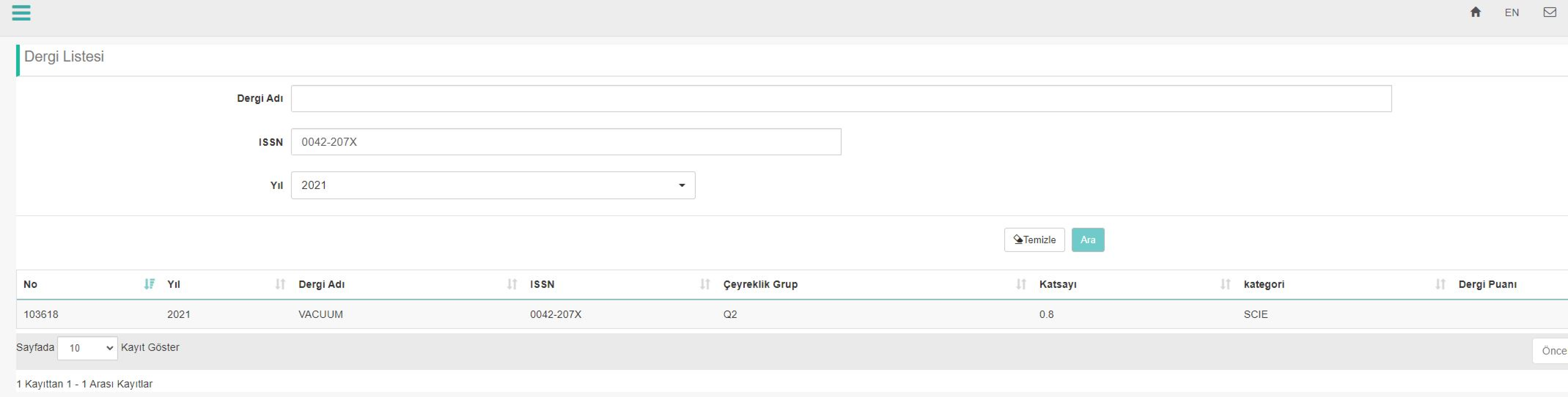

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In-situ thermal cleaning of the sapphire substrate and temperature effect on epitaxial AlN

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ABSTRACT

The impact of thermal surface cleaning on epitaxial AlN thin films grown on sapphire is investigated in this study at various temperatures. The sapphire substrate is cleaned in a hydrogen environment. Structural, optical, and surface morphology properties of the samples are investigated by using high-resolution X-ray diffraction, Raman spectroscopy, UV–visible spectroscopy, and atomic force microscopy, respectively. Because wastes from the surface of sapphire, which begins to decompose after 1200 °C, cannot be entirely removed at such a low temperature, grain distribution and grain size in the nucleation layer are impacted. Moreover, when a sapphire is cleaned at a high temperature, the rate of breakdown of oxygen atoms from the surface rises, and islands appear on the surface. Roughnesses on the sapphire surface cause misoriented grains and deteriorated the structure. The surface of the sapphire, which is cleaned at 1245 °C, was both sufficiently cleaned and not etched too much, uniformly distributed, and large-sized particles formed in the nucleation layer. Thus, the AlN thin film has grown with high quality in terms of structure, optics, and surface. Experimental results have demonstrated that the in-situ thermal cleaning temperature has a critical influence on the properties of the AlN.

1. Introduction

AlN is a remarkable material thanks to its wide bandgap, good thermal stability, and high thermal conductivity [1–4]. The remarkable properties mentioned have led to the use of AlN in very important application areas like high-power electronic and optoelectronic devices such as radio-frequency filters [5], high electron mobility transistors [6], microelectromechanical systems [7], quantum cascade laser [8], UV photodetectors [9], missile-warning systems [10], etc. AlN growth is generally considered an essential template for devices. It is important to be able to obtain high-quality AlN templates to produce high-performance devices. Epitaxial films that are grown using lattice-matched and thermal expansion coefficient-matched substrates contain less dislocation density. Although homoepitaxial growth is the best approach for developing high-efficiency, long,-lasting, and high-performance devices, AlN substrates are rarely employed because of their small size and high cost. Low-cost substrates like sapphire are commonly used in the mass production of AlN growths. High-quality AlN thin films are difficult to develop owing to the limited mobility of Al, gas-phase parasitic interactions (between TMA1 and NH_3), and lattice mismatch (approximately 13%) [11,12], and thermal mismatch (between AlN and sapphire, 44%) [13]. By solving these challenges, high-quality AlN thin films with excellent crystal quality, smooth surface, and low threading dislocation density have been attempted in the literature. Two-step growth technique (it has been the growth of high-temperature AlN thin film on the nucleation layer at low temperature) [14,15], high-temperature growth (above 1300 °C) [16], epitaxial lateral growth technique [14,17], migration enhanced epitaxy [13,16] and PALE technique [13,18] are some of these attempts.

