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Self-limiting low-temperature growth of crystalline AlN thin films by plasma-enhanced atomic layer deposition

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ABSTRACT

We report on the self-limiting growth and characterization of aluminum nitride (AlN) thin films. AlN films were deposited by plasma-enhanced atomic layer deposition on various substrates using trimethylaluminum (TMA) and ammonia (NH_3). At 185 °C, deposition rate saturated for TMA and NH_3 doses starting from 0.05 and 40 s, respectively. Saturative surface reactions between TMA and NH_3 resulted in a constant growth rate of ~0.86 Å/cycle from 100 to 200 °C. Within this temperature range, film thickness increased linearly with the number of deposition cycles. At higher temperatures (≥225 °C) deposition rate increased with temperature. Chemical composition and bonding states of the films deposited at 185 °C were investigated by X-ray photoelectron spectroscopy. High resolution Al 2p and N 1s spectra confirmed the presence of AlN with peaks located at 73.02 and 396.07 eV, respectively. Films deposited at 185 °C were polycrystalline with a hexagonal wurtzite structure regardless of the substrate selection as determined by grazing incidence X-ray diffraction. High-resolution transmission electron microscopy images of the AlN thin films deposited on Si (100) and glass substrates revealed a microstructure consisting of nanometer sized crystallites. Films exhibited an optical band edge at ~5.8 eV and an optical transmittance of >95% in the visible region of the spectrum.

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1. Introduction

Aluminum nitride (AlN) attracts considerable interest since it offers a unique combination of material properties including wide and direct band gap of 6.2 eV, good dielectric properties, piezoelectric response, high thermal conductivity, as well as stability and low expansion at high temperatures [1]. When processed in the form of thin film, AlN finds applications as buffer layers for the growth of high quality materials [2,3], insulators for thin film transistors [4], interfacial layers for metal oxide semiconductor field effect transistors [5,6] and high-electron mobility transistors [7], surface passivation layers for surface channel field effect transistors [8] and sensitive GaAs structures [9], transparent substrates [10] and active layers [11] for extreme UV light-emitting diodes and photodetectors. AlN films are also used in the fabrication of surface acoustic wave devices [12], mechanical resonators [13], piezoelectric transducers [14], and gas sensors [15].

Metal-organic chemical vapor deposition (MOCVD) [16], molecular beam epitaxy (MBE) [17], pulsed laser deposition [18], and sputtering [19] are the most common techniques employed for the growth of AlN. Although MOCVD offers the most efficient process due to its ability to deposit high quality materials with significant growth rates, deposition of AlN requires high temperatures (>1000 °C) [16]. Use of temperature-sensitive device layers (e.g. In-rich $In_xGa_{1-x}N$ [20]) and substrates (e.g. flexible polymeric substrates), therefore, necessitates the adaptation of low-temperature growth methods.

Atomic layer deposition (ALD) is a special type of low-temperature chemical vapor deposition, in which the substrate surface is exposed to sequential pulses of two or more precursors separated by purging periods [21,22]. Unless decomposition of the precursor occurs, each pulse leads to surface reactions that terminate after the adsorption of a single monolayer. When compared to other low-temperature methods, ALD stands out with its self-limiting growth mechanism, which enables the deposition of highly conformal thin films with sub-nanometer thickness control.

AlN growth by ALD has been studied by several research groups [23–29]. Lee et al. [23] reported plasma-enhanced ALD (PEALD) of AlN at 350 °C using aluminum chloride ($AlCl_3$) and NH_3/H_2 plasma. Growth rate of this process saturated at ~0.42 Å/cycle, resulting with films composed of microcrystallites of wurtzite (100) in an amorphous AlN matrix [24]. Thermal [25,26], plasma-enhanced [26], and UV-assisted [27] ALD of AlN using trimethylaluminum (TMA) and ammonia (NH_3) have been studied within the temperature ranges of 320–470, 250–470, and 240–370 °C, respectively — however, no self-limiting growth behavior was observed. This was explained by Riihela et al. [25] by the fact that surface reactions between TMA and NH_3 occur with reasonable rates only at temperatures where TMA self-decomposition takes place. Recently, Bosund et al. [28]

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used the same precursors to produce AlN films by PEALD at temperatures ranging from 100 to 300 °C. Films deposited in their study were amorphous except for the one grown at 300 °C. Kim et al. [29], on the other hand, have reported remote plasma ALD of amorphous AlN thin films using TMA and N_2/H_2 plasma. In their study, growth rate saturated at ~1.25 Å/cycle within the range of 100–400 °C and then decreased with increasing temperature.

