Influence of the PALE growth temperature on quality of MOVPE grown AlN/Si (111)

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ABSTRACT

In the present study, the PALE-AlN (pulsed atomic layer epitaxy) epilayers were grown on the Si (111) substrates at different growth temperatures by metal organic vapor phase epitaxy (MOVPE) tecnique. The oxygen (O) and silicon (Si) concentrations of grown PALE-AlN epilayers and interface between epilayer and substrate were investigated by secondary ion mass spectroscopy (SIMS). It was observed that O and Si concentration change with growth temperature of epilayers as well as the interface significantly. HRXRD (high-resolution x-ray diffraction) analyses showed that the highest growth temperature results with the lowest full width at half maximum (FWHM) value for both ω scans. Scanning electron microscope (SEM) and atomic force microscopy (AFM) analyses indicated that relatively low growth temperature grown samples gave rise to 2D-like growth mode with openings while increased growth temperature resulted in change the growth mode to a columnar mode with increasing V-shape pits because of the increasing diffusion coefficient of O impurities and Si atoms in AlN epilayers.

Keywords: AlN, MOVPE, diffusion, pulsed atomic layer epitaxy.

Introduction

III-nitride semiconductors have been taking remarkable attention due to their wide bandgap energy and, their high performance optoelectronic applications [1-3]. AlN, one of the III-nitride semiconductor materials, has the widest energy bandgap in the III-nitride semiconductors with an effective mechanical strength, and high chemical and thermal stability [4-8]. These extraordinary properties provide wide application areas in optoelectronic and electronic devices such as UV photodetectors-light emitting diodes, quantum cascade lasers, missile-warning systems etc. [9-18]. Moreover, AlN buffer layer have played a crucial role for GaN/Si high-power/high-frequency devices in recent years in order to be used in electrical vehicle, wind tribunes, fast-chargers etc. Since it is not possible to grow GaN directly on Si substrate due to the dissolving of silicon by gallium, commonly known as melt back etching [19-24], it is by passed effectively via the use of AlN buffer layers. But, several important inquiries stay unanswered about the initial phases of buffer AlN growth by MOVPE and how pre-growth exposure and other growth parameters affect film quality. All mentioned devices above require high-quality AlN templates and there are two options to have high quality films; homoepitaxy on AlN substrate and heteroepitaxy on Si and Al_2O_3 substrates. Apparently, the most suitable option for the development of high-efficiency, long-lifetime and high-performance devices is homoepitaxial growth on AlN substrates, but they are not generally preferred due to the limited size and extremely high cost [25, 26]. One of the sustainable and affordable option for heteroepitaxial growth is to use Si as a substrate because of its scalability, low cost, and the opportunity to combine III-nitride devices on a single wafer with silicon microelectronics [1, 27, 28]. However, there are many challenges in epitaxial growth of

AlN on Si such as high thermal expansion coefficient difference, high lattice-mismatch (19 %), Si diffusion to the epilayer, low mobility of Al adatoms, and parasitic reaction between precursors etc. [19, 29-32]. There are some reports which demonstrate that it is inevitable to supply the TMAl (trimethylaluminum) to the reactor first to prevent the possible amorphous layers which occur at the interface [30, 33, 34]. Li et al. and Radtke et al. designed and investigated the effect of a low temperature nucleation layer to grow high quality AIN epitaxial film on Si substrate which prevents the formation SiN_x amorphous layer [2, 35]. It was suggested PALE growth of AlN to avoid the parasitic reactions between TMAl and NH_3 precursors [29, 30, 36]. Furthermore, it was investigated the effect of removing organic wastes, oxides and impurities on the silicon surface by applying different cleaning procedures [1]. It was also been shown that TMAI pre-seeding duration had a very important role on morphology and dislocation density of AIN [37]. Chernykh et al. reported a study about the growth of low temperature (LT) AlN nucleation layer with various conditions in the presence of TMAl preflow. They investigated the effect of growth conditions on high quality AIN film. They suggested that the rate of the substrate coating by TMAl preflow was a very important factor on the crystal quality [38]. Tran et al. proved that AlN quality was improved by using NH_3 pulsed flow and epitaxial lateral overgrowth technique [16]. Çörekçi et al. emphasized that the flow rate and layer thickness were crucial parameters and they stated that thicker AlN films enhanced the crystallinity of the asymmetric $(10\bar{1}2)$ plane [39]. Another important growth parameter is growth temperature. Chen et al studied the influence of the growth temperature of AlN nucleation layer to obtain high quality AlN template. They explained the growth evolution of AlN nucleation layer at different temperatures and shown the influence of AlN nucleation layer