The state of the film interface between the substrate and epitaxial thin film is another critical element for high-quality thin film growth. In semiconductor device structures, as device size decreases and integration scale increases, the quality of interfaces has become an increasingly important issue. When contaminants are removed from the substrate surface, it is simpler to achieve sharp substrate/film interfaces, enabling the manufacturing of high-quality devices. Surface cleaning methods are the foundation for the manufacture of semiconductor devices [19]. At higher operating powers and higher oscillation frequencies, parasitic

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resistances and capacitances at the contact surfaces become more damaging [20]. Losses at the contact interfaces account for a major fraction of the losses in many devices, and hence have a significant influence on device performance [20]. As a result, attempts to enhance the sapphire surface have been made. Heinlein et al. [21] studied the sapphire surface condition to generate GaN thin films by exposing the sapphire surface to hydrogen (H_2) at various temperatures before development. Yumiya and Fuke [22] investigated its effect on GaN thin film by exposing it to carrier gases of nitrogen (N_2) and H_2 at temperatures between 800 and 1080 °C for 10 min. It was observed that the sapphire surface exposed to H_2 gas above 950 °C had a positive effect on the GaN thin film. Kim et al. [23] investigated the effect of temperature by exposing the sapphire substrate to an H_2 environment at temperatures of 1040–1120 °C. They reported that sapphire cleaned by exposure to H_2 at 1070 °C for 10 min was a more suitable procedure for GaN films. These studies aided in the creation of suitable substrate surfaces as well as a better knowledge of GaN heteroepitaxy.

Keeping the substrate under H_2 flow for a certain period at high temperatures before it starts to grow to clean the surface of impurities such as water vapor and improve the surface is called desorption that is, it is a kind of thermal surface cleaning process [24,25]. For the first time in the literature, the influence of varied desorption temperatures on AlN thin films grown on sapphire by metal-organic vapor phase epitaxy (MOVPE) was studied in the current study.

2. Experimental

AlN thin films were grown on sapphire using AIXTRON 200/4 RF-S horizontal flow low-pressure metal-organic vapor phase epitaxy system. High purity trimethyl aluminum (TMAl) metal-organic source and ammonia (NH_3) hydride source were used for AlN films. The in-situ process is to continue the process without exposing the sample to the air environment. Before starting the AlN nucleation layer growth, the impurities on the sapphire surface were cleaned by thermal surface cleaning in the H_2 atmosphere for 10 min at different temperatures (1100 °C, 1175 °C, 1245 °C, 1315 °C, 1377 °C) in MOVPE The nucleation layer was grown at 1080 °C for 5 min (~15 nm), then high-temperature AlN thin films were grown at 1415 °C using the pulse atomic layer epitaxy (PALE) technique (~180 nm). To further analyze the effect of thermal surface cleaning temperature, the growths were divided into three sets. Details of the determined sets are given in Table 1. In the first set (Fig. 1 a), only thermal surface cleaning was performed on sapphires at different temperatures. In the second set (Fig. 1 b), the AlN nucleation layers were grown at 1080 °C on thermally cleaned sapphires at different temperatures. In the third set (Fig. 1 c), high-temperature AlN thin films were grown on the structure specified in the second set. The thermal surface cleaning temperature in Table 1 is the sapphire cleaning temperature for three sets. In this study, the effect of thermal surface cleaning temperature on sapphire, the effect on the nucleation layer, and the effect of AlN thin film were investigated for the first time in the literature by using MOVPE to grow AlN on sapphire was studied in detail. The characterization of thin films was carried out using high-resolution X-ray diffraction (HRXRD), atomic force microscopy (AFM), UV–visible spectrophotometry (UV–Vis or UV/Vis), and Raman spectroscopy.

Table 1
Name of samples according to thermal surface cleaning temperature change.

Thermal Surface Cleaning (Desorption) Temperature (°C)	Step		
	First	Second	Third
	Thermal Surface Cleaning	Thermal Surface Cleaning + Nucleation Layer	Thermal Surface Cleaning + Nucleation Layer + HighTemperature AlN Film
1100	Sample A1	Sample A2	Sample A
1175	Sample B1	Sample B2	Sample B
1245	Sample C1	Sample C2	Sample C
1315	Sample D1	Sample D2	Sample D
1377	Sample E1	Sample E2	Sample E

3. Results and discussion

The desorption process provides an Al-rich surface by removing surface contamination such as carbon and oxygen. The reactions between sapphire and H_2 are given below:

$Al_2O_3(s) + 2H_2(g) = 2AlH(g) + H_2O(g) + 2O(surface)$ (1) [26].

