Ga_2O_3 focus on two areas. One is to grow the drift-layer for β-Ga_2O_3 Schottky barrier diodes, the other is to grow β-Ga_2O_3 films as the substrate or the buffer layers for III-nitrides epitaxy.

4.1. β-Ga_2O_3 Schottky barrier diodes

β-Ga_2O_3 is expected to have much larger Baliga's figure of merit than SiC and GaN due to the predicted high breakdown electric field (~ 6–8 MV/cm) and high electron mobility (μ ~ 200–300 $cm^2/(V{\cdot}s)$). Currently, Schottky barrier diodes (SBDs) and FETs are being intensively investigated as the fundamental device components for power conversion systems. β-Ga_2O_3 based Schottky barrier diodes (SBDs) have been demonstrated, showing the reasonably good device characteristics[41–45]. The reverse blocking voltage of field-plated lateral β-Ga_2O_3 SBD on sapphire is now more than 3 kV[46].

In the early years, the development Ga_2O_3 SBDs was behind FETs, due mainly to a lack of suitable epitaxial growth techniques for the growth of thick n-Ga_2O_3 drift layer. In order to ensure the sufficient break down voltage, relatively thick n-Ga_2O_3 layers need to be grown with controlled thickness, good electrical conductivity in a cost-effective manner. HVPE method offers the possibility of growing this thick drift layer with high quality. Recently, the full-scale Ga_2O_3 SBDs were fabricated on epitaxial wafers with HVPE grown Si-doped n-Ga_2O_3 drift layers on n^+-Ga_2O_3 (001) substrates[45]. A Ga_2O_3 SBD power rectifier with a breakdown voltage of 400 V was successfully demonstrated. In 2016, Higashiwaki *et al.*[47] improved the breakdown voltage up to 500 V by increasing the drift layer thickness and doping concentration. The device structure is shown in Fig. 4. The Ron values included a substrate resistance as

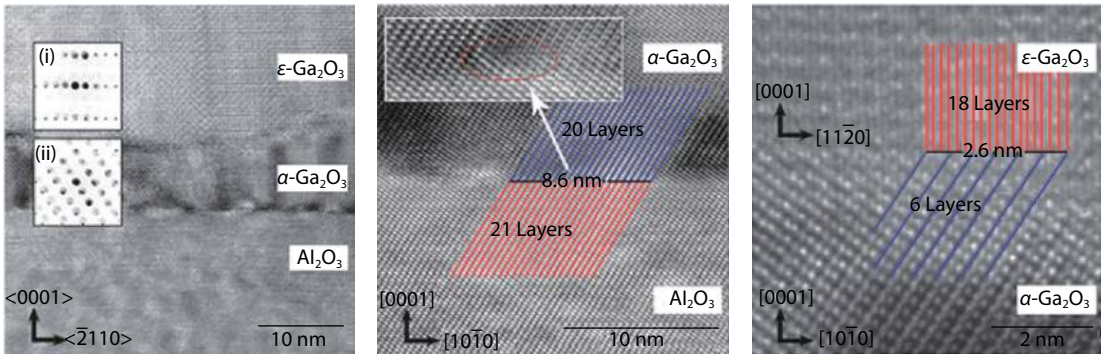

Fig. 3. (Color online) The cross-section HRTEM of α- and ε-Ga_2O_3 on c-plane sapphire[40].

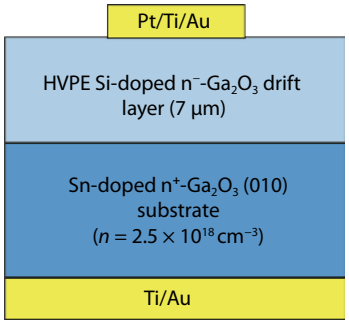

Fig. 4. (Color online) Schematic Ga_2O_3 SBD structure with an HVPE-grown n-Ga_2O_3 drift layer.

large as 1.0 mΩ·cm^2, which is near to the ideality factors of the SBDs (~1.03–1.07).

Until now, the highest reported breakdown voltage is ~1.1 kV for β-Ga_2O_3 drift-layer based field-plated SBD grown by HVPE[48]. Yang *et al.*[43] reported that the reverse breakdown voltage of β-Ga_2O_3 rectifiers without edge termination over 1 kV and the ideality factor increased from 1.08 to 1.28 with decreasing the Schottky barrier height. The schematic Ga_2O_3 SBD device structures is shown in Fig. 5.

The above-mentioned results show that β-Ga_2O_3 Schottky rectifiers fabricated by HVPE technology are promising candidates for high power devices.

