

Structural properties, crystal quality and growth modes of MOCVD-grown AlN with TMAI pretreatment of sapphire substrate

This content has been downloaded from IOPscience. Please scroll down to see the full text.

Download details:

IP Address: 109.171.137.210

This content was downloaded on 13/08/2017 at 08:00

Manuscript version: Accepted Manuscript

Sun et al

To cite this article before publication: Sun et al, 2017, J. Phys. D: Appl. Phys., at press:

<https://doi.org/10.1088/1361-6463/aa8503>

This Accepted Manuscript is: © 2017 IOP Publishing Ltd

During the embargo period (the 12 month period from the publication of the Version of Record of this article), the Accepted Manuscript is fully protected by copyright and cannot be reused or reposted elsewhere.

As the Version of Record of this article is going to be / has been published on a subscription basis, this Accepted Manuscript is available for reuse under a CC BY-NC-ND 3.0 licence after the 12 month embargo period.

After the embargo period, everyone is permitted to copy and redistribute this article for non-commercial purposes only, provided that they adhere to all the terms of the licence

<https://creativecommons.org/licences/by-nc-nd/3.0>

Although reasonable endeavours have been taken to obtain all necessary permissions from third parties to include their copyrighted content within this article, their full citation and copyright line may not be present in this Accepted Manuscript version. Before using any content from this article, please refer to the Version of Record on IOPscience once published for full citation and copyright details, as permission will likely be required. All third party content is fully copyright protected, unless specifically stated otherwise in the figure caption in the Version of Record.

When available, you can view the Version of Record for this article at:

<http://iopscience.iop.org/article/10.1088/1361-6463/aa8503>

Structural properties, crystal quality and growth modes of MOCVD-grown AlN with TMAI pretreatment of sapphire substrate

Haiding Sun¹, Feng Wu¹, T. M. Al tahtamouni², Nasir Alfaraj¹, Kuang-Hui Li¹, Theeradetch Detchprohm³, Russell D. Dupuis³, Xiaohang Li¹

¹ King Abdullah University of Science and Technology (KAUST), Advanced Semiconductor Laboratory, Thuwal, 23955-6900, Saudi Arabia

² Materials Science and Technology Program, College of Arts and Sciences, Qatar University, Doha 2713, Qatar

³ Center for Compound Semiconductors and School of Electrical and Computer Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332, USA

Email: xiaohang.li@kaust.edu.sa

Abstract

The growth of high quality AlN epitaxial films relies on precise control of the initial growth stages. In this work, we examined the influence of the trimethylaluminum (TMAI) pretreatment of sapphire substrates on the structural properties, crystal quality and growth modes of heteroepitaxial AlN films on (0001) sapphire substrates. Without the pretreatment, the AlN films nucleated on the smooth surface but exhibited mixed crystallographic Al- (N-) polarity, resulting in rough AlN film surfaces. With increasing the pretreatment time from 1 to 5 s, the N-polarity started to be impeded. However, small islands were formed on sapphire surface due to the decomposition of TMAI. As a result, small voids became noticeable at the nucleation layer (NL) because the growth started as quasi three-dimensional (3D) but transformed to 2D mode as the film grew thicker and got coalesced, leading to smoother and Al-polar films. On the other hand, longer pretreatment time of 40 s formed large 3D islands on sapphire, and thus initiated a 3D-growth mode of the AlN film, generating Al-polar AlN nanocolumns with different facets, which resulted into rougher film surfaces. The epitaxial growth modes and their correlation with the AlN film crystal quality under different TMAI pretreatments are also discussed.

Keywords: TMAI pretreatment, AlN film, polarity, crystal quality, growth mode

1. Introduction

As members of the III-nitrides family, $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloys have become a key material for deep ultraviolet (DUV) optoelectronic devices [1-5]. Even though bulk AlN substrates have been developed and are suitable for device applications, they are still expensive and only available in small sizes. Recently, the development of AlN films on silicon (Si) substrates showed the potential for realizing UV devices [6]. However, the crystal quality of AlN on Si was relatively low due to the large lattice and thermal mismatches between AlN and Si. Unlike sapphire, Si is also UV-absorptive. Thus, the growth of AlGaN-based devices is often carried out on AlN film grown on sapphire substrate. Extensive studies have been carried out on the growth optimization of the AlN films on sapphire. In particular, the initial growth conditions including hydrogen (H_2) annealing [7], growth temperatures [8, 9], thickness control of the nucleation layers (NLs) [10, 11], and the III/V ratio [12] have shown significant impacts on the surface morphology, polarity and crystal quality of the MOCVD-grown AlN films. Inappropriate growth temperatures and thicknesses of the NL could lead to generating mixed polarities and inversion domains in AlN films.