temperature on the crystalline quality of AlN template [40]. Chang et all observed that the temperature of AlN nucleation layer on Si substrate significantly affects the crystalline quality and surface morphology [41]. There are many studies in the literature on AlN growth parameters on Si to increase the crystal, optical, electrical, etc. properties of AlN epilayers. But the interface effects of AlN/Si structures are very complicated and there are still many unanswered questions.

It is customary to grow high temperature (HT) AlN layer on LT-AlN nucleation layer to obtain better crystal quality using MOVPE. In this study, in contrast to the effect of nucleation layer temperature, the effect of HT-AlN layers with different growth temperatures on crystal, structural, morphological, elemental, and interfacial properties of PALE-AlN/Si (111) structure is investigated. HRXRD, scanning electron microscopy (SEM), AFM, transmission electron microscope (TEM), Raman spectroscopy, SIMS techniques are used to analyze the properties of grown AlN/Si (111) structures.

Experimental

The epitaxial AlN layers were grown by AIXTRON 200/4 RF-S horizontal flow MOVPE reactor on Si (111) substrate. Aluminum and nitrogen sources were obtained from TMAl and ammonia (NH_3), respectively. Si substrate needs to be cleaned because cleaning the substrate surface from impurities such as organic contaminants, foreign microparticles metal ions and anions etc. which inevitably lead to the formation of structural defects in growing films allows high quality material [42]. Before the growths, RCA (Radio Corporation of America) cleaning procedure was applied to all Si (111) substrates. After the RCA chemical cleaning process, an in situ high temperature (~1100 ºC) desorption was applied for 10 minutes to remove the final possible remaining oxide layer on the Si (111)

substrate. The reactor was then cooled down to 850 ºC and the growth was initiated by first feeding TMAl into the reactor for 5 seconds before introducing NH_3; this technique assures that an aluminum-rich layer is initially nucleated, preventing the formation of the amorphous SiN_x interlayer and involving an Al face growth front [30]. It was demonstrated that the duration and flow amount of TMAl precursor was crucial to keep step flow growth mode and to get a high-quality AlN template before the main AlN layer [14, 38, 43-45]. Following, a thick AlN nucleation layer (~20-30 nm) was grown at a low temperature. Then the PALE method was employed at different high pyrometer temperatures (1365 ºC, 1410 ºC, and 1450 ºC) to grow three AlN samples (HT-AlN). These HT-AlN samples were labeled as Sample A, Sample B, and Sample C. The PALE technique to grow HT-AlN epilayers described in detail in our previous study [46]. The parameters for growing HT-AlN epilayers; the thicknesses were 250 nm, the growth rate was around ~1 nm per pulse and the reactor pressure was 50 mbar.

Different characterization measurements (HRXRD-Rigaku SmartLab, AFM-Hitachi AFM 5100N in dynamic force mode, SEM-Zeiss Sigma 300, TEM-FEI Tecnai Talos FEG/TEM-EAG Labs, SIMS-EAG Labs, and Raman spectroscopy-Witec) were carried out to investigate the impact of growth temperature on properties (crystal, structural, and surface quality, interface sharpness and impurity concentration) of Sample A, Sample B, and Sample C.

