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Ferroelectric-like behavior in HfO$_2$/Al$_2$O$_3$/AIN metal-insulator-semiconductor capacitor through AlN thermal stress

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Abstract

By modulating the thermal stress during film growth, the strained aluminum nitride (AIN) thin films with ferroelectric-like behavior were successfully grown by metal organic chemical vapor phase deposition (MOCVD) on silicon (Si) (111) substrate. The capacitors with strained AlN were fabricated and experimentally demonstrated, the linear to ferroelectric-like behavior on the fabricated AlN capacitors was observed, and the behavior was explained in terms of residual strains in the film. This work reports the ferroelectric-like properties of AlN film grown under specific deposition conditions for the first time.

1. Introduction

Due to superior properties such as the wide bandgap, high electron mobility, and high piezoelectric polarization, GaN-based semiconductors have become very important semiconductor materials for next generation power switching and millimeter-wave device applications in the recent decade [1–4]. Among them, aluminum nitride (AlN) which is consisted of Al and N in the wurtzite structure, has been an essential III–V semiconductor material for commercial optoelectronics and high electron mobility transistor (HEMT) applications [5–7]. Furthermore, the polar wurtzite structure of AlN within space group P6$_3$mc results in piezoelectricity, the Al in the compound can be substituted by scandium and boron to form aluminum scandium nitride (Al$_{1-x}$Sc$_x$N) and aluminum boron nitride (Al$_{1-x}$B$_x$N) ternary materials with the ferroelectric properties in recent years [8–13].

So far, AlN is well-known to be polar piezoelectric, and no ferroelectricity has ever been reported for this material in the past. Though the wurtzite structures suffering the stress were predicted to possess the ferroelectricity by reducing the potential surface of relative cation displacement in the unit cell [14–16], only a few studies previously reported the partial ferroelectric switching for pure AlN thin films [12, 17, 18]. Recently, a study also demonstrates a similar polarization reversal in pure AlN films [13]. These papers indicate the possibility of the realization of ferroelectricity for AlN.

In this work, we experimentally demonstrate a strained AlN thin films with ferroelectric-like behavior grown by MOCVD on Si (111) substrate for the first time. The AlN thin films grown at 500, 800, and 1100 °C were studied for the crystal structure and the strain in the film. Moreover, the transition from linear to ferroelectric-like behavior is observed for the hafnium oxide (HfO$_2$)/aluminum oxide (Al$_2$O$_3$)/AlN metal-insulator-semiconductor (MIS) capacitors with different AlN deposition temperatures by polarization—electric field (PE) curve measurement.

2. Experiment

All AlN films were grown by MOCVD (Thomas Swan 6 × 2″ close couple showerhead, Aixtron, Herzogenrath, Germany) on a highly doped p-type Si (111) substrate which was also used as the bottom electrode (BE). Before
loading the silicon wafers into the load-lock chamber, RCA clean was performed to clean the surface. Trimethylaluminum (TMA) and ammonia (NH₃) were used as the chemical precursors of Al and N with hydrogen (H₂) as the carrier gas. Prior to the growth of AlN films, the baking treatment with high H₂ flux at 1100 °C was performed in the MOCVD chamber to remove the native oxides and particles. During the AlN film growth, the TMA and NH₃ were injected under H₂ ambience at 100 torr, which was simultaneously injected at 500, 800, and 1100 °C, respectively. At the end of the AlN growth, the films were cooled with NH₃ and N₂ of 200 torr until the temperature reached 400 °C.

MIS capacitors were fabricated using a p-type Si (111) substrate with boron doping (0.01–0.025 Ω cm). After the surface cleaning, the HfO₂/Al₂O₃/AlN dielectric stack was deposited at 250 °C on the as-grown AlN samples by atomic layer deposition (ALD) (Fiji G2, Veeco, Plainview, United States). To prevent the o-phase formation of HfO₂, the annealing process was not performed in this study. Subsequently, AlN MIS capacitors were fabricated and the PE characteristics were evaluated. The Ti (50 nm) metal was deposited as top electrodes (TE) by electron beam evaporator while patterned by lift-off method to form MIS capacitors, the structure was as shown in figure 1 (a).