Moreover, it has been reported that the pretreatment of sapphire substrates either by ammonia (NH_3) or trimethylaluminum (TMAI) is strongly correlated with the surface morphology [13,14], polarity [15], and crystal quality [16]. Researchers demonstrated that atomically smooth Al-polar AlN films were obtained without nitridation [16]. Other research group have shown that mixed polarity by pre-growth nitridation of the sapphire substrate results in rough film surfaces because of the different growth rates of N-polar and Al-polar AlN films [8,17]. Theoretically, from ab initio studies, the nitridation is described as the N-rich limit of growth, with sapphire as the source of Al, and may result in different polarities of AlN films [18]. Experimentally, it was proposed that the formation of $\text{Al}_x\text{O}_y\text{N}_z$ is what leads to the rough surfaces and the mixed polarity of the AlN films [19, 20]. Recently, Mohn et al. achieved Al-polar AlN films on a nitridated sapphire substrate by carefully controlling the $\text{Al}_x\text{O}_y\text{N}_z$ intermediate layer followed by a high-temperature annealing process [13].

Nitridation is recognized as a crucial step and thus has been investigated extensively for the sake of achieving desirable AlN or GaN films grown by MOCVD, yet not much attention was paid to understanding the crystallographic polarity and growth evolution of nitride films achieved through TMAI pretreatment (i.e.

alumination). The topic of defective and rough-surface AlN/sapphire templates resulting from the TMAI pretreatment has been addressed before [21, 22]. Reentila et al. have argued that neither nitridation nor alumination gave the best structural quality [16]. In contrary, studies have shown that better AlN and GaN crystal quality can be achieved after pretreatment using TMAI [23, 24]. Recently, we revealed that it was the carbon presence at the AlN/sapphire interface after TMAI pretreatment that changed the polarity of AlN film. However, the crystal

quality of AlN film after the pretreatment was not analyzed [25].

In this study, we present a comprehensive investigation of the structural properties, including surface morphology, polarity, and crystal quality of AlN templates under different TMAI pretreatment durations of sapphire substrates. We also analyzed the correlation between the growth modes induced by different pretreatment and the crystal quality of AlN templates.