Here we report on the self-limiting growth of crystalline AlN thin films at low temperatures by PEALD using TMA and NH_3 as the aluminum and nitrogen precursors, respectively. Process parameters including TMA pulse time, NH_3 flow rate and duration, purge time, deposition temperature, and plasma power were investigated. Structural, optical and surface characterization results of the films deposited at 185 °C are also presented.

2. Experimental details

AlN thin films were deposited on pre-cleaned substrates at temperatures ranging from 100 to 500 °C. Depositions were carried out in a Fiji F200-LL ALD reactor (Cambridge Nanotech) with a base pressure of 30 Pa, using Ar as the carrier gas. Unless stated otherwise, NH_3 plasma flow rate and power were 50 sccm and 300 W, respectively. System was purged for 10 s after each precursor exposure. Prior to depositions, Si (100), Si (111), c-plane sapphire, MOCVD-grown GaN on c-plane sapphire, and glass (Pyrex) substrates were cleaned by sequential ultrasonic agitation in 2-propanol, acetone, methanol, and deionized (DI) water. Solvent cleaned silicon substrates were further dipped into dilute hydrofluoric acid solution for ~1 min, then rinsed with DI water and dried with N_2.

Ellipsometric spectra of the films were recorded in the wavelength range of 300–1000 nm at three angles of incidence (65°, 70°, and 75°) by using a variable angle spectroscopic ellipsometer (J.A. Woollam). Optical constants of the ~33 nm thick AlN thin films deposited by thermal ALD at 450 °C and PEALD at 185 °C were modeled by the Cauchy dispersion function, and used for the estimation of ALD- and PEALD-grown film thicknesses, respectively. Measurement results were confirmed by cross-sectional transmission electron microscopy (TEM). Deposition rates were calculated by dividing film thicknesses to the number of ALD cycles.

Chemical composition and bonding states of the films were determined by X-ray photoelectron spectroscopy (XPS) using Thermo Scientific K-Alpha spectrometer with a monochromatized Al Kα X-ray source. Sputter depth profiling was carried out with a beam of Ar ions having an acceleration voltage and spot size of 1 kV and 400 μm, respectively. Grazing-incidence X-ray diffraction (GIXRD) measurements were performed in a PanAnalytical X'Pert PRO MRD diffractometer using Cu Kα radiation. FEI Tecnai G2 F30 transmission electron microscope at an operating voltage of 300 kV was used for the imaging of samples prepared by FEI Nova 600i Nanolab focused ion beam (FIB) system. Samples were prepared at an acceleration voltage of 30 kV, using various beam currents ranging from 50 pA to 21 nA. Damage layer was removed by FIB milling at beam voltages of 5 and 2 kV. An atomic force microscope (AFM, Asylum Research MFP-3D) operating in the contact mode was used to reveal surface morphology of the AlN thin films. Room temperature transmission and absorption measurements were performed with a UV–VIS spectrophotometer (Varian Cary 100).