The crystal structure and crystal quality of the AlN thin films were studied by high resolution x-ray diffraction (HRXRD) (PAAnalytical X Pert Pro, Malvern Panalytical, Worcestershire, United Kingdom) with Cu Kα radiation. The reciprocal space mapping (RSM) for AlN grown at different deposition temperatures were performed for the evaluation of strain by PAAnalytical X Pert Pro. The transmission electron microscope (TEM) and energy-dispersive x-ray spectroscopy (EDX) were utilized for the material characterizations. The polarization-electric field (PE) curves were measured (Precision LC II, Radiant Technologies, Albuquerque, United States) at 1 kHz and applied by the positive-up-negative-down (PUND) procedure at room temperature. As shown in figure 1 (b), four voltage pulses were applied to the AlN MIS capacitors. The first and second pulses were positive; the sample was polarized in 1 ms (positive) pulse, so the corresponding current included ferroelectric (FE) and non-FE contributions, while the current during the second (up) pulse contained only non-FE contributions. Therefore, their subtraction only represented FE contributions. The same principle can be applied to the third (negative) and fourth (down) pulses in the negative state.
3. Results and discussion

The $\theta - 2\theta$ scan results of the $\sim 100$ nm AlN grown at 500, 800, and 1100 °C are as shown in figure 1(c), respectively. The peaks of the $\theta - 2\theta$ pattern of wurtzite AlN grown at 1100 °C are located at 28.45°, 36.13°, 58.90°, and 76.65°, the angles of the peaks correspond to Si (111), AlN (002), Si (222), and AlN (004) \cite{19, 20}, respectively. Meanwhile, the peaks of 37.70°, 43.99°, 64.30°, and 77.50° are matched to Al (111), Al (200), Al (220), and Al (311) \cite{21}, where the peaks are due to the aluminum holder. Therefore, the AlN film grown at 1100 °C consists of wurtzite-structured AlN along the c-axis. Similar to the sample grown at 1100 °C, all peaks are located in the same positions for the growth temperature of 500 °C and 800 °C, except for the AlN (002) peak which shifts to different location. Owing to the different growth temperatures, the c-axis in the AlN unit cell are affected, resulting in the shift of the AlN (002) peak. In figure 1(d), (002) peak shifts toward a higher angle with the increased growth temperature of AlN is observed in zoomed 2θ range. For the AlN grown at 500 to 1100 °C, the angle of (002) plane shifts from 35.66° to 36.13°, indicating an enormously residual stain along the c-axis, in particular for AlN grown at 500 °C. Similar results of AlN (002) peak shifts were reported for the post-deposition annealing on AlN/Si heterostructures \cite{22}.

The MIS capacitor with the AlN film grown at 500 °C was analyzed by TEM and EDX analysis, as respectively shown in figures 2(a) and (b). With the same ALD deposition conditions, the material characteristics of the HfO$_2$ and Al$_2$O$_3$ films for three MIS capacitors should be the same. The interface between AlN and Al$_2$O$_3$ is quite rough, which is because of the low growth temperature-induced low kinetic energy for Al adatoms \cite{23}, as shown in figure 2(a). Thus, the Al$_2$O$_3$ thin film is observed to be discontinuous between the HfO$_2$ and AlN films. Moreover, both HfO$_2$ and Al$_2$O$_3$ layers are amorphous phase. Figure 2(b) shows the distributions of chemical elements for the HfO$_2$/Al$_2$O$_3$/AlN MIS capacitor with the AlN grown at 500 °C. Due to the rough interface between the HfO$_2$/Al$_2$O$_3$ and the prolonged time during the ALD growth, the small amounts of Al unintentionally interdiffused through the HfO$_2$/Al$_2$O$_3$ dielectric stack to form the mixture of HfO$_2$ and Al$_2$O$_3$.

To evaluate both in-plane and out-of-plane strains of the AlN film grown at different growth temperatures, HRXRD RSM of the AlN grown at 500, 800, and 1100 °C are measured and the results are presented in figures 3(a)–(c). The MOCVD growth, three AlN/Si heterostructures went through the same process and the HfO$_2$ and Al$_2$O$_3$ layers were deposited by ALD simultaneously, so the stress in the MIS capacitors resulted by the HfO$_2$/Al$_2$O$_3$ layers was negligible.