2. Experimental details

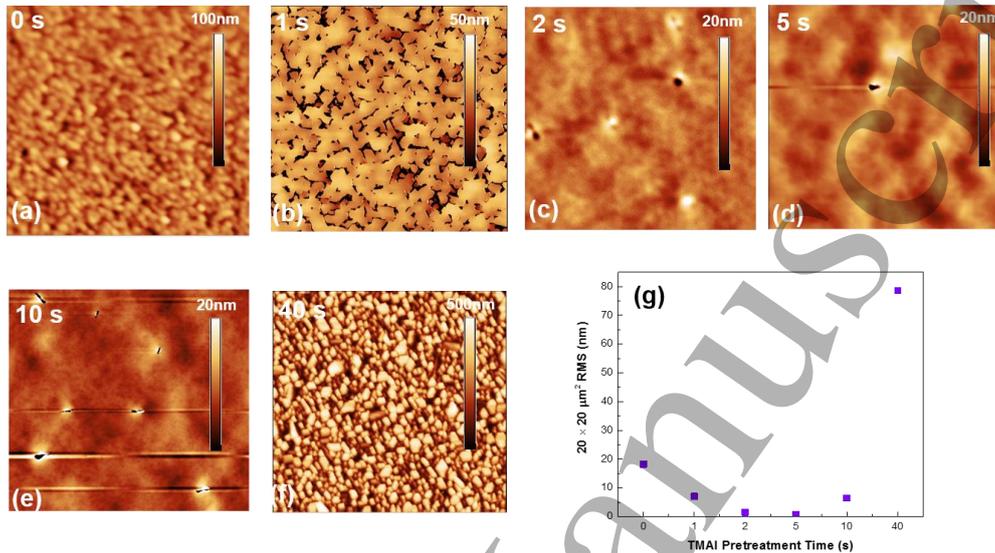


Figure 1. (a) – (f) show the $20 \times 20 \mu\text{m}^2$ AFM images of the surface morphology of $3 \mu\text{m}$ AlN/sapphire layers grown with pretreatment times of 0, 1, 2, 5, 10 and 40 s, respectively. (g) corresponds to the extracted RMS roughness value from AFM images with different pretreatment times.

The AlN heteroepitaxial layers were grown on a c-plane (0001) sapphire substrate using an AIXTRON Closed-Coupled Showerhead metalorganic chemical vapor deposition (MOCVD) system which has a base pressure of $\sim 1.5 \times 10^{-2}$ Torr. TMAI and NH_3 were used as precursors and H_2 as a carrier gas. The sapphire substrates were taken from the original package and loaded into the reactor immediately without solvent cleaning. The AlN growth processes comprised four steps. First, the susceptor and sapphire substrates were baked at 1100°C for 5 min in the H_2 ambient environment. Second, a preflow of TMAI was introduced to the substrate with pretreatment times of 0, 1, 2, 5, 10, and 40 s at a same flow rate of 1.72×10^{-5} mol./min with a chamber pressure of 85 mbar during the pretreatment. Then the growth started with a 15 nm AlN NL at a temperature of $\sim 900^\circ\text{C}$. Last, a $\sim 3 \mu\text{m}$ AlN layer was grown at $\sim 1100^\circ\text{C}$. Except for the pretreatment times of the TMAI, the other growth parameters were the same for all samples. The reactor pressure was 85 mbar during the growth. Besides AlN films, we also prepared two samples with 5 and 40 s TMAI pretreatment (hereafter TMAI-only samples) under the identical MOCVD condition, without the subsequent AlN growth. The surface morphology and roughness of the AlN films and TMAI-only samples were examined by atomic force microscopy (AFM). Microstructures were revealed via high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) at an acceleration

voltage of 300 kV in an FEI Titan Probe microscope system. The TEM specimens were prepared by focused ion beam (FIB) using an FEI Helios SEM system with a Ga ion source. High resolution-X-ray diffraction (HR-XRD) was performed to characterize the crystalline quality.

3. Results and discussions

Figure 1 shows the AFM root mean square (RMS) roughness and surface morphology of the AlN templates. The results exhibit significant difference in surface morphology. Without TMAI pretreatment, the sample exhibited a rough surface with pits. The surface morphology gradually smooths out as the pretreatment time increased from 0 to 5 s. Small pits (threading dislocation centers) are still visible in the sample with the 5 s pretreatment. However, the surface became rougher again with a longer pretreatment time for the sample at 10 s duration. After the 40 s pretreatment, the sample had the roughest surface among the investigated AlN templates. The smoothest AlN film was obtained at under the 5 s pretreatment time with an RMS roughness of less than 1 nm. The sample with the 40 s pretreatment time yielded an RMS value of 80 nm, indicating a 3D growth condition of AlN film might be established after such long pretreatment. Thus, without and excessive pretreatment

shows the atomic resolution image, confirming the Al-polarity. As a result of the homogeneous and uniform Al-polarity across the wafer and lack of inversion domains, we obtained a uniform growth rate across the entire 2-inch wafer. In accordance with the AFM measurement, the result suggested that the RMS roughness value reduces after the pretreatment time increased beyond 0 to 5 s. The improved surface morphology is mainly attributed to uniform polarity given that the proportion of Al-polarity increased by suppressing the oxygen diffusion from the sapphire after the pretreatment. With the 5 s pretreatment, the formation of N-polarity regions was completely impeded [32].