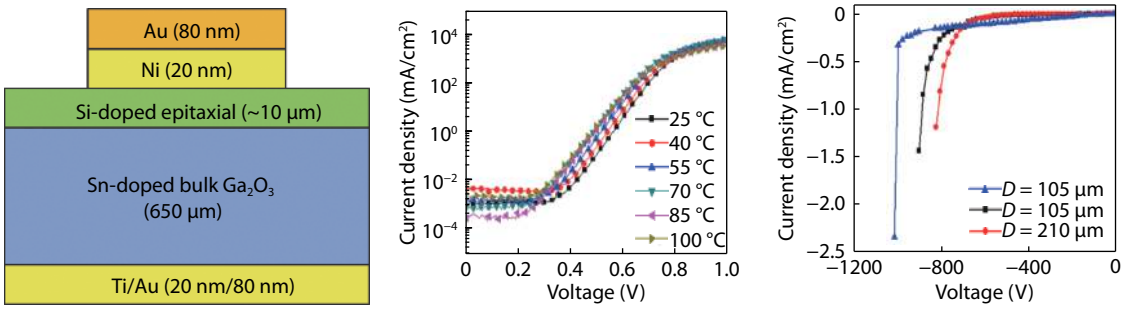

Fig. 5. (Color online) Schematic structure, forward and reverse J–V characteristics of Ni/Au Ga_2O_3 SBD.

4.2. HVPE β-Ga_2O_3 films for III-nitrides epitaxy

β-Ga_2O_3 is also promising as substrate or buffer layer for epitaxial growth of GaN and related III-nitrides. Native GaN substrates are the best choice for the fabrication of III-nitride devices as only homoepitaxial growth provides the lowest defect density, zero lattice and thermal mismatch. However, native GaN substrates are costly and difficult to obtain despite recent progress in bulk GaN single crystal synthesis. For this reason, the majority of GaN-based devices are produced by heteroepitaxial growth on foreign substrates such as sapphire, Si or SiC. However, a lot of challenges still exist because of the insulating nature of sapphire, high price and high UV absorption of SiC, large lattice mismatch and chemical interaction of Si with GaN. As a result, bulk GaN remains prohibitive for the majority of device applications.

Sapphire is generally used as the substrate for GaN growth, but it is still limited because of poor lattice match (13.9%) and insulating nature of sapphire. In contrast, the lattice mismatch between β-Ga_2O_3 and GaN is significantly lower. β-Ga_2O_3 is excellent to serve as substrate for the growth of III-nitrides. It also combines the advantages of being transparent and conductive. Especially, high quality bulk β-Ga_2O_3 crystals can be produced by melt-growth techniques at low cost and high throughput.

As a result, there have been several reports published recently on the growth of GaN epilayers on differently oriented β-Ga_2O_3 substrates by MOVPE, MBE and HVPE[49–57]. For GaN films grown on β-Ga_2O_3, the epitaxial relationship of (011) β-Ga_2O_3||(10-10) GaN only shows a lattice mismatch of ~2.6%[7]. M Muhammed *et al.*[52] studied GaN growth on (-201) β-Ga_2O_3 substrates. They found that the epitaxial relationship is (010) β-Ga_2O_3||(11-20) GaN with a lattice mismatch of 4.7%. Recently, the growth of GaN-based light emitting diodes on β-Ga_2O_3 substrates has been demonstrated by Xie *et al.*[51].

Nikolaev *et al.*[57] reported GaN growth on bulk crystal Ga_2O_3 substrates by HVPE technique. Earlier, pseudo-HVPE growth of bulk GaN directly on single crystalline (100) β-Ga_2O_3 were reported, where the β-Ga_2O_3 substrate was *in situ* heat-treated in NH_3 ambient before GaN epitaxy[56]. After the nitridation of Ga_2O_3 substrate, a porous GaN buffer layer would be formed on the surface of β-Ga_2O_3 substrate[58] and hence the

crystal quality of subsequent GaN epilayers were significantly improved.

In these cases, high quality bulk crystal β-Ga_2O_3 have been used as the substrate for GaN epitaxy. However, bulk crystal β-Ga_2O_3 is still very expensive. Recently, we also have grown β-Ga_2O_3 thin films by HVPE[33] and *in situ* regrown GaN thick films, and successfully obtained free-standing GaN substrate[58]. It should be noted that the growth and nitridation of β-Ga_2O_3 and re-growth of GaN can all be *in situ* completed in the HVPE system, which facilitates the growth process of GaN. Self-separation of GaN from the substrate can be realized due to easy cleavage of the Ga_2O_3 buffer layer. As a result, we expect that HVPE could be developed as a universal equipment for the epitaxy of GaN, Ga_2O_3 and other related novel heterostructures.

5. Conclusions

In this paper, we have reviewed the HVPE growth and the latest progress in the area of the ultra-wide bandgap Ga_2O_3. It is concluded that HVPE is a promising candidate for the epitaxy of large-area Ga_2O_3 substrates and for the fabrication of Ga_2O_3-based high power devices. We also present discussions on the technological advantages and aspects of HVPE for epitaxy growth and high-power devices (such as SBDs) of Ga_2O_3. We expect that HVPE can be further developed as an all-purpose equipment for the large-scale, low-cost epitaxy of GaN and Ga_2O_3.

Acknowledgements

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