Results and Discussion

The quantitative depth profiles of impurity concentrations of grown AlN epilayers have been investigated by SIMS measurements. Figure 1 indicates that the O and Si concentration change through depth. It can be seen that there is a high level of O in all

samples, which results in mass interference for Si, so the Si levels plotted in Figure 1 are an upper limit and the actual concentrations are lower. It is shown that the O impurity originating from SiO_2 which occurs by oxidizing Si substrate [47]. Even though the substrate is cleaned by RCA procedure just before the growth it is thought that the Si substrate oxidizes instantly and it is known that the AlN surface is sensitive to oxidation depending on the environment [48, 49]. It is clearly seen from Figure 1 that diffusivity and the level of O in the epilayer increase with increasing the growth temperature. Also the growth conditions may change the growth mode and thus surface terminations which result in with different impurities such as O, C etc. [50]. It is thought that surface roughness and defects may cause tailing and profile broadening by increasing growth temperature up to 1450 ºC (Sample C). As it is shown that the level of O impurity decreases at the interface region while it increases inside of the AlN because of its high diffusion coefficient at higher temperature. Figure 1 demonstrates the Si distribution through the AlN epilayer and Si substrate. The Si concentrations are the same and the values vary around the AlN/Si interface. It is indicated that Si diffusion into the AlN epilayers at higher growth temperatures is an important issue about the electronic applications of AlN/Si structures [1, 51]. Moreover, the increase in growth temperature changes the Si concentration in the epilayer and interface because of the increasing diffusion coefficient of Si atoms in the AlN epilayer. It is considered that the step-like behavior near the substrate and epilayer interface is the result of defects at the interface. Sample C has the lowest step-like behavior due to the reduced O concentration at the interface while Sample A has the highest one. On the other hand, it will be shown that the increase in the Si and O impurities in the AlN epilayer is going to change the growth behavior.

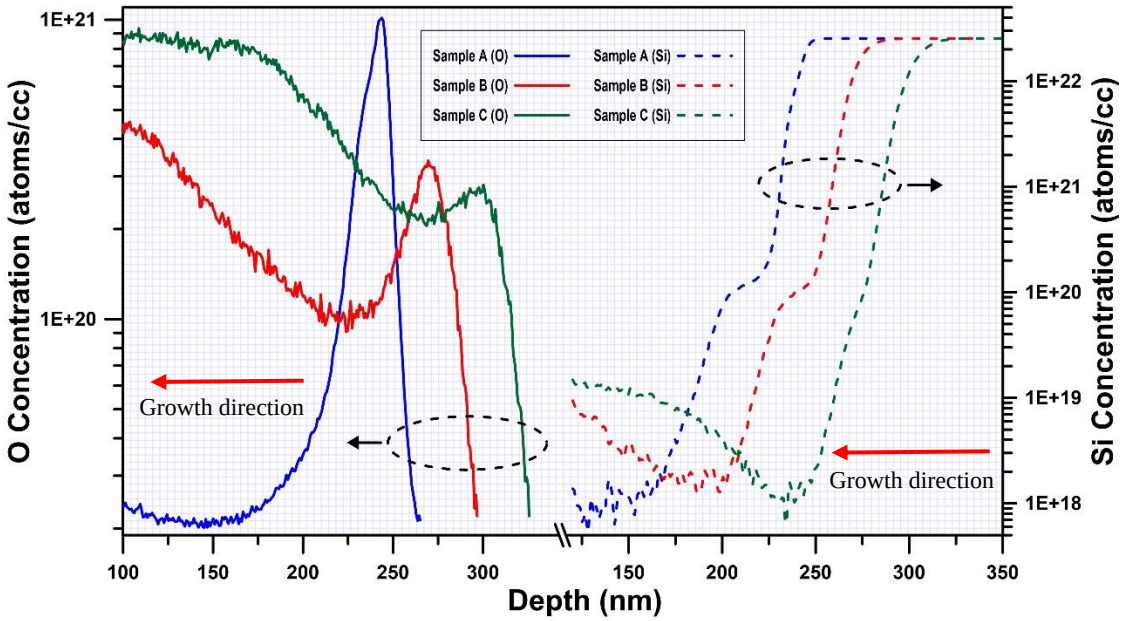


Figure 1. The O and Si concentrations change with depth for Sample A, Sample B, and Sample C. Curves shifted horizontally for clarity.