Figure 3(b) shows that the overall HAADF-STEM image of the AlN/sapphire template with the 40 s pretreatment that exhibited the roughest surface among the investigated samples. The surface had faceted nanocolumns. Meanwhile the AlN/sapphire interface included a high density of voids and TDs. This is primarily due to excessive pretreatment of the sapphire, resulting in the formation of high density large 3D islands (domains) at NL. Thus, a 3D growth mode was immediately established after the pretreatment, creating nanocolumns with well-defined boundaries. Most of the TDs formed at the interface or diminished at the boundary, leaving less density at the top part of the nanocolumns. Similar to the other two samples, the TDs rapidly vanished as the thickness of the AlN layer increased beyond 1 μm (white dashed line). The inset image confirms the Al-polarity of this film.

Next, we will address the correlation of TMAI-pretreated sapphire surface with the crystal quality and the growth mechanism of AlN films.

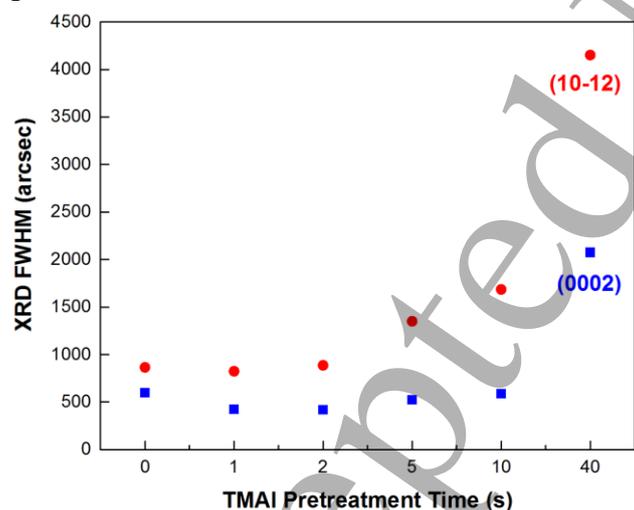


Figure 4. FWHM's of XRD (002) and (102) ω -scans on the AlN templates with different pretreatment time.

The crystal quality of the AlN templates was studied by measuring the full width at half maximum (FWHM) of the XRD symmetric (002) and asymmetric (102) ω -scans, as shown in Figure 4 [33-35]. Except for the sample with the 40-s pretreatment, the obtained FWHM values of XRD peaks were largely comparable with the ones reported by other groups, falling in the range of 100~500 and 800~1500 arcsec for the (002) and (102) plane, respectively [36-39]. The FWHM values of the symmetric

(002) and asymmetric (102) ω -scans corresponded to and were proportional to the screw dislocation and the edge dislocation densities, respectively. It is generally noted that the screw dislocations and edge dislocations originate from the stacking faults that cause tilting and twisting grain boundaries. A close examination of Figure 4 reveals different trends of the crystalline quality with different pretreatment times. The (002) FWHM (blue curve) is nearly constant during the first 10 s pretreatment, indicating that our relatively lower temperature ($\sim 900^\circ\text{C}$) pretreatment processes did not result in a large tilt of AlN nuclei, thus the density of screw dislocation did not change too much. For the (102) FWHM (red curve), it slightly decreases after the 2 s pretreatment and then increases significantly from 5 to 40 s pretreatment duration, indicating higher density of edge dislocations. The smallest (002) and (102) FWHMs were obtained when the pretreatment time was 1~2 s and the (102) FWHM is more sensitive to the pretreatment time. A similar XRD trend was observed in the study of the pretreatment of sapphire using TMAI [32] for MOCVD-grown GaN templates. However, our observation is contrary to what O. Reentila et al. reported earlier [16]. They presented both FWHMs increased dramatically with the increase of TMAI exposure time, and the surface roughness of their films remained below 1 nm, even after the 80 s pretreatment. Thus, we believe the discrepancy is probably due to higher growth temperature of 1500 $^\circ\text{C}$ and 1000 $^\circ\text{C}$ for their AlN template and AlN-NL respectively. Normally, higher growth temperatures (generally more than 1400 $^\circ\text{C}$) are favorable for the migration of Al adatoms, which benefits the increase of crystal quality of AlN templates [40]. Another study has shown that the roughness of the NL decreased by increasing the growth temperature of the NL (T_{NL}) because the diffusion length of Al adatoms and lateral coherence length at sapphire surface increased as T_{NL} was increased [41]. So the growth temperature plays such significant role in the process of AlN film by MOCVD.

