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Molecular-beam epitaxial growth of insulating AlN on surface-controlled 6H–SiC substrate by HCl gas etching

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Insulating AlN layers were grown on surface-controlled 6H–SiC subtrates by molecular-beam epitaxy (MBE) using elemental Al and rf plasma-excited nitrogen (N*). HCl gas etching was introduced as an effective pretreatment method of substrate for MBE growth of AlN. 6H–SiC substrates pretreated by HCl gas etching had no surface polishing scratches and an atomically flat surface. In addition, evident $(\sqrt{3}\times\sqrt{3})R30°$ surface reconstruction was observed even before thermal cleaning. AlN layers grown on this substrate had no defects related to surface polishing scratches and excellent insulating characteristics. © *2002 American Institute of Physics.*
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Silicon carbide (SiC) is one of the most promising materials for ultra low-loss high-voltage power devices. Although silicon dioxide (SiO_2) is widely used as a gate insulator in SiC field-effect transistors (FETs), electric field in the oxide becomes too high for a reliable operation. Aluminum nitride (AlN) is expected as an insulator of SiC power metal-insulator-semiconductor FETs (MISFETs) due to its high relative dielectric constant ($\epsilon_{AlN}=8.5\epsilon_0$)[1] compared to SiO_2 ($\epsilon_{SiO_2}=3.9\epsilon_0$). Although there are a few reports on AlN/SiC MIS devices,[2,3] insufficient quality of AlN layer is an obstruction to realize high-performance devices.

In order to obtain a high-quality AlN layer, a pretreatment of substrate surface is very crucial as well as the growth condition. In this study, HCl gas etching is suggested as an effective pretreatment method of substrate for molecular-beam epitaxial growth of AlN. It has been examined by atomic force microscopy (AFM) that HCl gas etching could remove surface polishing scratches and realize an atomically flat surface.[4] In this letter, surface features of HCl gas etched substrate and AlN layers were examined by using reflection high-energy electron diffraction (RHEED) and AFM. The electrical property of AlN layers was investigated by a current–voltage ($I-V$) measurement.

Substrates used in this study are commercially available n-type on-axis 6H–SiC $(0001)_{Si}$ face wafers (off angle $<0.2°$). The doping level of substrates is around $1\times10^{18}\,cm^{-3}$.

The substrates were prepared with a conventional chemical cleaning, and then loaded into a SiC chemical vapor deposition system for HCl gas etching. The HCl gas etching was carried out at 1300 °C for 10 min under a gas flow of HCl (3 sccm) diluted with H_2 carrier gas (1 slm). The details of HCl gas etching have been reported elsewhere.[4] After the etching, the substrates were transferred through the air into a molecular-beam epitaxy (MBE) system for AlN growth.

The MBE system is equipped with an effusion cell of elemental Al and a rf plasma cell for producing active nitrogen (N*). Substrate temperature was measured by a thermocouple located just behind the substrate. Although the actual substrate temperature may be lower than the thermocouple temperature, the measured temperature without any corrections is used as the substrate temperature in this letter. The substrate was thermally cleaned at 950 °C for 30 min in an ultrahigh vacuum, and then an AlN layer was grown at 900 °C under an optimized condition. The optimized condition was an Al flux of 3.3×10^{-7} Torr, rf power of 150 W, and nitrogen (N_2) flow rate of 0.5 sccm. Smaller and larger Al flux resulted in a rough surface and Al droplet formation, respectively. The growth rate under this condition was 0.3 μm/h.

Figure 1(a) shows an AFM image of HCl gas etched 6H–SiC substrate. Polishing scratches, which are usually observed on the surface of as-received wafer, were removed and an atomically flat terrace structure with a 6 monolayer (ML)-height atomic step was formed, as previously reported by Nakamura *et al.*[4] Removing surface polishing scratches will reduce defects of AlN layer and improve electrical properties of an AlN/SiC heterostructure.