Al2O3(s) + H2(g) = 2Al(g) + H2O(g) + 2O(surface) (2) [26].

It was observed that reaction (1) took place at temperatures below 1300 °C and the reaction (2) at a temperature above 1300 °C [26]. In their study on sapphire substrate, Akiyama et al. [27] reported that the decomposition started after 1200 °C under H_2 gas. For high-quality AlN films, it is critical to comprehend the decomposition mechanism.

Firstly, the XRD characterization method has been performed to look at the effect of thermal surface cleaning temperatures on sapphire. The XRD results of the growths divided into three parts have been given below. Symmetrical and asymmetrical scans are needed to find the different properties of materials. In symmetric scanning, strain and compositional changes in the structure might show similar peak shifts. Asymmetric scans are required to better analyze strain and relaxation. Full-width-half-maximum (FWHM) values obtained from 2 theta/omega, symmetric (002), and asymmetric omega (102) scans of thermal surface cleaning to be applied on the sapphire surface are indicated in Fig. 2. Symmetrical diffraction (002) and asymmetrical diffraction (102) FWHM values give information about screw dislocation density and edge dislocation density, respectively. When the FWHM values of the asymmetric omega (102) scan are examined, an increase is observed when the thermal surface cleaning temperature is increased from 1100 °C to 1175 °C. It is noteworthy that the FWHM values remain almost constant at temperatures after 1175 °C. The FWHM values of the symmetric omega scan shows that increasing the temperature has a favorable effect on the (002) plane. The influence of in-situ thermal cleaning on the sapphire structure can be attributed to a very short-term annealing effect. In a cleaning experiment carried out at 1245 °C, the sapphire structural quality improved. As it is observed in Fig. 2, FWHM values are almost stable after 1245 °C. Sapphire has been claimed to be stable at temperatures of 1200 °C and above [28]. The impact of sapphire substrate annealing on AlN thin film was ascribed by Liu and Zhang to three factors [28]. Firstly, it facilitates obtaining single dominantly oriented AlN thin films by creating a controlled nucleation layer by reducing the surface energy due to the smooth and sharp atomic steps that will occur on the sapphire surface [29,30]. Secondly, obtaining atomically flat terrace surfaces and finally, after annealing, the dislocation density decreases as the entire sapphire surface ends in the same way [28].

The XRD results of the nucleation layer (NL) developed on sapphires used in the thermal surface cleaning method at various temperatures are revealed in Fig. 3. The NL was grown to reduce the lattice mismatch and the limitations of the thermal expansion coefficient mismatch between AlN and sapphire [31]. On the NL, the sapphire surface's initial effects are visible. After cleaning, the steps generated on the surface reveal information about NL. Since the AlN nucleation layer (AlN NL) is rather too thin, asymmetric omega scanning for it could not be performed. In both the 2theta/omega scan and the omega (002) scan, Sample C2 exhibits the smallest FWHM. When omega (002) is studied, it is discovered that a 33% rise in FWHM has a detrimental impact on the NL quality after desorption temperatures greater than 1245 °C. FWHM values of the symmetric (002) plane mainly give information about the screw dislocation density produced within the nucleation islands (NIs) [32]. It is also related to tilting. It is thought that the reason for the high FWHM

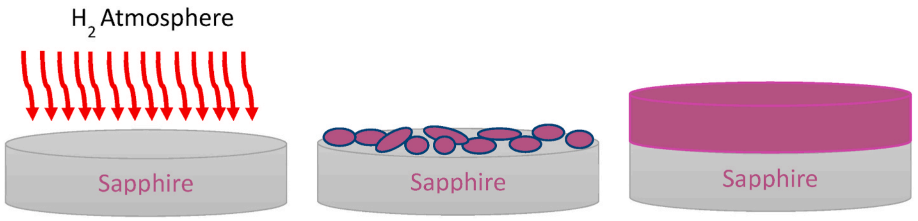

Fig. 1. **(a)** Thermal surface cleaning, **(b)** Thermal surface cleaning + nucleation layer, **(c)** Thermal surface cleaning + nucleation layer + high temperature AlN film.