3. Results and discussion

Thermal ALD experiments were carried out within the temperature range of 300–500 °C, where one cycle consisted of 0.5 s TMA pulse/10 s purge/7.5 s (200 sccm) NH_3 flow/10 s purge. Fig. 1(a) shows the deposition rates of AlN thin films at different growth temperatures. Although deposition rates of 0.07 and 0.14 Å/cycle were estimated for 300 and 350 °C, respectively, XPS studies have revealed that there is no AlN deposition at 300 °C. For 350 °C, on the other hand, only trace amount of AlN was detected. These results indicate that deposition of AlN with practical rates starts at 400 °C with 0.8 Å/cycle. For higher temperatures, growth rate increased with temperature and reached a value of 2 Å/cycle at 500 °C. Deposition rate as a function of precursor doses is also given in Fig. 1(a). TMA pulse time and NH_3 flow duration (flow rate = 150 sccm) were increased simultaneously from (0.1 s, 1 s) to (2 s, 20 s) at 450 °C. Deposition rate increased with the increasing amount of precursors. Saturation was not observed neither as a function of the temperature nor the precursor doses. Fig. 1(b) shows the effect of purge time on deposition rate at 450 °C. Deposition rate decreased from 0.95 to 0.79 Å/cycle as the purge time increased from 2 to 10 s, and remained constant at this value for purge times up to 40 s. Purge time of 10 s used in thermal ALD experiments therefore avoids overlapping of the precursor pulses and does not have any effect on the saturation behavior. These results are in agreement with those reported in the literature [25,26], where no self-limiting growth behavior was observed due to the fact that surface reactions between TMA and NH_3 occur at temperatures where TMA self-decomposition takes place. Self-limiting growth of AlN is therefore not possible with thermal ALD using TMA and NH_3 precursors. In order to achieve self-limiting surface reactions, TMA (i.e., $Al(CH_3)_3$) self-decomposition must be avoided by the use of lower deposition temperatures. In this respect, high temperatures needed for the NH_3 reaction can be lowered by using a plasma-assisted process.

Saturation curves of aluminum and nitrogen precursors obtained by PEALD at 185 °C are shown in Fig. 2. For the TMA saturation curve, NH_3 flow rate, duration, and plasma power were kept constant at 50 sccm, 20 s, and 300 W, respectively. Deposition rate increased

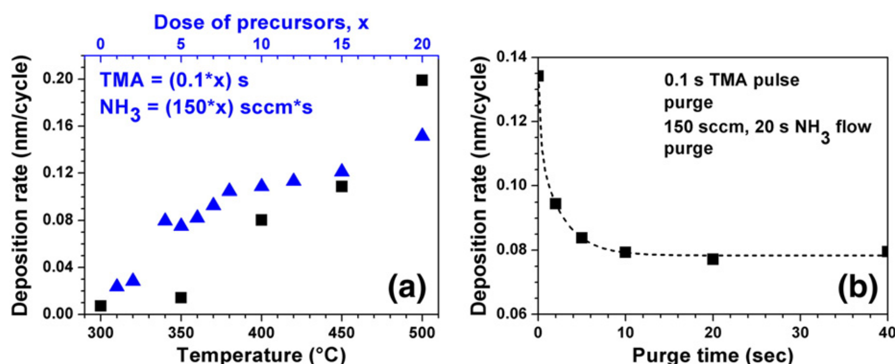

Fig. 1. Thermal ALD experiments, where no self-limiting growth behavior was observed. (a) Deposition rates of AlN thin films at different temperatures (■). Deposition rate as a function of TMA and NH_3 doses at 450 °C (▲). (b) Deposition rate as a function of purge time at 450 °C.

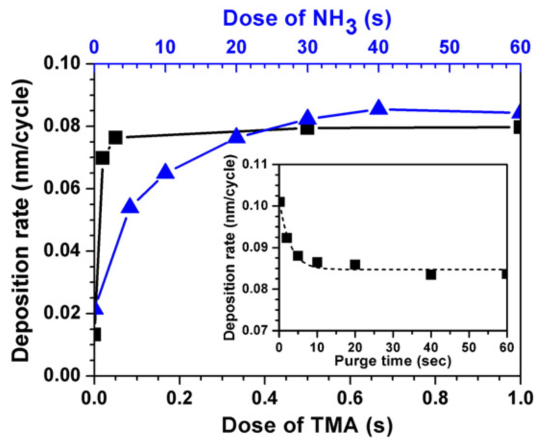

Fig. 2. Precursor saturation curves at 185 °C: NH_3 dose was kept constant at 20 s for the TMA saturation curve (■), and TMA dose was kept constant at 0.05 s for the NH_3 saturation curve (▲). (Inset) Deposition rate as a function of purge time: TMA dose and NH_3 flow duration were constant at 0.1 and 40 s, respectively.

with increasing TMA dose until 0.05 s, where growth rate saturated at ~0.76 Å/cycle. For the NH_3 saturation curve, TMA dose was constant at 0.05 s and NH_3 flow duration was varied with constant flow rate and plasma power (50 sccm, 300 W). Deposition rate increased with increasing NH_3 flow duration until maximum rate of ~0.86 Å/cycle was obtained at 40 s. Increasing NH_3 dose to 60 s did not increase the deposition rate. The effect of purge time on deposition rate was also studied at 185 °C (inset of Fig. 2). Deposition rate decreased from 1.01 to 0.86 Å/cycle as the purge time increased from 0.2 to 10 s. For longer purge times, deposition rate remained almost constant. Increased deposition rates at short purge times are due to insufficient purging, where one precursor is introduced into the reactor before the other one is completely removed. These results show that 10 s purge time is already a good estimation for 185 °C and does not require any further optimization.

