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Improved surface morphology of flow-modulated MOVPE grown AlN on sapphire using thin medium-temperature AlN buffer layer

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Abstract

High-temperature (HT) AlN films were grown on (0 0 0 1) sapphire by low-pressure flow-modulated (FM) metal organic vapor phase epitaxy (MOVPE) with and without inserting a thin medium-temperature (MT) AlN layer. To suppress parasitic reactions between the sources of trimethylaluminum (TMA) and ammonia (NH_3), TMA and NH_3 was introduced to the reactor of MOVPE by alternating supply way. Surface morphology and crystalline quality were characterized by a scanning electronic microscopy (SEM), atomic force microscopy (AFM) and X-ray rocking curve (XRC) measurements of (0 0 0 2) and (10–12) diffractions. The AFM and SEM measurements indicated that the thin MT-AlN layer had a strong influence on the surface morphology of the HT-AlN films. The surface morphology became quite smooth by inserting the thin MT-AlN layer and surface RMS roughness values were 0.84 nm and 13.4 nm for the HT-AlN films with and without inserting the thin MT-AlN buffer layer, respectively. By etching the samples in aqueous KOH solution, it was found that the polarity of AlN films was different, the HT-AlN film with the thin MT-AlN layer could not be etched, indicating that the film had an Al-polar surface; however, the film without the MT-AlN layer was etched, which was explained that that film had a N- or mixed-polar surface. The mechanism for the origin of the different polarity of HT-AlN with and without the thin MT-AlN layer was proposed and discussed in detail.

Keywords: Metalorganic vapor phase epitaxy; AlN; Polarity; AFM; SEM; Flow-modulated method

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1. Introduction

AlN has attracted great attention because of its many important properties, which makes it lots of promising applications [1]. Its wide band gap of about 6.0 eV at room temperature (RT) has a potential application in deep ultraviolet (UV) devices [2]. It can be also used in high-power, high-frequency and high-temperature electronic devices because of its high breakdown voltage and high thermal conductivity (2.85 $W/cm\ K^2$) [3]. In addition, AlN is an ideal substrate material for deep UV, near UV and even high-efficiency blue emitters. Since, at present, it is difficult to fabricate the high-quality bulk AlN substrate, a high-quality AlN film on sapphire or SiC, i.e. AlN template, has been considered as a good choice of substrate for epitaxial growth of devices. Considering the cost, usually sapphire is used as a substrate for AlN film growth and several groups have reported MOVPE-grown high-quality AlN on sapphire substrate [4–6]. However, there are still some problems, required further investigation, in growth of AlN by MOVPE. For example, some groups reported growth of high-quality AlN on sapphire by using low-temperature AlN buffer layer [4,5], the others studied and found that it was no necessary of AlN buffer for growth of high-quality HT-AlN film [6]. Moreover, the crystalline quality and polarity of HT-AlN is very sensitive to initial growth state. Usually, a rough surface of HT-AlN film with N-polar or mixed-polar surface was formed when HT-AlN was grown on nitrided sapphire [7,8] and a smooth surface can be obtained if the AlN film has an Al-polar surface [9]. Therefore, in order to obtain high crystalline quality and Al-polar MOPVE-grown HT-AlN, careful control of the initial process condition is required. In this study, to avoid the strong parasitic reaction between TMA and NH_3, a flow-modulated method, that is TMA and NH_3 was alternatively introduced to the reactor, was employed. A thin medium-temperature AlN layer was used to control the polarity of MOVPE-grown HT-AlN and the origin of N-polar surface formation and the role of MT-AlN were discussed in detail.

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2. Experiment procedures

Approximately 600 nm-thick HT-AIN films with and without inserting thin MT-AIN layer were grown on (0 0 0 1) sapphire by FM-MOVPE. Trimethylaluminum (TMA) and ammonia (NH_3) were used as precursors. The flow rates of TMA and NH_3 are 40 sccm and 0.5 slm for growth of HT-AIN and the growth rate is about 1.2 nm per cycle. Fig. 1 shows the schematic structure of HT-AIN grown on sapphire and growth program with (Fig. 1a and b) and without (Fig. 1c and d) the thin MT-AIN layer. After thermal cleaning the sapphire substrate in H_2 atmosphere at 1100 °C, the growth procedure was started and no intentional nitridation was carried out. The thin MT-AIN layer (~50 nm) was deposited at 800 °C using conventional method, i.e. TMA and NH_3 was simultaneously introduced into the reactor with a V/III ratio of 32,000 and the HT-AIN films, however, were grown at 1150 °C by FM method with a growth pressure of 100 Torr. To avoid sapphire surface intentionally nitrided, the sequence of TMA and NH_3 of FM epitaxy method started from TMA and the following was NH_3. Because of the MOVPE system limited, there was 1 s interruption between the TMA and NH_3 supply. For excluding the effect of growth temperature on the surface morphology of HT-AIN films, HT-AIN was also grown at 1250 °C without the thin MT-AIN layer and at 1200 °C with the thin MT-AIN layer.