The in-plane and out-of-plane strains in AlN were calculated from the formulas shown below,

$$\varepsilon_{\text{in-plane}}(\text{AlN}) = \frac{a_m - a_0}{a_0}$$  \hspace{1cm} (1)

and

$$\varepsilon_{\text{out-of-plane}}(\text{AlN}) = \frac{c_m - c_0}{c_0}$$  \hspace{1cm} (2)

by utilizing the measured lattice parameters $a_m$, $a_0$ (AlN = 0.311197 nm), $c_m$, and $c_0$ (AlN = 0.498089 nm), respectively \cite{24, 25}. A typical RSM for (10–14) reflection of AlN films grown at different temperatures within (313) reflection of a silicon substrate are shown in figure 3, extracting both lattice parameters $a_m$ and $c_m$ by
the reduction of the potential surface of ferroelectric due to the relative cation displacement in the unit cell. The shift of the process, the AlN grown at 500 °C demonstrates a larger strain along the a-axis because of larger thermal expansion coefficient and smaller lattice parameters [28]. With the increase of the growth temperature, AlN film demonstrates a larger strain along the a-axis because of larger thermal expansion coefficient (CTE). However, the AlN films grown at 500 and 800 °C suffer from tensile strains along the c-axis, except for the AlN grown at 1100 °C. It is particularly noteworthy that the AlN film grown at 500 °C has a larger out-of-plane strain of 1.015%. The AlN grown at 500 °C demonstrates the intrinsic compressive stress instead of the intrinsic tensile stress along a-axis because the Al adatoms lack the kinetic energy to coalesce each island and form the columnar AlN structure at the lower growth temperature. After the completion of the relaxation and the coalescence process, the AlN grown at 500 °C suffers the compressive stress along the a-axis as observed from the negative shift of (002) angle. The wurtzite structures suffering the stress are predicted to possess ferroelectricity owing to the reduction of the potential surface of ferroelectric due to the relative cation displacement in the unit cell [14–16].

The AlN films grown at 800 and 1100 °C in this material system are found to suffer from the in-plane tensile strain along the a-axis and demonstrate the relaxation state due to the strain difference between AlN and Si. This is reasonable because compared to the silicon substrate, III-nitrides have significantly higher thermal expansion coefficient and smaller lattice parameters [28].

| \( T_{\text{AlN}} (°C) \) | \( Q_z (µm) \) | \( Q_x (µm) \) | in-plane strain (%) | out-of-plane strain (%) |
|-----------------------------|----------------|----------------|-------------------|------------------------|
| 500                         | 0.2762         | 0.8137         | −1.429            | 1.015                  |
| 800                         | 0.2754         | 0.8157         | 1.301             | 0.335                  |
| 1100                        | 0.2741         | 0.8182         | 3.496             | −0.256                 |