Besides the variation of the crystal quality, TEM studies (Figure 2 and 3) revealed the drastic microstructure difference between samples underwent various pretreatment conditions. Most notably, the growth modes have undergone significant transformations as a result of changing the pretreatment times. Here we present the investigation on the surface morphology of the TMAI-only samples using AFM prior to the AlN epitaxy, as shown in Figure 5. Without pretreatment, the sapphire surface was extremely smooth, with a measured RMS value of 0.2 nm. The cross-section profile across the scanned surface shows less than 1 nm in height. However, after 5 s pretreatment, we clearly observed spotty surface with a measured RMS value of 2.3 nm, indicating a formation of rough surface with small islands on the sapphire substrate. The cross-section profile indicates an 8 - 10 nm in height. With 40 s pretreatment, irregular nanoscale 3D islands (50 - 100 nm in horizontal dimension) on sapphire substrates were formed, with a measured RMS value of 6.3 nm. The cross-section profile indicates a measured height ranges of 15 - 20 nm.

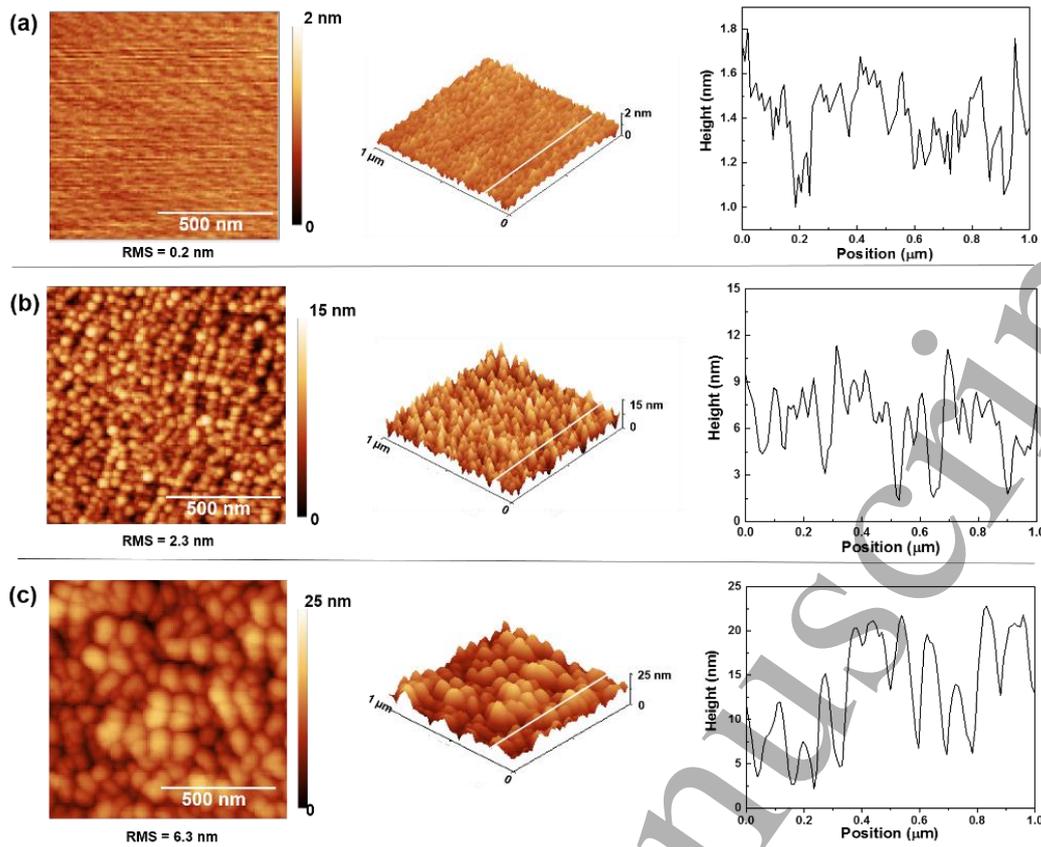


Figure 5. (a), (b) and (c) are AFM images of surface morphology of TMAI-only samples (without AlN epitaxy) with the 0, 5 and 40 s pretreatment, respectively. The left, middle, and right are the plan-view, 3D image and cross-section profiles, respectively, corresponding to the marked line in the 3D image.