Apart from the impurity profile property, the effect of growth temperature on the crystalline quality of Sample A, Sample B, and Sample C are studied by measuring (0002) symmetric ω scan (Figure 2) using HRXRD. These results imply that not only the AlN epitaxial films are grown on the Si substrate, but also an out-of-plane epitaxial relationship of AlN (0002) and Si (111) is obtained [52]. It is known that the FWHM values measured by HRXRD are extensively used to calculate the dislocations in AlN epitaxial film [53]. The FWHM values for Sample A, B and C are calculated as 8593, 7272 and 5523 arcsec, respectively. The screw-type dislocation density associates with the FWHM of AlN (0002) XRD ω scan. The dislocation density of AlN epitaxial film is calculated by the following equation [8, 54, 55]:

$$D_{screw} = \frac{\beta_{0002}^2}{4.35\, b_{screw}^2}$$

where D_{screw} is the screw-type dislocation density, β_{0002} is the FWHM measured by HRXRD ω scan, and b_{screw} is the Burgers vector length (b_{screw} = 0.4980 nm). D_{screw} for Sample A, B, and C are calculated to be 1.57E+11, 1.14E+11, and 6.35E+10 cm^{-2}, respectively. HRXRD analyses show that Sample C has the lowest D_{screw} and FWHM value which proves that there is a significant improvement effect of growth temperature on AlN crystalline quality. It was shown in the literature that the growth mechanism of epilayers depends on the generalized kinetic coefficients, which are functions of not only the diffusion coefficients of the components (atoms), but also the concentrations of the components and their free path length. It is believed that the increasing growth temperature enhances the given energy to the Al adatoms and their migration capability with the help of increased free path length on the surface to find the best site for growing epitaxially [29, 56-61]. These results obtained from HRXRD measurements are compatible with studies which indicated the positive effect of growth temperature of AlN on crystalline quality [62, 63].

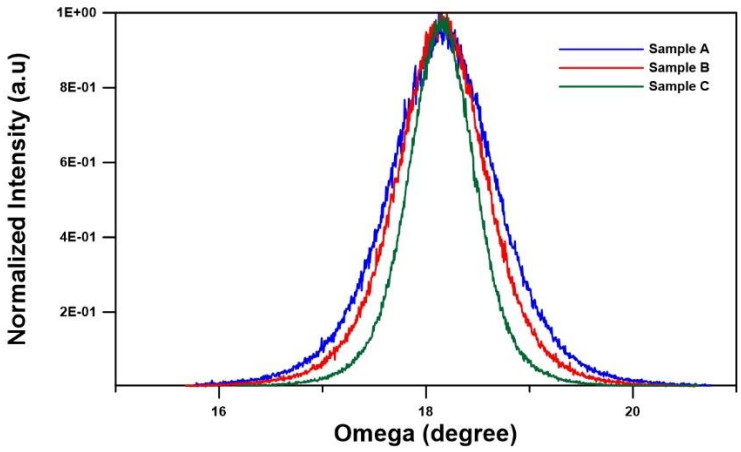


Figure 2. HRXRD symmetric (0002) ω scan for Sample A, Sample B, and Sample C

Further structural analysis was performed with Raman spectroscopy on PALE grown AlN layers. Figure 3 (a) shows Raman spectrum on each Sample A, B, and C. As shown from the figure, all the three samples show vibration belong to the AlN at 617.5, ~648.0, ~670.0 and 880.0 cm^{-1} wavenumbers which correspond to A_1(TO), E_2(high), E_1(TO) and A_1(LO) modes. E_1(TO) mode is shown as a shoulder right at the higher wavenumber side of the E_2(high) mode. This affects to find exact wavenumber of E_1(TO) mode. From these modes, E_2(high) generally is used to determine the structural quality by evaluating shift of its wavenumber compared to the bulk AlN value of 657 cm^{-1} [64], and the FWHM value of the peak. It seems that the wavenumber of the E_2(high) shifts almost 10 cm^{-1} which corresponds to existence of the tensile strain on the grown AlN layers [65]. The wavenumber of the E_2(high) mode is 646.6 cm^{-1} for the Sample A, while it is 647.8 cm^{-1} for Sample B indicating that the larger tensile strain appears in Sample A. FWHM of E_2(high) mode values are almost 6 cm^{-1} confirms relatively high structural quality, as the 3 cm^{-1} wavenumber of bulk value is considered. Figure 3 (b), (c) and (d) show the Raman mapping of the Sample A, B, and C, respectively. The images were obtained by taking care of the intensity of the E_2(high) mode of the PALE grown AlN layers. Brighter color in the images indicates the larger intensity of the mode, while darker places showing the very low or no intensity observed in the E_2(high) mode. One of the first observation from images is the darker lines in the Sample A and B. These are edge cracks as observed similar to the SEM images for these samples. Also, the circle dark points are seen in these two samples. These are probably the dislocations formed on these samples which are also observable from both SEM and AFM images. On the other hand, no cracks and also no clear circle dark points are observable for Sample C, even though there are larger amount of darker