The surface morphology and structural quality of the AIN films were characterized by scanning electron microscopy (SEM), atomic force microscopy (AFM), high-resolution X-ray diffraction (HR-XRD) measurements. Surface polarity was evaluated by wet etching the HT-AIN films in aqueous KOH solution (10%) at 150 °C for 1 min.

3. Results and discussion

Fig. 2a shows a bird-eyes SEM image of HT-AIN grown directly on sapphire, and the inlet is the enlarged image of the HT-AIN. It can be seen that the surface of the HT-AIN is quite rough and a lot of hexagonal pyramid islands are observed on the HT-AIN surface. However, the FM-MOVPE grown HT-AIN with the thin MT-AIN layer has a very smooth surface and no additional islands appeared on the surface (Fig. 2b). AFM measurements with 10 μm × 10 μm scanning area further confirmed that the surface of HT-AIN on sapphire without MT-AIN layer is rough and that with MT-AIN layer is smooth. Their RMS (root-mean-square) roughness values are 13.54 nm and 0.84 nm, respectively (Fig. 3). It is well known that growth temperature has a great influence on the surface morphology and higher temperature (>1200 °C) is required to obtain a smooth surface, especially in the case of conventional MOVPE growth method [10]. Therefore, to check the influence of growth temperature on the surface morphology, the HT-AIN films were also grown at 1250 °C without the thin MT-AIN buffer layer and grown at 1200 °C with the thin MT-AIN buffer layer. The AFM measurements indicate that there are also many islands on the surface of the HT-AIN grown without the thin MT-AIN layer, conversely, the surface of the HT-AIN grown with the thin MT-AIN layer is mirror-like and quite flat. The RMS roughness (5 μm × 5 μm area) values of two samples are 11.94 nm and 0.54 nm, respectively. The results demonstrate that the effect of growth temperature on surface morphology of FM-MOVPE grown HT-AIN is not obvious.

Wu et al. investigated nitridation effect on the polarity, microstructure, and morphology of conventional MOVPE-grown

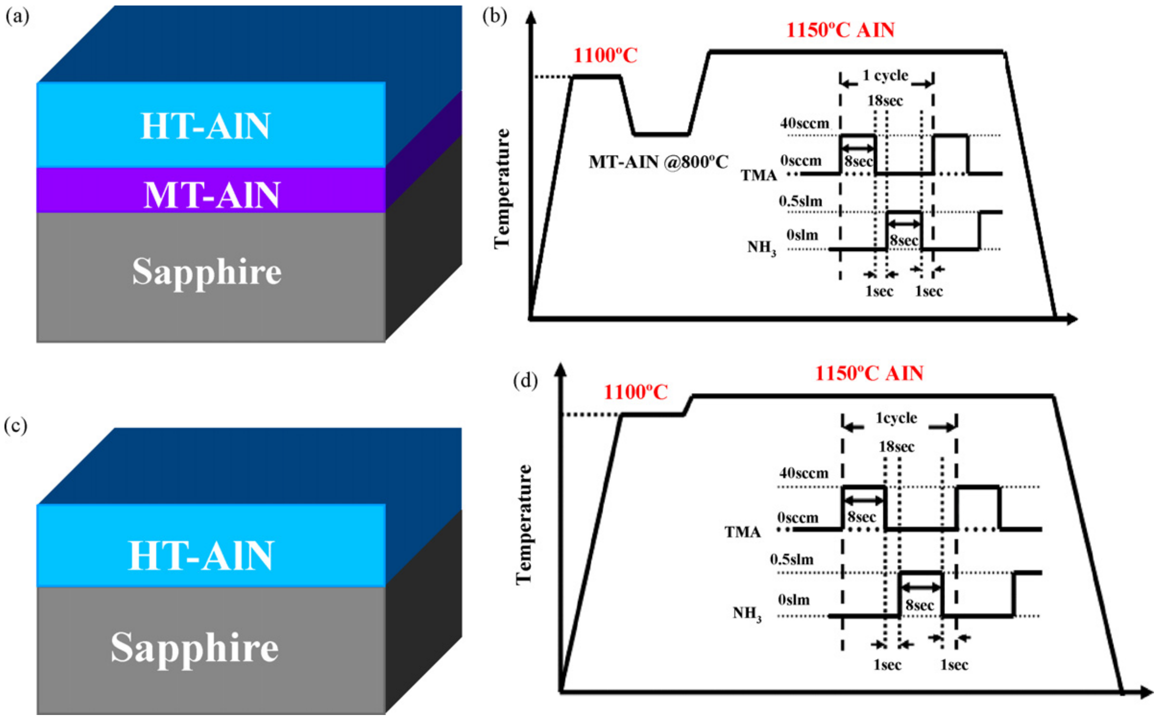

Fig. 1. Schematic structures of the HT-AIN films grown on sapphire with (a) and without (c) the thin MT-AIN layers; and schematic growth program and sequence of the source introduced into the reactor using flow-modulated method for the HT-AIN growth (b) and (d).