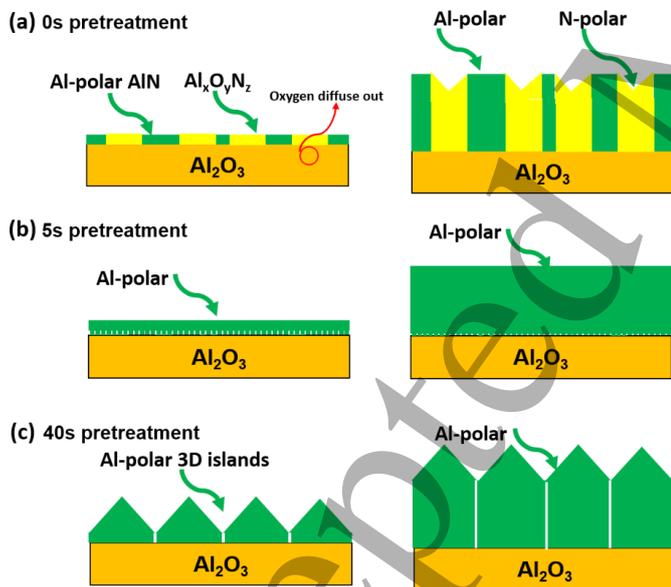


Figure 6. Schematic illustration of the growth modes, the evolution of the AlN templates with (a) 0 s, (b) 5 s, and (c) 40 s pretreatment

Based on the observation of the structural characteristic of AlN films and TMAI-only samples above, here we propose the growth mechanism under 0, 5 and 40s pretreatment. Figure 6(a) shows the creation of rough AlN films without pretreatment. Although the AlN started nucleation on smooth sapphire, because of the formation of $\text{Al}_x\text{O}_y\text{N}_z$ in the first few monolayers of AlN growth [13, 17], Al-polarity was partially transformed to N-polarity and this has led to the mixed polarity of AlN film, thus created a rough surface. However, after the TMAI pretreatment, the process not only helped establish

the Al-polar growth condition but also create a slightly roughened surface due to the TMAI decomposition, confirmed by AFM in Figure 5(b) [42]. The residuals of decomposed TMAI formed a cluster of small islands, thus a quasi-3D growth mode was introduced. The formation of small islands, which was accounted for inducing the quasi-3D growth mode, was proved to be beneficial to create higher quality of AlN and GaN films [23, 24]. As the growth continued, these small AlN grains coalesced and the surface became smooth, which meant a 2D-growth (a layer-by-layer growth) was established in the end. This process is similar to the high-quality and smooth GaN growth on sapphire with a combination of quasi-3D and then 2D growth modes [43, 44]. Figure 6 (b) presents the schematic of such growth mechanism for the 5 s pretreatment. A uniform and smooth surface of AlN template was obtained. In addition to the improvement of surface morphology, an increase of the density of edge dislocations ((102) FWHMs of XRD ω -scan) was observed by increasing the pretreatment time. This might be due to the increasing size of islands formed on sapphire after longer pretreatment time, as shown in Figure 5, preventing the migration of arriving Al and N atoms horizontally. These islands facilitated in generating more twisted grain boundaries while establishing the initial quasi-3D growth mode, thus a higher density of edge dislocations. Also, the larger the islands prior to the AlN epitaxy on sapphire, the thicker subsequent AlN film might be required to get fully coalesced while creating small voids in the bottom AlN layer [23]. Nevertheless,

these islands did not contribute to the tilt of in-plane AlN nuclei, thus little impact on the density of screw dislocations after increasing the pretreatment time from 1 to 10 s. Once the sapphire substrate is over-treated, a high density of larger 3D islands were formed on sapphire (Figure 5(c)), leading to extreme rough NL. The NL might act as a mask and further prevent Al-adatoms from diffusion to different nucleation islands, thus leading to a 3D growth mode throughout the subsequent template growth. The Al-polar AlN template with nanocolumns was grown on those islands which are consistent with the AFM measurements, as shown in Figure 6(c). Interestingly, Eom et al. were able to fabricate AlN 3D nanostructures using KOH etching by controlling the polarity of the AlN films due to the TMAI preflow [45].