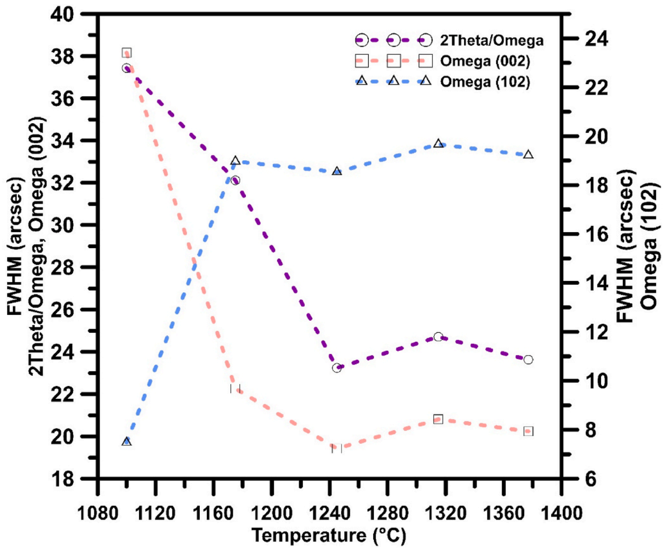

Fig. 2. FWHM values of sapphire 2theta/Omega, Omega (002), and asymmetric omega (102) XRD scans of sapphire versus in-situ thermal surface cleaning temperature.

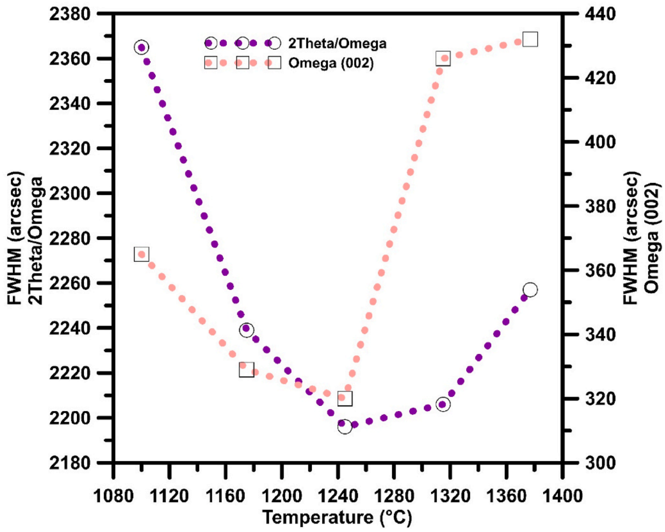

Fig. 3. FWHM values of NL AlN 2theta/Omega and Omega (002) XRD scans versus in-situ thermal surface cleaning temperature.

values at low cleaning temperatures may be since the cleaned sapphire surfaces are not completely free of contamination or a mixed polarity is formed on the sapphire surface. Pits may form on the sapphire surface because of the etching of the high thermal cleaning temperature. It is thought that the roughness of the sapphire surface affects the orientation of the grains in the nucleation layer, forming misoriented grains. The observation of the minimum FWHM value at 1245 °C can be considered as an indication that high-quality NL has been successfully grown on sapphire.

Fig. 4 demonstrates the AFM images and root mean square (RMS) roughness values of the grown NL and full structure (nucleation layer + AlN thin film) on sapphire. In Fig. 4 (a), the images of the NL are given in the left part while the images of the full structure are given in the right part. When the AFM images of the NL are examined, it is observed that the grain size and distribution of the particles in Sample D and Sample E are random compared to the others. Increasing the desorption temperature is not induce grain coalescence in Sample D and Sample E. The void between grains is large due to the inhomogeneous grain sizes and their random distribution in the NL. It may be necessary to eliminate these voids by increasing the thickness to coalescence. Fig. 4 (a) shows that the nucleation islands of Sample C2 have a more homogeneous distribution and larger average grain sizes. These results could be correlated with the reduced dislocation density of Sample C2 as described in the XRD results. Fig. 4 (b) indicates the RMS roughness values for AlN NL and full structure. The RMS roughness value of Sample E, whose NL is grown on the sapphire cleaned at 1377 °C, is the highest. Akiyama et al. [27] reported that sapphire decomposition started at 1200 °C, the rate-limiting reaction differed after 1300 °C and the activation energy increased above 1300 °C. Above 1200 °C, the oxygen (O) on the sapphire surface decomposes and becomes a surface rich in Al. As the detachment of oxygen atoms from the surface increases, a three-dimensional island is formed on the surface and this causes an increase in surface roughness. From the RMS graph, it is seen that the AlN thin film grown on sapphire thermally cleaned at 1245 °C is the smoothest.

Fig. 5 exhibits the variation of the FWHM values obtained for the full structures (NL AlN + PALE AlN) grown on sapphire. When the FWHM value increases, that is, when the peak becomes wider, the dislocation density increases. Al-polar AlN is more stable above 1220 °C than N-polar AlN [33]. Increasing Al-polar AlN and decreasing N-polar AlN will decrease threading dislocation density [33]. When thermal surface cleaning is performed, an Al-rich sapphire surface will emerge as shown in reaction (2). The dislocation density of AlN thin films grown on this surface will also decrease. As seen in Fig. 5, it is seen that the thermal surface cleaning process performed at 1245 °C is suitable for growing a high-quality AlN layer at high temperatures. The FWHM value is 171 arcsec in the 2 theta/omega scan and 89 arcsec in the omega scan. In the light of the XRD results obtained, it can be said that thermal cleaning at 1245 °C may be beneficial for AlN thin film.