places in the image compared to that of Sample A and B. This might indicate that there is no coalescence between the grown structures showing columnar growth similar to the observed SEM and AFM data.

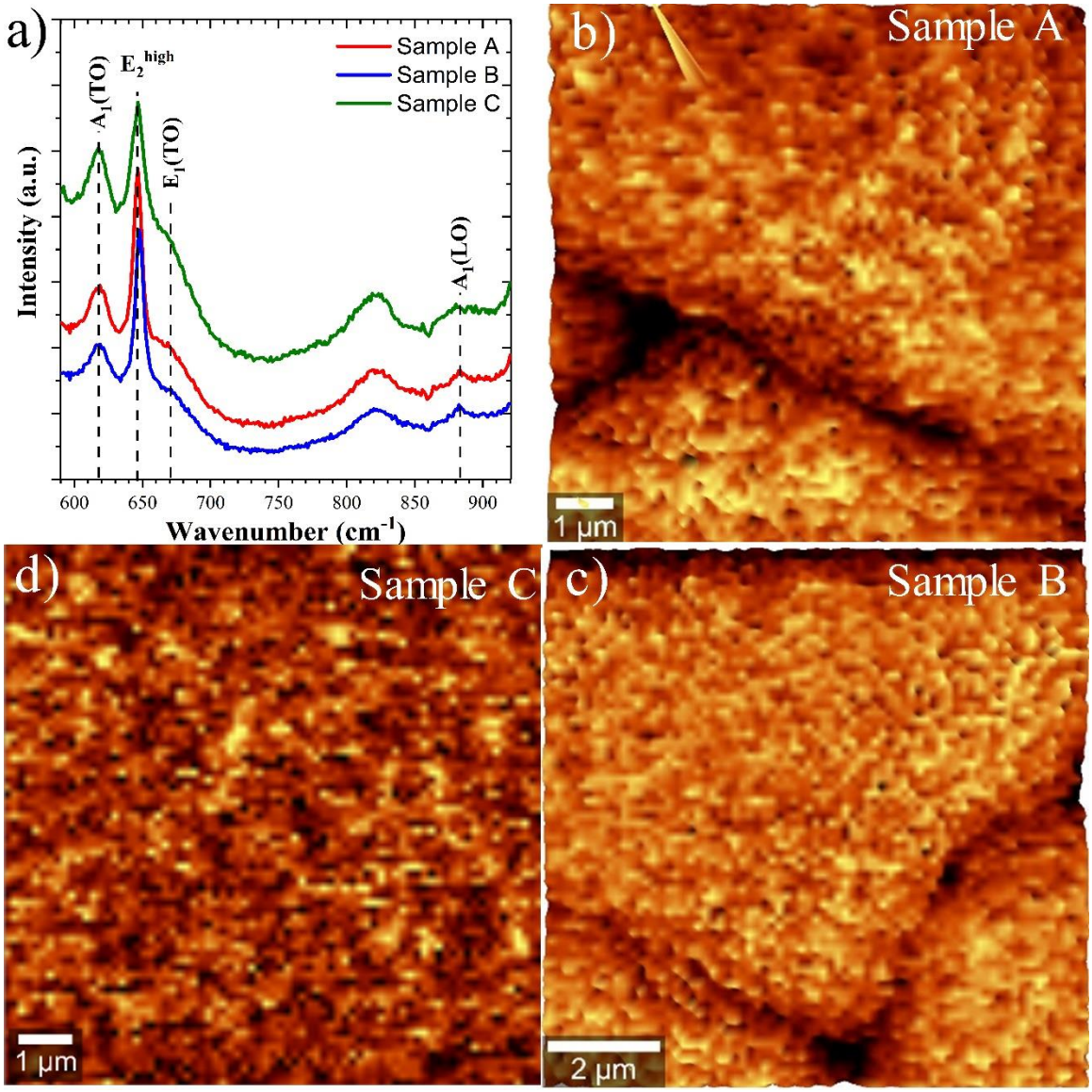


Figure 3. a) Raman spectra of Samples A, B, and C. Raman mapping of the intensity of the E_2(high) mode of AlN for **b)** Sample A, **c)** Sample B and **d)** Sample C.