Deposition rates of AlN thin films at different temperatures are given in Fig. 3(a). For these experiments, 100 cycle depositions were carried out with cycles consisting of 0.1 s TMA pulse/10 s purge/40 s (50 sccm, 300 W) NH_3 flow/10 s purge. Deposition rate remained constant at ~0.86 Å/cycle for temperatures up to 200 °C, and then increased with temperature. Temperature dependency of the deposition rate, observed within the range of 200–400 °C, may be due to the effect of temperature on the number and type of reactive sites present on the surface before and after the chemisorption, or the effect of temperature on the preferred reaction mechanisms [22]. Self-decomposition of TMA molecules may also be responsible from the increasing growth rate with temperature, however this is unlikely since the lowest temperature reported for the decomposition of TMA is ~300 °C [30]. In order to investigate this further, TMA and NH_3 saturation characteristics were studied at higher deposition temperatures. Fig. 3(b) shows TMA saturation curves at 185, 250, 350, and 450 °C. As the temperature increased, curves gradually deviated from the self-limiting behavior. NH_3 saturation curves at 185 and 450 °C are given in Fig. 3(c). As the temperature increased from 185 to 450 °C, deposition rate increased without any deviation from the self-limiting behavior. Assuming that the decomposition of TMA does not occur until 300 °C, the deviation observed in TMA saturation curve at 250 °C might be due to the increase in saturation TMA dose. TMA dose needed for the saturation of NH_3-terminated surface may increase with temperature if the number of reactive sites or sticking coefficient has temperature dependency [31].

Film thickness vs. number of deposition cycles is given in Fig. 4. Film thickness increases linearly with increasing number of cycles, confirming that the deposition rate is constant at 185 °C. Results also suggest that deposition starts immediately with the first cycle, without any incubation period. Inset of Fig. 4 shows the deposition rate as a function of plasma power at 185 °C. As the plasma power increased from 50 to 100 W, deposition rate increased from 0.66 to

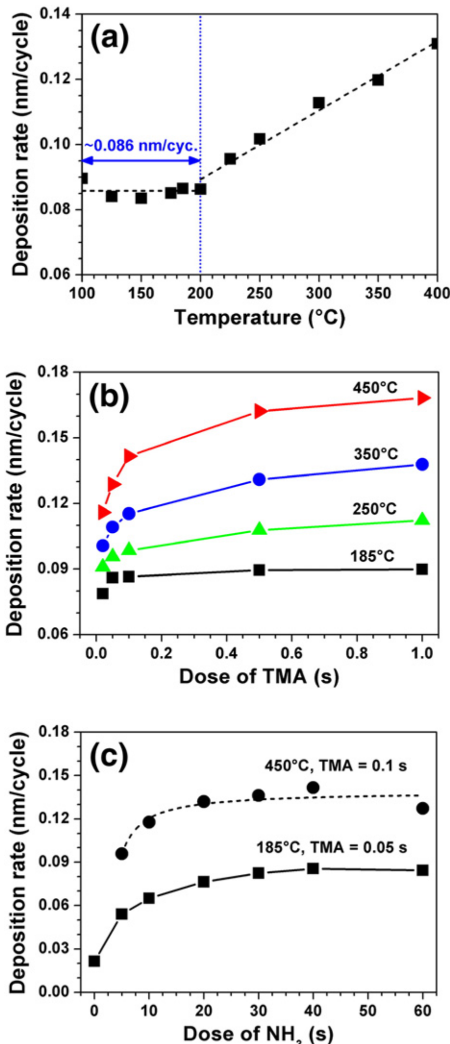

Fig. 3. (a) Deposition rates of AlN thin films at different temperatures. (b) TMA saturation curves at 185, 250, 350, and 450 °C, where NH_3 flow duration was kept constant at 40 s. (c) NH_3 saturation curves at 185 and 450 °C.