The transmittance spectra (200–800 nm) of AlN grown on sapphires with thermal surface cleaning at different desorption temperatures are shown in Fig. 6. It has been observed that interference from the substrate-film interface and reflections from the film surface causes oscillations [34–36]. Since the interference patterns are due to the smooth reflective surfaces of the interface and surface [31,37], the samples do not have much reflection loss (about 80%). It can be noted that Sample E shows a significant loss of reflection. As can be seen from the AFM results in Fig. 4, its surface is rough. As the roughness on the surface reflects the light more, it causes the transmittance to decrease. The sharpness of the absorption edge given in Fig. 6 is an indication of the material quality. Sample A and Sample C appear to have been the

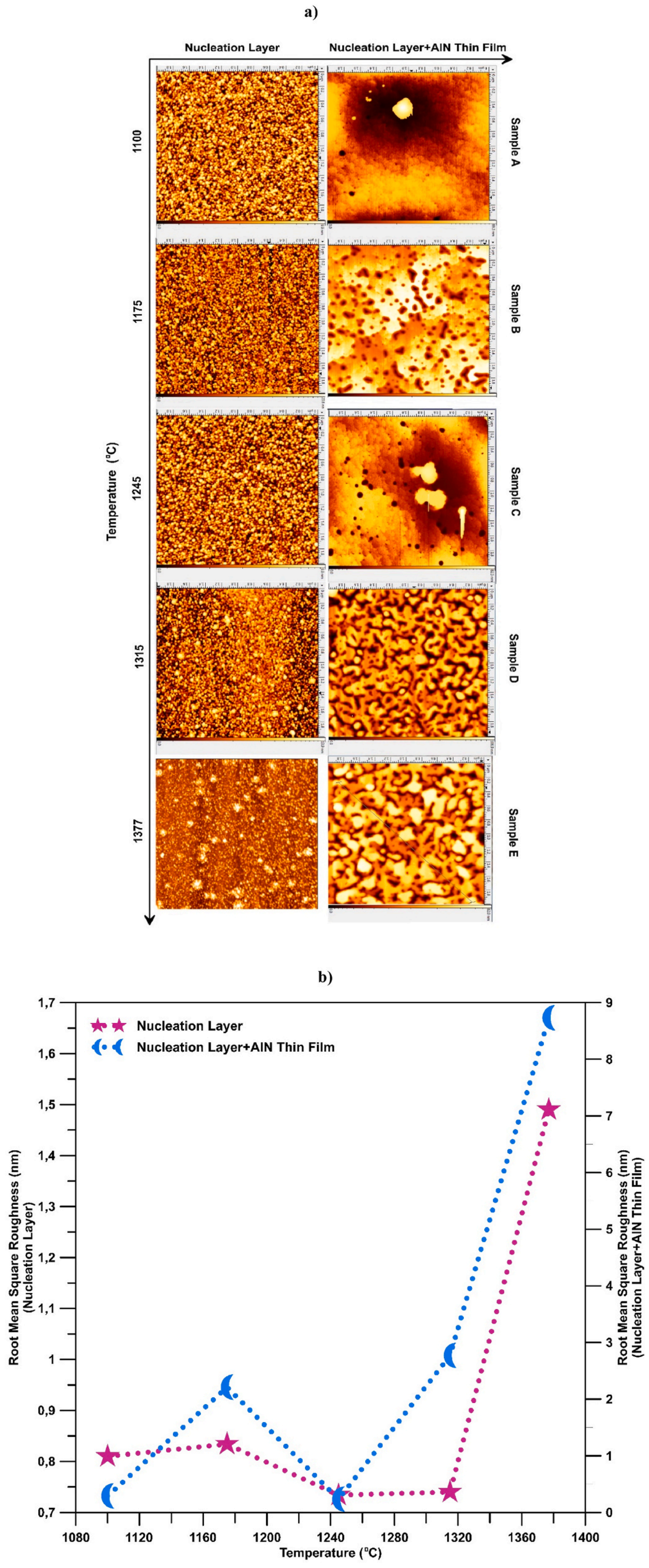

Fig. 4. **(a)** AFM images and **(b)** RMS roughness values of samples.