SEM and AFM measurements were carried out to examine in detail how the growth temperature affects the surface morphology of AlN epilayers grown on the Si (111) substrate. SEM images recorded for AlN epilayers grown on the Si (111) substrate at

different grown temperatures (Sample A, Sample B, and Sample C) are revealed in Figure 4. When large area SEM images of Sample A and Sample B were examined in Figure 4 (a) and (b), it was observed that edged cracks were formed on the surface of the AlN layers grown on the Si (111) substrate. The causes of cracks that occur have been investigated in the literature [29, 30, 66] and it has been reported that these cracks can be given rise to by the thermal expansion coefficient difference between Si substrate and AlN epilayer [6]. Unlike other samples, it is obviously seen in Figure 4 (c) that Sample C has a crack-free surface. The probable cause of this situation is that because of the growth of Sample C in the columnar structure, the stress in the structure have been partially decreased, thus the cracks observed on the large area surface are reduced. Similar results can be observed in high resolution scanning electron microscopy (HR-SEM) images (Figure 4 (d), (e), and (f)). As can be seen from the Figure 4 (a) and 4 (b), dislocation related circle shapes are seen on the surface, the radius of those circles are larger in sample compared to that of Sample A, while no clear dislocation created morphology appears in Figure 4 (d). A similar observation was reported by Abdullah et al. [67]. They stated the upper AlN film grown on the Si (111) substrate displays a columnar structure. It should be noted that the results obtained by SEM measurements show a good agreement with the results obtained from HRXRD analysis. AFM is a type of high-resolution scanning probe microscope that has a resolution that can be measured in fractions of a nanometer. AFM images of 2μm x 2μm area of all samples grown at different growth temperatures are indicated in Figure 4. AFM measurements show similar observations to HR-SEM images. As in HR-SEM images, Sample C has shown in the form of a columnar growth, while Sample A and Sample B have observed to form opening dislocations. The crater produced by the dislocation appears

on the surface is larger for Sample B and it is less dense compared to that of Sample A. Moreover, the root-mean-square (rms) surface roughness value of Sample A, Sample B, and Sample C was determined as 3.2, 5.3, and 14.0 nm, respectively. This large surface roughness might be due to the observed 3D columnar growth.