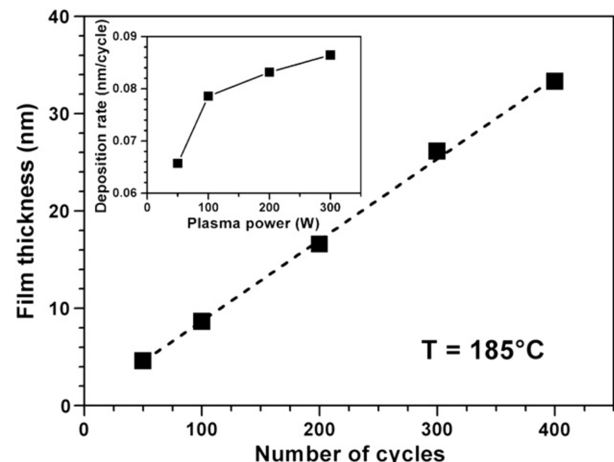

Fig. 4. Film thickness vs. number of deposition cycles. (Inset) Deposition rate of AlN as a function of plasma power.

0.79 Å/cycle. As the power further increased to 300 W deposition rate increased to 0.86 Å/cycle. These results indicate that dissociation efficiency of NH_3 and consequently deposition rate of AlN can be further increased by the use of higher plasma powers, i.e. > 300 W.

When compared to $AlCl_3$, use of TMA as the aluminum precursor resulted in higher deposition rates at lower temperatures. Moreover, saturation was achieved at much lower doses. Use of metal organic precursors (such as trimethyl metal compounds) instead of metal chlorides also avoids the formation of corrosive byproduct (i.e., hydrogen chloride) and hence increases the lifetime of deposition system.

Compositional characterization of ~100 nm thick AlN film deposited on Si (100) at 185 °C was carried out by using XPS. Survey scans detected peaks of aluminum, nitrogen, oxygen, and carbon. Fig. 5(a) is the compositional depth profile showing that atomic concentrations of Al and N are constant in the bulk film. Although O concentration is high (35 at.%) at the film surface, it decays rapidly within the first 120 s. At 600 s, concentrations of Al, N, and O are 59.8, 37.6, and 2.6 at.%, respectively. Carbon is detected only at the film surface and there are no C impurities in the bulk film, indicating that efficient removal of methyl groups from TMA was achieved by the use of NH_3 plasma.

O 1s high resolution XPS scan given in Fig. 5(b) represents the film surface, whereas Al 2p and N 1s scans given in Fig. 5(c) and (d) refer to bulk film (etch time = 150 s). O 1s scan was fitted by two peaks located at 529.96 and 531.46 eV, corresponding to Al–O–N [32] and Al–O [33] bonds, respectively. Results, which indicate oxidation at the film surface, are similar to those reported in the literature for air-exposed AlN thin films deposited by plasma source MBE [33]. Al 2p data was fitted with a single peak at 73.02 eV (Fig. 5(c)), which is assigned to the Al–N bond [33,34]. Additional information about the chemical bonding states in the films is provided by the N 1s spectrum that was fitted by two peaks as shown in Fig. 5(d). N 1s peak at 396.07 eV, which is assigned to the N–Al bond [35], confirms the presence of AlN. Peak at 398.0 eV (N–Al–O bond [33]), on the other hand, corresponds to the <5 at.% O present in the bulk film.

AlN thin films deposited at 185 °C were polycrystalline as determined by GIXRD. Fig. 6 shows the GIXRD pattern of ~100 nm thick AlN film on Si (100) substrate, where (100), (002), (101), (102), (110), (103), and (112) reflections of the hexagonal wurtzite phase were observed. Similar patterns were obtained for the films deposited on Si (111), c-plane sapphire, MOCVD-grown GaN on c-plane sapphire, and glass substrates, with only difference being the more pronounced intensity of (100) reflection in the case of sapphire and GaN/sapphire substrates.