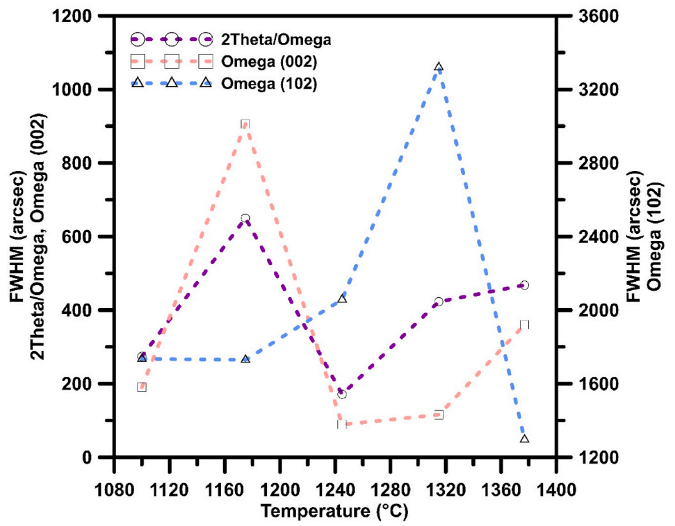

Fig. 5. FWHM values of full structure (NL AlN + PALE AlN) 2theta/Omega, Omega (002), and Asymmetric Omega (102) XRD scans versus thermal surface cleaning temperature.

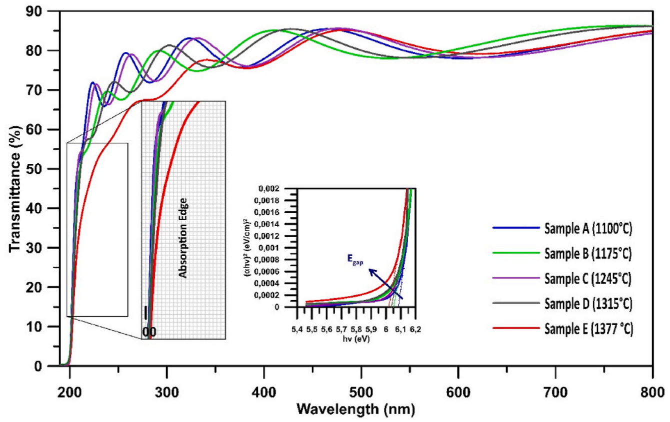

Fig. 6. Transmittance graph of full structure (NL AlN + PALE AlN) growths.

sharpest edge. The Tauc plot, in which the optical bandgap is determined, is given in Fig. 6 (inset). The energy bandgap can be determined from the intercept of the extrapolated linear part of the curve with the x-axis [38–40]. The optical bandgap value for Sample A, Sample B, Sample C, Sample D, and Sample E is determined as 6.09, 6.05, 6.05, 6.04, and 6.02 eV, respectively. Compressive tension is known to increase optical bandwidth [41]. Thus, it can be said that the compression behavior of the structure changes as the desorption temperature increases.

The Raman spectra shown in Fig. 7 (a) and Fig. 7 (b) belong to the sapphire subjected to thermal surface cleaning at different temperatures and the samples in which the NL is grown on these substrates. In both samples, the spectra contain six bands. In the literature, the wavelengths of the bands have been reported as 378, 418, 432, 451, 578, and 751 cm^{-1} [42]. As a result of the Raman analysis, it was observed that three bands were strong, while the others were weak. Table 2 and Table 3 give Raman peak and FWHM values for sapphire and NL, respectively. The numbering in the tables belongs to the phonon modes of the sapphire, which is also indicated in Fig. 7. So, number 1, number 2, and number 3 represent 418 cm^{-1} (A1$_g$) mode, 578 cm^{-1} (E$_g$(int)), 751 cm^{-1} (E$_g$(int)), respectively. The peaks of both sapphire and AlN NL are obtained at almost the same position. Due to the very thin AlN NL, AlN peaks can not be seen in the spectrum. When Tables 2 and 3 are examined, it is observed that the peak position and FWHM values in part 1 remain almost the same except for Sample C2. The FWHM value is related to defects and crystallinity, and the peak position change is related to the stress/strain change in the structure.