The crystal structure of AlN is wurtzite (2H), which differs from that of a 6H–SiC substrate. To eliminate stacking mismatch boundary (SMB)[5] between AlN nuclei on different terraces of 6H–SiC, the step height of SiC should be 6 ML. A step height was well controlled to be 6 ML by HCl gas etching,[4] which is very important to grow high-quality AlN.

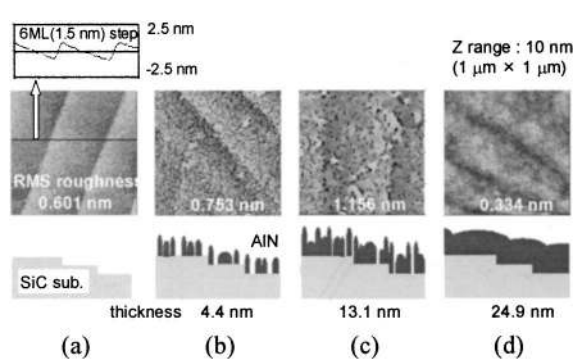

FIG. 1. AFM images of HCl gas etched 6H–SiC and AlN layers. (a) HCl gas etched 6H–SiC, (b) AlN 4.4 nm, (c) 13.1 nm, and (d) 24.9 nm.

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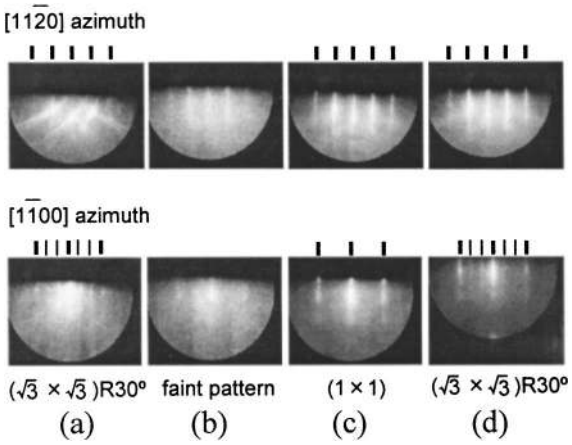

FIG. 2. RHEED patterns during AlN growth (a) HCl gas etched 6H–SiC surface, (b) AlN surface during 1 min from start of growth, (c) AlN surface during growth, and (d) AlN surface after growth.

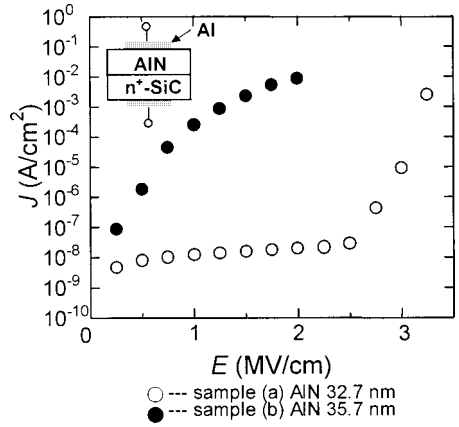

FIG. 3. Results of $I-V$ measurement. Sample (a) was a 6H–SiC substrate with HCl gas etching and sample (b) was a 6H–SiC substrate without HCl gas etching.

The surface feature of HCl gas etched substrate was investigated by using RHEED. Just after starting RHEED observation (~150 °C), a sharp and intense streak pattern with $(\sqrt{3}\times\sqrt{3})R30°$ surface reconstruction of 6H–SiC was already evident. The RHEED pattern at 900 °C is shown in Fig. 2(a). The observation of streak pattern with clear Kikuchi lines indicates that an atomically flat and clean surface was obtained by HCl gas etching. This fact corresponds to the AFM image as shown in Fig. 1(a). *In situ* x-ray photoelectron spectroscopy (XPS) measurements revealed that the Si/C intensity ratio of the surface is about 20% larger than that of SiC substrate without HCl gas etching. The $(\sqrt{3}\times\sqrt{3})R30°$ surface reconstruction has been reported as a 1/3 ML excess Si adsorbed surface.[6,7] Our XPS results agree with those reports.