4. Conclusions

In conclusion, we have studied the influences of the TMAI pretreatment on the structural properties, crystal quality and growth mode of AlN films on sapphire substrates by MOCVD. Due to the absence of the TMAI pretreatment, the AlN films had a mixed polarity, but after a few seconds of pretreatment, the polarity of AlN films tended to convert to Al-polarity, while the surface roughness and crystal quality were improved according to the FWHM variations of the (102) and (002) rocking curves. However, excessive pretreatment results in poorer crystal quality and a rough surface. A detailed epitaxial growth mechanism under different pretreatment time were presented. A shift of growth mode from quasi-3D to 3D was identified after increasing the pretreatment time. We conclude that pretreatment times that are too short or too long should be avoided as neither is favorable for AlN epitaxy on sapphire. These results give practical advice and guidance in changing the growth mode of AlN film to achieve desirable AlN films.

Acknowledgements

The KAUST authors would like to acknowledge the support of GCC Research Program REP/1/3189-01-01, Baseline BAS/1/1664-01-01, and Equipment BAS/1/1664-01-07. The work at QU was supported by GCC Research Program GCC-2017-007. The work at Georgia Institute of Technology was supported in part by DARPA under grant W911NF-15-1-0026 and NSF under grant DMR-1410874. RDD acknowledges the additional support of the Steve W. Chaddick Endowed Chair in Electro-Optics and Georgia Research Alliance.