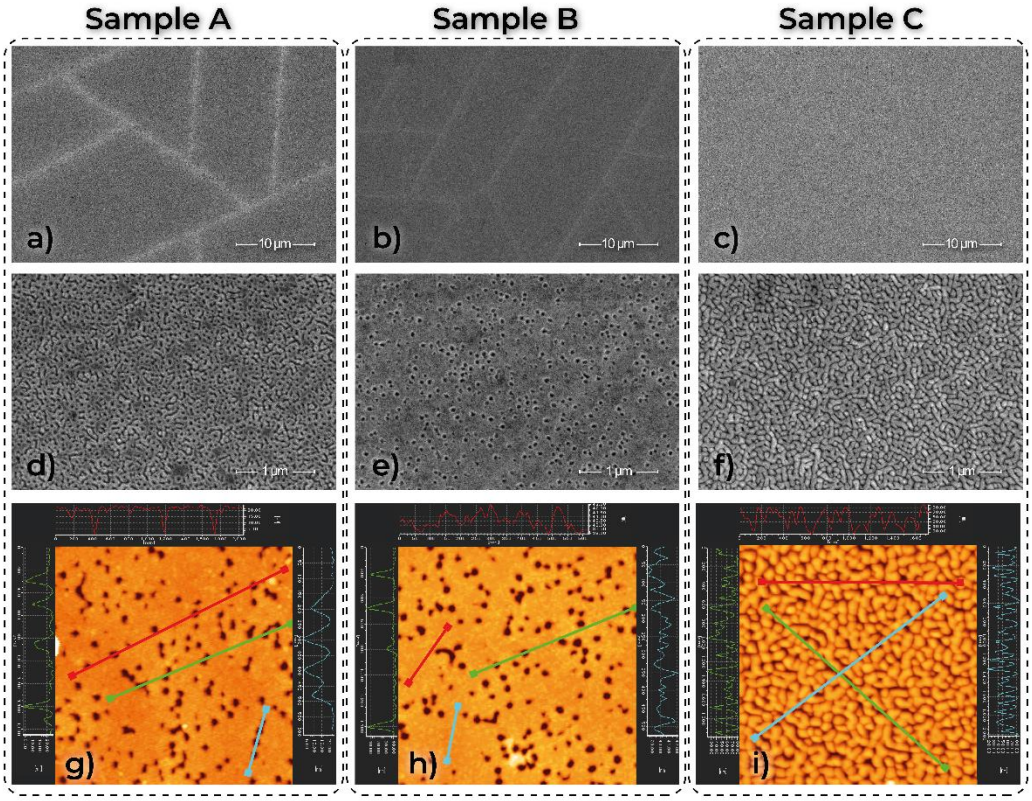


Figure 4. SEM (a-c), HR-SEM (d-f) and AFM (g-i) images of AlN epilayers grown on the Si (111) substrate at different grown temperatures

In order to further study the effect of growth temperature on interfacial and structural property of AlN epitaxial films grown on Si (111) substrate, cross-sectional TEM measurements (bright field TEM-BFTEM and high-resolution TEM-HRTEM) are employed. Figure 5 (a), (b), (c), and (d) show the BFTEM measurements of Sample A and Sample C. It is seen that there are some V-shape pits shown by white circles on the surface for both samples. The V-shape pits are generally created on the interface between Si

substrate and AlN epilayer then continues upwards to the surface. It is also believed that some of the V-shape pits are generated from the merging of AlN islands [28]. It is seen from the Figure 5 that our samples have V-shape pits and actually, these pits are determined to be kind of black dots and circles from the SEM and AFM measurements shown by Figure 4. It is demonstrated that when the PALE AlN growth temperature is increased, growth mode changes from 2D-like (Sample A) to columnar type (Sample C) and result in the increased V-shape pit density. Figure 5 (b) displays that there are more V-shape pits on the surface of Sample C. In our opinion, the increasing growth temperature may change the properties of low temperature AlN nucleation layer and cause diffusion of Si-O atoms as shown by SIMS measurements. It is believed that the increasing growth temperature increased the contact surface of AlN epilayer and Si substrate which result in columnar growth type. Also, it is calculated that the angles of V-pits for Sample C is around 60^{o} which corresponds to a semi-polar orientations [50]. The growth that continues on these planes results in smaller growth rates that growth on out-plane direction. As a result of these findings, it is obtained that the thickness or growth rate of Sample C is different when TEM and in-situ reflectance results are compared. There are dark and bright diffraction contrasts observed in the AlN epilayer and these regions are the results of highly distorted regions (grain boundaries) which are frequently generated at the interface. The contrast difference between distorted regions is a result of a slight tilt from one region to another. The large number of dislocations densely located thickness about 40 nm. There is less number of dislocations above the distorted region as the layer thickness increased. It is thought that the reason of the contrast difference is that the deviation from the Bragg diffraction condition by the distortions around the dislocations [68]. Figure 5 (e) and (f)

represent HRTEM images of Sample A and Sample C, respectively. As shown in HRTEM images, there is an abrupt cubic (Si substrate)-hexagonal (AlN epilayer) lattice interface which is an indication of coherent epitaxial growth [9]. There are ongoing debates on the characteristics of this interfacial layer in the literature [13, 17, 69]. It is obtained that, the thickness of the interfacial layer for both samples is around 1 nm.