Figures 4(a)–(c) show the PE curves at 1 kHz and figures 4(e)–(f) show the PUND results for the AlN MIS capacitors with different AlN deposition temperatures, respectively. To eliminate the leakage and improve the interfacial quality, we deposited the HfO\(_2\)/Al\(_2\)O\(_3\) (15/3 nm) dielectric stacks for the AlN MIS capacitors [29]. The AlN grown at 1100 °C exhibits the polarization switching curve which can sustain the applied electric field up to ±3 MV cm\(^{-1}\), then the dielectric breakdown happens. Meanwhile, the AlN grown at 800 °C exhibits the linear curves, indicating that AlN is a dielectric material. However, the AlN film grown at 500 °C reveals the partial ferroelectric reversal curves, but a PE curve can be measured with an approximate Pr of 30 µC cm\(^{-2}\), as shown in figure 4(a). The asymmetric polarization was also found in other nitride materials, associated with the polarization-dependent leakage [30, 31]. The asymmetric polarization behavior is due to the different interfaces at the Ti-HfO\(_2\) and AlN-Si. The existence of threading dislocation density at the interface of AlN-Si causes the vertical leakage current [32, 33], resulting in the asymmetric polarization in the MIS capacitor. Adjusting the growth conditions of MOCVD to either reduce in-plane compressive strain or enhance in-plane tensile strain.
may be possible to enable the enclosed demonstrations of polarization reversal. While there are indications of polarization switching, DC leakage current may lead to the confusion about the ferroelectricity in pure AlN thin films grown at 500 °C. Thus, we call this behavior as ‘ferroelectric-like’. A transition from linear to ferroelectric-like behavior can be clearly observed in the capacitors with the decrease of AlN growth temperature below 3 MV cm⁻¹. To eliminate the interference of the non-ferroelectric charge effect, the PUND method was utilized. After the subtraction of non-FE contributions, the PE curves of AlN grown at 500, 800, and 1100 °C are shown in figures 4(d)–(f). Similar to figure 4(a), the open PE curve of the MIS capacitor with AlN grown at 500 °C with ∼9 μC cm⁻² is observed after considering the PUND compensation when the applied electric field is 5 MV cm⁻¹. As shown in figures 4(g)–(i), all PE curves reveal low polarization intensity (<1 μC cm⁻²), except for the AlN grown at 500 °C, which reveals a larger polarization of 9 μC cm⁻². Nonetheless, the AlN grown at 500 °C still exhibits the ferroelectric-like behavior with the extremely asymmetry polarization in the negative electric field. Besides, the clearly enhanced polarization switching under the larger external applied electric filed is observed in the MIS capacitor with AlN grown at 500 °C. On the other hand, the MIS capacitor with AlN grown at 800 °C shows the linear curves from 1 to 4 MV cm⁻¹. Three AlN/Si heterostructures went through the same process and were deposited the HfO₂ and Al₂O₃ layers by ALD simultaneously, so the only variable factor was the AlN growth temperature. And thus, the linear curves should be mainly contributed by the AlN film grown at 800 °C. At the electric field of 5 MV cm⁻¹, the MIS capacitor with the AlN grown at 800 °C exhibits that the polarization can be slightly switched. The MIS capacitor with the AlN grown at 800 °C demonstrates the ferroelectric-like curve before the dielectric breakdown happens. With the increase of electric field, the polarization cannot increase simultaneously in the MIS capacitor with the AlN grown at 1100 °C. Even though the AlN grown at 1100 °C exhibits the ferroelectric-like behavior with the electric field of 3 MV cm⁻¹, the clear linear behavior is obtained only in the AlN grown at 1100 °C below 3 MV cm⁻¹. The non-electric field dependence-polarization is distinctly observed from 1 to 3 MV cm⁻¹. Therefore, the PE curve at 3 MV cm⁻¹ is considered to be linear rather than ferroelectric-like curve, associated with the large leakage current for the AlN grown at 1100 °C. Both measurement methods used in this letter exhibit half polarization curves only. Even with
the drive and sense probes exchange, the polarization curves are still partial and completely opposite. (not shown here) It may be attributed to the asymmetry polarization-dependent leakage due to the poor crystal quality caused by the low growth temperature and the asymmetric interfacial condition for measurements (top–to-bottom) [30]. For further study on the ferroelectric-like behavior of wurtzite-structure AlN, the improvements of the material growth and evaluation methods are needed. The mechanism of polarization rotation is not well investigated for the AlN yet. Two possible hypotheses are proposed for the strained AlN to exhibit ferroelectric-like behavior: (1) AlN must be suffered anomalously large tensile strains along in-plane and out-of-plane directions, which distorted the AlN crystal structure. With the lower growth temperature of AlN, the symmetry crystal structure of wurtzite AlN was worse. This asymmetry in AlN may result in the polarization switching of Al atoms under large enough electric field. (2) The tensile stress along the c-axis is effective in lowering the potential barrier (∼0.5 eV/f.u.) of wurtzite AlN [14–16], which results in the polarization switching with a lower electric field.

4. Conclusions

In this work, we have experimentally demonstrated the strained AlN with ferroelectric-like behavior on Si (111) substrate by using MOCVD growth method to modulate the strain in the film. The transition from linear to ferroelectric-like behavior depends on the AlN growth temperature as observed in the strained AlN films below 3 MV cm⁻¹. Because of the residual thermal strain, strained AlN grown at 500 °C suffers an in-plane compressive strain of 1.429% and an out-of-plane tensile strain of 1.015%, and the ferroelectric-like behavior in the proposed HfO₂/Al₂O₃/AlN/Si MIS capacitor may be induced by the strains in the AlN film with the contribution of dielectric insulation from the HfO₂/Al₂O₃ layer.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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