The RHEED patterns during the growth of AlN are also shown in Fig. 2. Just after the start of AlN growth, the RHEED pattern became faint. Then, a (1×1) streak pattern of AlN gradually appeared and became evident after 1 min growth. After the AlN growth, the (1×1) pattern changed to a $(\sqrt{3}\times\sqrt{3})R30°$ pattern under N* irradiation as shown in Fig. 2(d). The observation of streak pattern during the growth and surface reconstruction after the growth suggests that a flat and uniform AlN layer was grown on a 6H–SiC substrate.

Figure 1(b)–(d) show the surface morphology of AlN layers with various growth times. During 1 min from the start of AlN growth, island-like growth occurred, resulting in a rough surface. As AlN growth proceeded, islands coalesced each other and the surface became flat. The root-mean-square roughness decreased with increasing layer thickness and approached about 0.3 nm which is small enough for device applications.

Structural properties of AlN layers on HCl gas etched 6H–SiC substrates have been studied by using high-resolution x-ray diffraction.[8] The evolution of a *c*-axis lattice constant with increasing AlN layer thickness clearly indicated that lattice relaxation occurred at the critical thickness (~5 nm) owing to 1% lattice mismatch between AlN and 6H–SiC. It is thought that AlN layers beyond the critical thickness contain misfit dislocations. The detailed investigations on the crystalline quality of AlN layers are discussed elsewhere.[8]

The electrical property of AlN layers was investigated by using Al/AlN/n^+-SiC MIS diode structures. Al electrodes (thickness 100 nm and diameter 300 μm) were formed on the AlN surface by vacuum evaporation. The back side contact for the SiC substrate was obtained by using silver paste. Two samples were fabricated. Samples (a) and (b) were grown on 6H–SiC substrates with and without pretreatment of HCl gas etching, respectively. The AlN layer thicknesses were about 30 nm. Figure 3 shows the result of $I-V$ measurement. The bias direction is an accumulation mode, i.e., the Al electrode is biased to positive. The electric field (E) applied to the AlN layer was calculated by dividing the applied voltage with the AlN layer thickness. Leakage current in the opposite bias direction (not shown here) was smaller than that in the accumulation mode.

The sample (a) exhibited an excellent insulating property. The resistivity of this AlN layer was $6.8\times10^{13}\,\Omega$ cm. Leakage current was as small as 10^{-8} A/cm^2 below 2.5 MV/cm. Although misfit dislocations are thought to exist in this AlN layer, the insulating property was not deteriorated remarkably. Gate leakage was one of serious problems for AlN/SiC MISFETs.[3] Therefore, this is a considerably hopeful result to apply an MBE-grown AlN for an insulator of SiC MISFET. The breakdown field was around 3 MV/cm, which is relatively small. Further study of breakdown mechanisms is under investigation.

The sample (b) showed poor insulating characteristics with large leakage current even under low electric field. This is partly attributed to defects related to surface polishing scratches in the AlN layer as well as electric field crowding at the scratches or other defects related to SMB.

From these results, we can conclude that the HCl gas etching of 6H–SiC substrate is very significant to achieve a high-quality AlN layer in terms of structural properties and insulating characteristics.

In this letter, we introduced HCl gas etching as an effective pretreatment method of substrate for MBE-growth of AlN. 6H–SiC substrates pretreated by HCl gas etching had no surface polishing scratches and an atomically flat surface. In addition, evident $(\sqrt{3}\times\sqrt{3})R30°$ surface reconstruction corresponding to 1/3 ML/Si adsorption was observed even

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before thermal cleaning. The RHEED observation and AFM images revealed that very flat and high-quality AlN layers were grown on HCl gas etched 6H–SiC substrates. These AlN layers had no defects related to surface polishing scratches and excellent insulating characteristics ($\rho \sim 6.8 \times 10^{13}\ \Omega$ cm).

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