References

- [1] Janjua B *et al.* 2017 *Opt. Express* **25** 1381.
- [2] Janjua B *et al.* 2017 *Nanoscale* **9** 7805.
- [3] Sun H and Moustakas D T 2014 *Appl. Phys. Express* **7** 012104.
- [4] Pecora E F, Sun H, Negro L D and Moustakas T D 2015 *Opt. Mater. Express* **5** 809.
- [5] Sun H, Yin J, Pecora E F, Dal Negro L, Paiella R and Moustakas T D, 2017 *IEEE Photon. J.* DOI: 10.1109/JPHOT.2017.2716420
- [6] Tran B T, Maeda N, Jo M, Inoue D, Kikitsu T and Hirayama H 2016 *Sci. Rep.* **6** 35681.
- [7] Kumagai Y, Akiyama K, Togashi R, Murakami H, Takeuchi M, Kinoshita T, Takada K, Aoyagi Y and Koukitu A 2007 *J. Cryst. Growth* **305** 366.
- [8] Wu Y, Hanlon A, Kaeding J F, Sharma R, PFinì T, Nakamura S and Speck J S 2004 *Appl. Phys. Lett.* **84** 912.
- [9] Li X H *et al.* 2015 *J. Cryst. Growth* **414** 76.
- [10] Miyagawa R, Yang S, Miyake H, Hiramatsu K, Kuwahara T, Mitsuhashi M and Kuwano N 2012 *Appl. Phys. Express* **5** 025501.
- [11] Li H N, Sadler T C and Parbrook P J 2013 *J. Cryst. Growth* **383** 72.
- [12] Claudel A, Fellmann V, Gélard I, Coudurier N, Sauvage D, Balaji M, Blanquet E, Boichot R, Beutier G and Coindeau S 2014 *Thin Solid Films* **573** 140.
- [13] Mohn S *et al.* 2016 *Phys. Rev. Applied* **5** 054004.
- [14] Takeuchi M *et al.* 2007 *J. Cryst. Growth* **305** 360.
- [15] Jasinski J, Liliental-Weber Z, Paduano Q S and Weyburne D W 2003 *Appl. Phys. Lett.* **83** 2811.
- [16] Reentila O, Brunner F, Knauer A, Mogilatenko A, Neumann W, Protzmann H, Heuken M, Kneissl M, Weyers M and Trankle G 2008 *J. Cryst. Growth* **310** 4932.
- [17] Paduano Q S, Weyburne D W, Jasinski J and Weber Z L 2004 *J. Cryst. Growth* **261** 259.
- [18] Di Felice R and Northrup J E 1998 *Appl. Phys. Lett.* **73** 936.
- [19] Dwikusuma F and Kuech T F 2003 *J. Appl. Phys.* **94** 5656.
- [20] Losurdo M, Capezuto P, Bruno G, Namkoong G, Doolittle W A and Brown A S 2002 *J. Appl. Phys.* **91** 2508.
- [21] Kawaguchi K and Kuramata A 2005 *Jpn. J. Appl. Phys.* **44** L1400.
- [22] Kueller V, Knauer A, Brunner F, Mogilatenko A, Kneissl M and Weyers M 2012 *Phys. Status Solidi C* **9** 496.
- [23] Bao Q, Luo J and Zhao C 2014 *Vacuum* **101** 184.
- [24] Bak S J, Mun D H, Jung K C, Park J H, Bae H J, Lee I W, Ha J S, Jeong T and Oh T S 2013 *Electron. Mater.* **9** 367.
- [25] Sun H, Wu F, Park Y J, Altahtamouni T M, Li K H, Alfaraj N, Deichprohm T, Dupuis R D, Li X, 2017 *Appl. Phys. Lett.* **110** 192106.
- [26] Hussey L *et al.* 2014 *Appl. Phys. Lett.* **104** 032104.
- [27] Wong M H, Wu F, Speck J S and Nakamura S 2010 *J. Appl. Phys.* **108** 123710.
- [28] Kumagai Y, Enatsu Y, Ishizuki M, Kubota Y, Tajima J, Nagashima T, Murakami H, Takada K and Koukitu A 2010 *J. Cryst. Growth* **312** 2530.
- [29] Harumoto T, Sannomiya T, Matsukawa Y, Muraishi S, Shi J, Nakamura Y, Sawada H, Tanaka T, Tanishiro Y and Takayanagi K 2013 *J. Appl. Phys.* **113** 084306.
- [30] Li C, Liu H and Chua S J 2015 *J. Appl. Phys.* **117** 125305.
- [31] Li X H *et al.* 2015 *Phys. Status Solidi B* **252** 1089.
- [32] Lim D H, Xu K, Arima S, Yoshikawa A and Takahashi K 2002 *J. Appl. Phys.* **91** 6461.
- [33] Wang H M, Zhang J P, Chen C Q, Fareed Q, Yang J W and Khan M A, 2002 *Appl. Phys. Lett.* **81** 604.
- [34] Heying B, Wu X H, Keller S, Li Y, Kapolnek D, Keller B P, DenBaars S P and Speck J S, 1996 *Appl. Phys. Lett.* **68** 643.
- [35] Ayers J E 1994 *J. Cryst. Growth* **135** 71.
- [36] Imura M *et al.* 2007 *J. Cryst. Growth* **300** 136.
- [37] Peng M Z *et al.* 2008 *Chin. Phys. Lett.* **25** 2265.
- [38] Chen Y, Song H, Li D, Sun X, Jiang H, Li Z, Miao G, Zhang Z and Zhou Y. 2014 *Mater. Lett.* **114** 26
- [39] Pantha B N, Dahal R, Nakarmi M L, Nepal N, Li J, Lin J Y, Jiang H X, Paduano Q S and Weyburne D, 2007 *Appl. Phys. Lett.* **90** 241101.
- [40] Sun X J, Li D B, Chen Y R, Song H, Jiang H, Li Z M, Miao G Q and Zhang Z W 2013 *Cryst. Eng. Comm.* **15** 6066.
- [41] Chen Y, Song H, Li D, Sun X, Jiang H, Li Z, Miao G, Zhang Z and Zhou Y 2014 *Mater. Lett.* **114** 26.
- [42] Ambacher A, Brandt M S, Dimitrov R, Metzger T, Stutzmann M, Fischer R A, Miehr A, Bergmaier A and Dollinger G, 1996 *J. Vac. Sci. Technol. B* **14** 3532.
- [43] Zhao D G, Zhu J J, Liu Z S, Zhang S M, Yang H and Jiang D S 2004 *Appl. Phys. Lett.* **85** 1499.
- [44] Xu Z Y, Xu F J, Wang J M, Lu L, Yang Z J, Wang X Q and Shen B 2016 *J. Cryst. Growth* **450** 160.
- [45] Eom D, Kim J, Lee K, Jeon M, Heo C, Pyeon J, and Nam O 2015 *J. Nanosci. Nanotechnol.* **15** 5144.