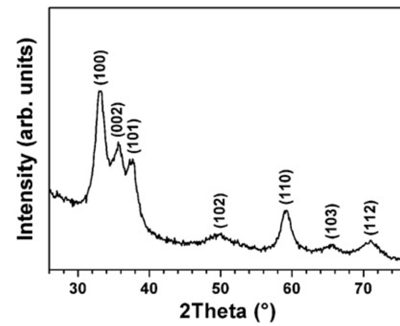

Fig. 6. GIXRD pattern of ~100 nm thick AlN thin film deposited on Si (100). Film is polycrystalline with a hexagonal wurtzite structure.

Fig. 7(a) is the cross-sectional TEM image of the AlN film deposited on Si (100) at 185 °C. Film thickness was measured as 95.9 nm from this image, which is in good agreement with the results obtained by ellipsometry. Fig. 7(b) and (c) are the high-resolution TEM (HR-TEM) images of the same sample, showing a film microstructure consisting of nanometer sized crystallites. Selected area electron diffraction (SAED) pattern of this film is also given in the inset of Fig. 7(b). In this pattern, reciprocal lattice points correspond to the diamond lattice of Si substrate. Diffraction rings, on the other hand, refer to AlN thin film and indicate a polycrystalline nature. HR-TEM image of the polycrystalline AlN thin film deposited on amorphous glass substrate is given in Fig. 7(d). SAED pattern of this sample consists of two polycrystalline diffraction rings (not shown here).

Fig. 8(a) shows the surface morphology of ~33 nm thick AlN film deposited on Si (100) substrate. Root-mean-square (rms) roughness

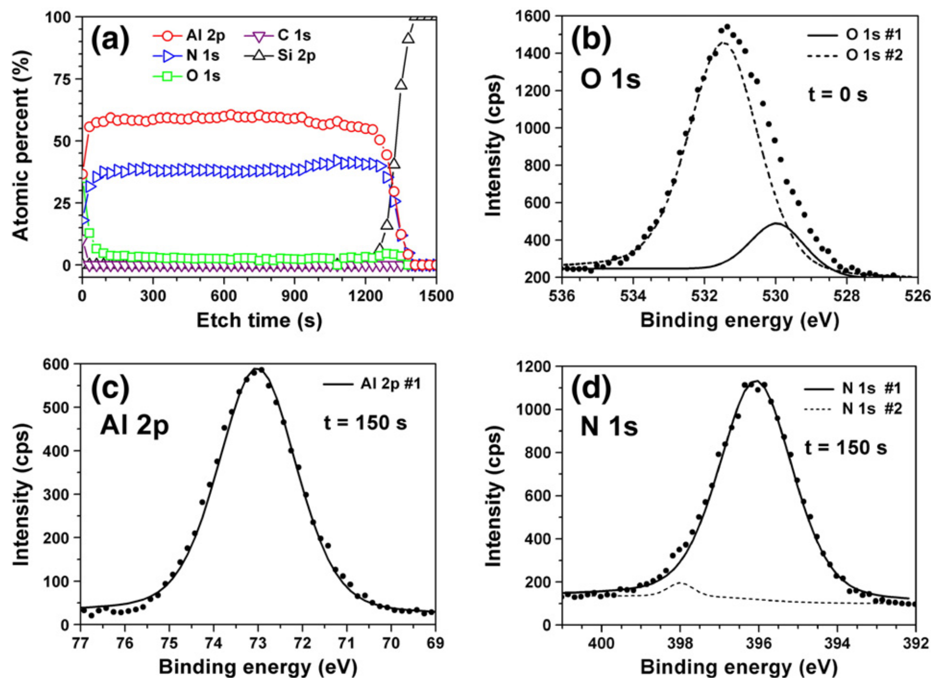

Fig. 5. (a) Compositional depth profile of ~100 nm thick AlN thin film. (b) O 1 s, (c) Al 2p, and (d) N 1 s high resolution XPS scans.