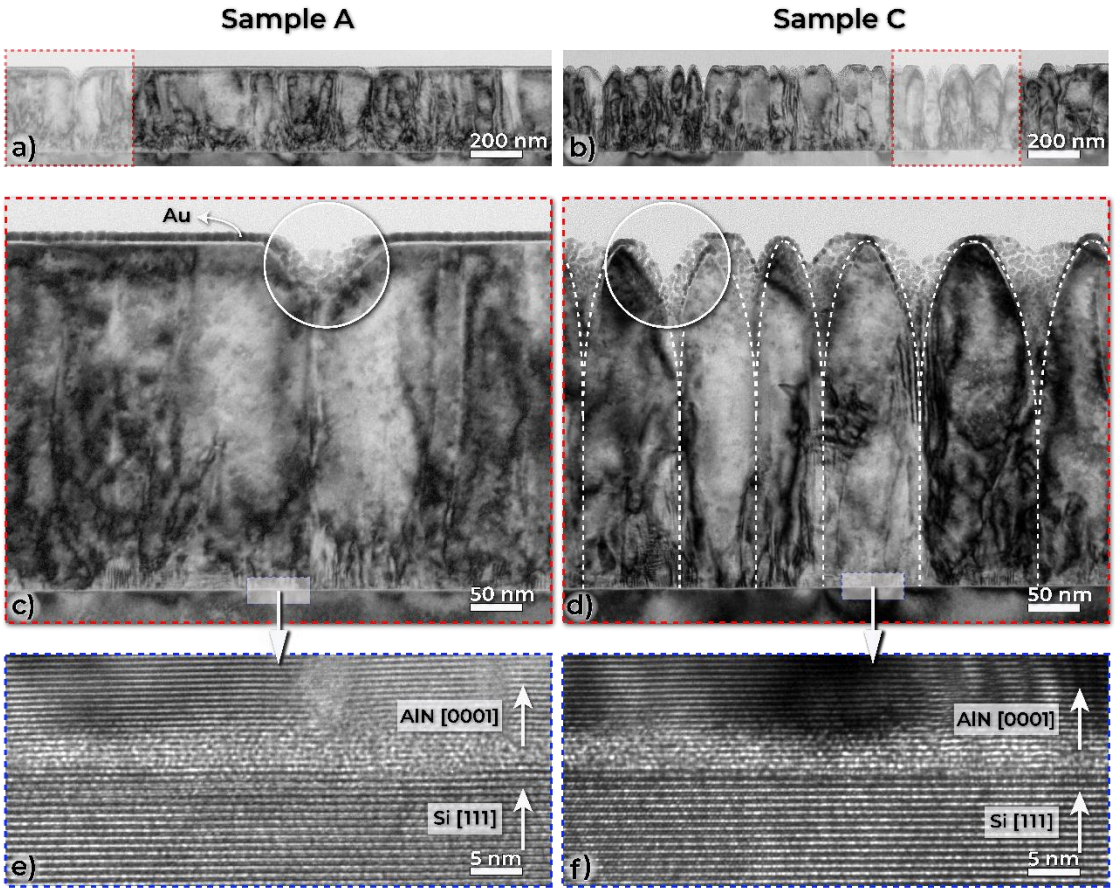


Figure 5. BFTEM (a-d) and HR-TEM (e, f) images of AlN epilayers grown on the Si (111) substrate at different grown temperatures.

Conclusions

As a conclusion, high quality AlN epilayers were grown on Si (111) substrates at various growth temperatures by using PALE via MOVPE. It was concluded from HRXRD, AFM and SEM measurements that the increasing growth temperature enhanced the crystalline

quality while it changed the growth mode from 2D-like to the columnar-type. This is mainly attributed to the presence of O impurities at the interfacial layer between the Si (111) substrate and AlN epilayer. It was also pointed out by TEM measurements that the increasing growth temperature may change the properties of low temperature grown thin AlN nucleation layer and enhanced the columnar growth resulted in V-shape pits on the surface. Additionally, it was proved that the V-shape pits changed the growth rate of AlN epilayer on different orientations that resulted in different thickness measurements results by TEM and in-situ reflectance (not shown here).

Acknowledgements

The authors acknowledge the usage of Nanophotonics Research and Application Center at Sivas Cumhuriyet University (CUNAM) facilities. This work is supported by the TUBITAK under project numbers 117F339, 118F425 and supported by the Scientific Research Project Fund of Sivas Cumhuriyet University under the project number M-772.

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