Raman analysis of the films is indicated in Fig. 8 (a). A$_1$ (TO), E$_2$

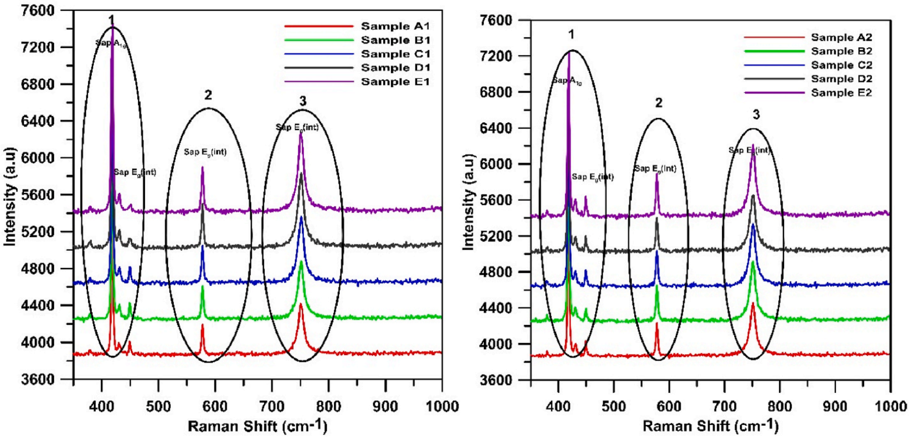

Fig. 7. Raman spectra of **(a)** thermally surface-cleaned sapphire substrate, **(b)** the NL was grown on a thermally surface-cleaned sapphire substrate.

Table 2
Data set of sapphire substrate with thermal surface cleaning.

Sample	Part 1 (418 cm^{-1}) A_{1g}		Part 2 (578 cm^{-1})		Part 3 (751 cm^{-1})	
	Peak Position (cm^{-1})	FWHM (cm^{-1})	Peak Position (cm^{-1})	FWHM (cm^{-1})	Peak Position (cm^{-1})	FWHM (cm^{-1})
A1	419.12	4.15	577.83	4	750.35	11.63
B1	419.14	4.19	577.83	4.03	751.44	11.81
C1	419.16	4.16	577.83	4.23	751.47	11.76
D1	419.14	4.17	577.83	4.3	750.22	11.89
E1	419.14	4.13	577.84	4.15	751.13	12.58

Table 3
Data set of NL grown on a thermally surface-cleaned sapphire substrate.

Sample	Part 1 (418 cm^{-1}) A_{1g}		Part 2 (578 cm^{-1})		Part 3 (751 cm^{-1})	
	Peak Position (cm^{-1})	FWHM (cm^{-1})	Peak Position (cm^{-1})	FWHM (cm^{-1})	Peak Position (cm^{-1})	FWHM (cm^{-1})
A2	419.12	4.21	577.81	4.13	751.47	11.78
B2	419.14	4.18	577.81	4.05	751.44	11.7
C2	416.74	4.87	577.81	4.28	751.45	12.11
D2	419.11	4.12	577.81	4.07	751.45	12.35
E2	419.14	4.19	577.83	4.07	751.45	11.97

(high), E_1 (TO), and A_1 (LO) modes of AlN were observed. As it is known, A_1(TO) mode is a forbidden mode [43]. It is not observed in films with perfectly (002) oriented grains. When the orientation of (002) is disrupted and other orientations are observed, the A_1 (TO) peak to the enhancement [43,44]. It is seen that both the peak intensity of the A_1(TO) mode and the distortions in the (002) orientation increase as the thermal cleaning temperature increases. A_1(LO) mode is present due to carrier concentrations (intentional/unintentional) [45,46]. It can be said that as the thermal surface cleaning temperature increases, the unwanted carrier concentration decreases, so this increase decreases the A_1(LO) peak density. With Raman's analysis, it is possible to have information about the strain in the films. E_2 (high) mode is considered in the stress analysis. It has been reported in the literature that the E_2 (high) phonon frequency of unstrained AlN is 657 cm^{-1} at 300 K [47,48]. Fig. 8 (b) shows the evolution of the AlN in-plane stress calculation from E_2 (high) peak frequency. While AlN growing, grain coalescence can cause tensile stress. Tensile stress is observed in films with small grain sizes, while compressive stress is observed in large grain sizes [49,50]. According to the data obtained from Fig. 8 (a), the E_2 (high) peak frequencies are 655.1, 658.2, 658.1, 658.8, and 659.3 cm^{-1} for Sample A, Sample B, Sample C, Sample D, and Sample E, respectively. As shown in Fig. 8 (b), Sample A has tensile stress, unlike other samples. Since Sample A has a smaller grain size, tensile stress is observed in it. Compressive stress is expected when the AlN film thickness is less than 1 μm [44,51] All of the samples in our study have almost the same thickness and are thinner than 1 μm. As can be seen from Fig. 8 (b), Sample C is the sample with the least stress.