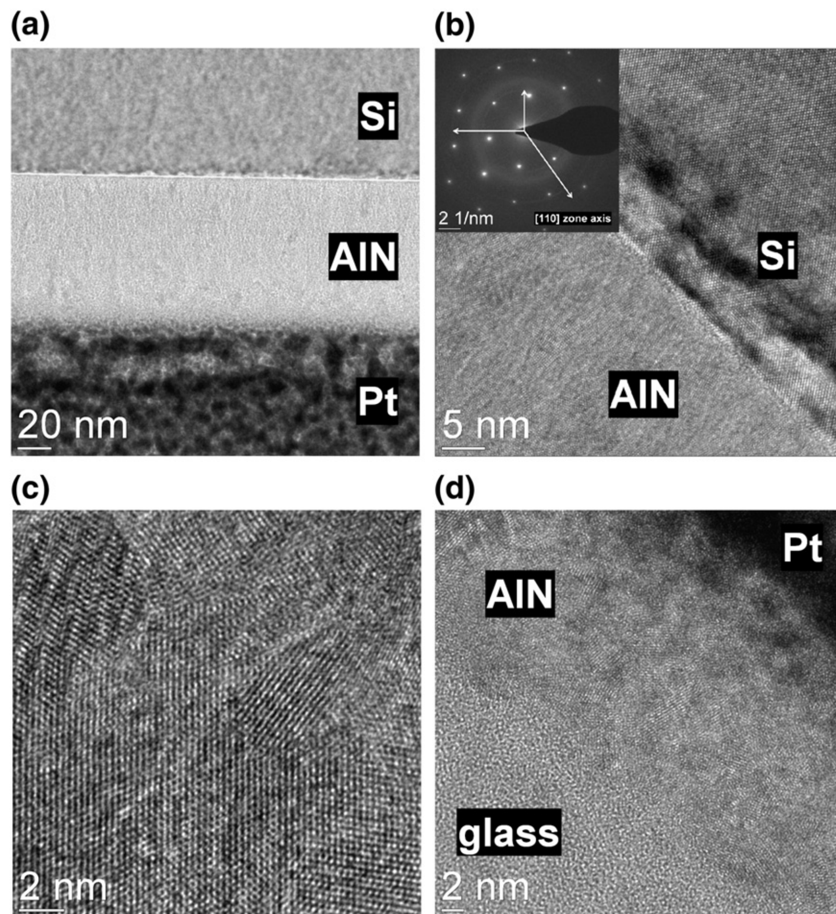

Fig. 7. (a) Cross-sectional TEM image of ~100 nm thick AlN thin film deposited at 185 °C on Si (100). (b, c) Cross-sectional HR-TEM images, and (inset) SAED pattern of the same sample. (d) Cross-sectional HR-TEM image of AlN thin film deposited on glass substrate.

of the film was measured as 0.32 nm from a 2 μm × 2 μm scan area. Rms roughness values of the AlN films deposited on GaN/sapphire (Fig. 8(b)) and sapphire substrates were 0.42 and 0.51 nm, respectively. AFM image revealing the surface morphology of ~100 nm thick AlN film deposited on Si (100) substrate is given in Fig. 8(c). As the film thickness increased from ~33 to ~100 nm, rms roughness increased from 0.32 to 1.25 nm.

Refractive index and extinction coefficient of AlN film deposited at 185 °C are shown in Fig. 9(a). Refractive index, which is 2.15 at 270 nm, decreases to 1.88 at 1000 nm. These values are in good agreement with those reported in the literature for polycrystalline AlN thin films [36]. Extinction coefficient (k), which is ~0.03 at 270 nm, decreases rapidly within the wavelength range of 270–335 nm. For higher wavelengths k is almost zero, indicating that films are transparent in this spectral region. Room temperature transmission spectrum, which is corrected for the substrate and spectrometer response characteristics, is given in Fig. 9(b) for ~100 nm thick AlN film deposited at 185 °C on double-side polished sapphire. Transmission increases with wavelength within the range of 185–350 nm, reaches to 95% at 350 nm and remains >95% until 3 μm (not shown here). A pronounced reduction of the transmission signal within 235–300 nm is also noteworthy. This absorption band, centered at ~255.5 nm (4.86 eV), is attributed to the oxygen- or vacancy-related defects [37]. Since AlN is a direct band gap material and absorption coefficient is directly proportional to the wavevector value, the square of the product of absorption vs. incident photon wavelength is shown in the inset of Fig. 9(b) in order to better determine the optical band edge (absorption edge) [38]. Experimental absorption spectrum of the sample shows that absorption decreases rapidly in the UV spectral range and becomes insignificant in the visible region. A straight line fitted through the measured data (dashed line shown in the inset of Fig. 9(b)) intersects the abscissa at ~5.8 eV, revealing the optical band edge of AlN thin films deposited by PEALD.

4. Conclusion

In this study, we have deposited crystalline AlN thin films by PEALD at temperatures as low as 100 °C. Constant growth rate of ~0.86 Å/cycle was observed from 100 to 200 °C. For higher temperatures (≥225 °C) deposition rate increased with temperature. At 185 °C, film thicknesses increased linearly with the number of deposition cycles and no incubation behavior was observed. AlN thin films deposited at 185 °C were polycrystalline with a hexagonal wurtzite structure regardless of the substrate selection as determined by GIXRD and HR-TEM. Films exhibited an optical band edge at ~5.8 eV and high transparency in the visible region of the spectrum.

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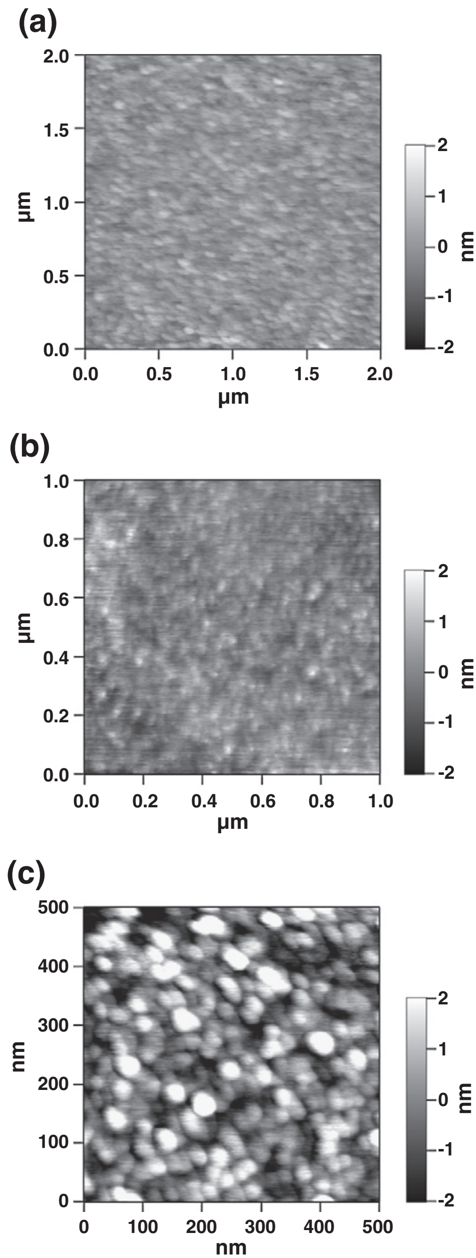

Fig. 8. Surface morphology of ~33 nm thick AlN thin films deposited at 185 °C on (a) Si (100), and (b) MOCVD-grown GaN on c-plane sapphire. (c) Surface morphology of ~100 nm thick AlN film deposited at 185 °C on Si (100).

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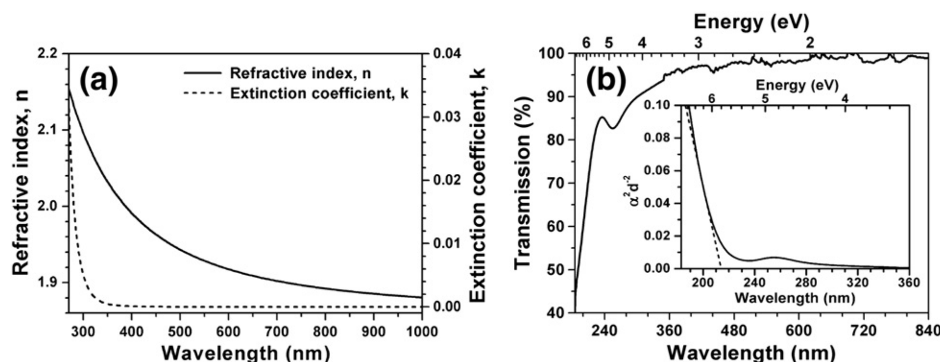

Fig. 9. (a) Optical constants (refractive index and extinction coefficient) of ~100 nm thick AlN film deposited on Si (100). (b) Optical transmission spectrum of the film deposited on double-side polished sapphire. (Inset) Absorption spectrum of the same sample.