4. Conclusions

In this study, in-situ thermal surface cleaning on the sapphire substrate was performed at different temperatures, and its effect on sapphire substrate, AlN NL, and AlN thin film was investigated in detail. It was observed that the FWHM values obtained from the XRD results of the sapphire were almost stable at 1245 °C and above. In parallel with the reported studies, it was noted that the sapphire undergoes thermal decomposition above 1200 °C. When the effect of temperature on the nucleation layer was examined, it was concluded that the sapphire surface was not completely cleaned or created mixed polarity due to the ineffective thermal decomposition on the sapphire surface at low temperature. Misoriented grains occur as a result etching of the sapphire surface at high temperatures. Thus FWHM values increased at temperatures above 1245 °C. When the effect of temperature on AlN thin film was examined, it was observed that an Al-rich sapphire surface appeared at high temperatures. It was thought that Sample C was less affected by etching at high temperature and also had a more Al-polar surface than samples cleaned at low temperature. The resulting RMS, FWHM for 2 theta/omega scans, and FWHM for omega scans were 0.234 nm, 171 arcsec, and 89 arcsec, respectively. It was observed that the transmittance of AlN thin films decreased due to the increase in roughness at high temperatures. Since the nucleation layer was very thin, no major changes were observed in Raman analysis, unlike sapphire. As a result of Raman analysis, it was noted that Sample C had homogeneous grain distribution and grains grown in a controlled manner with the effect of large grain size had less stress. When the results are evaluated in general, it is seen that the in-situ cleaning temperature of 1245 °C is the optimum temperature for MOVPE and AlN growths.

CRediT authorship contribution statement

Merve Nur Koçak: Writing – original draft, Investigation, Conceptualization. **Gamze Yolcu:** Writing – review & editing, Investigation. **Sabit Horoz:** Writing – review & editing, Investigation. **İsmail Altuntaş:** Investigation, Funding acquisition, Formal analysis. **İlkay Demir:** Writing – review & editing, Supervision, Project administration, Methodology, Funding acquisition.

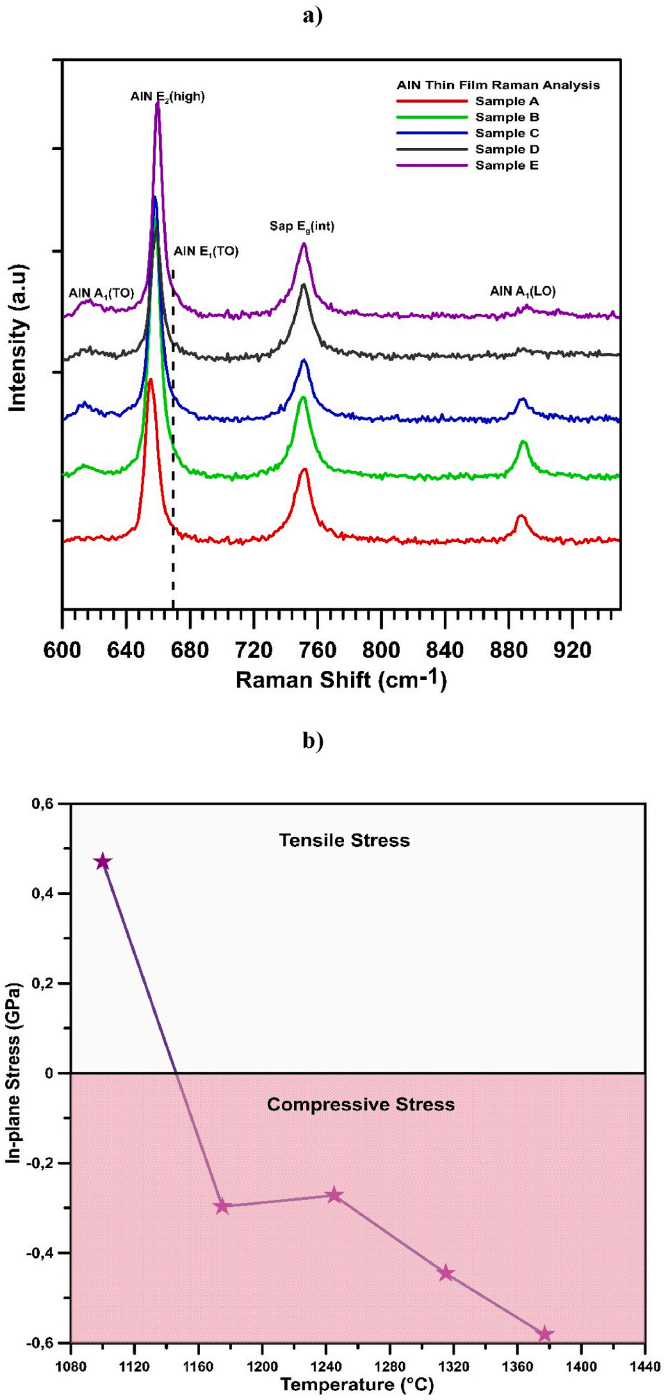

Fig. 8. **a)** Raman spectra of Samples A, B, C, D, and E b) Graph of Inplane-stress against thermal surface cleaning temperature.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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