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Effect of ammonia/gallium ratio and growth temperature towards the surface morphology of semi-polar GaN grown on m-plane sapphire via MOCVD

Kesan nisbah ammonia/gallium dan suhu pertumbuhan terhadap morfologi permukaan GaN semi-polar yang tumbuh pada safir m-pesawat melalui MOCVD

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Abstract

A single-crystalline semi-polar gallium nitride (11-22) was grown on m-plane (10-10) sapphire substrate by metal organic chemical vapor deposition. Three-step approach was introduced to investigate the grain size evolution for semi-polar (11-22) GaN. Such approach was achieved due to the optimized gallium to ammonia ratio and temperature variations, which led to high quality (11-22) oriented gallium nitride epilayers. The full width at half maximum values along (-1-123) and (1-100) planes for the overgrowth temperature of 1080°C were found to be as low as 0.37° and 0.49°, respectively. This was an indication of the enhanced coalescence and reduction in root mean square roughness as seen by atomic force microscopy. Surface analysis via atomic force microscopy indicated the orientation towards semi-polar plane. Field emission scanning electron microscopy analysis further indicates that higher temperature of 1080°C during the deposition of the overgrowth promoted closely packed surface coalescence. Room temperature Raman revealed that the overgrowth temperature of 1080°C portrayed compressive strain free as compared to other overgrowth temperature. Based on these results, the promising overgrowth temperature of 1080°C can be further utilized in future work for optoelectronics devices.

Keywords: Gallium nitride; m-plane; MOCVD; Semi-polar; Surface evolution

Abstrak

Satu kristal tunggal semi-polar gallium nitride (11-22) ditumbuh pada substrat nilam m-satah (10-10) oleh pemendapan wap kimia logam-organik. Pendekatan tiga-langkah diperkenalkan untuk menyelidiki evolusi ukuran zarah bagi semi-polar (11-22) GaN. Pendekatan sedemikian dicapai kerana nisbah V/III yang dioptimumkan dan variasi suhu yang menyebabkan epilayer gallium nitride berorientasi (11-22) berkualiti tinggi. Lebar penuh pada separuh nilai maksimum di sepanjang satah (-1-123) dan (1-100) untuk suhu pertumbuhan 1080 °C masing-masing didapati serendah 0,37 ° dan 0,49 °. Ini adalah petunjuk peningkatan gabungan dan pengurangan kekasaran kuadrat akar seperti yang dilihat oleh mikroskopi kekuatan atom. Analisis permukaan melalui mikroskop daya atom menunjukkan orientasi ke arah satah semi-polar. Analisis mikroskopi elektron pengimbasan pelepasan lapangan lebih jauh menunjukkan bahawa suhu 1080°C yang lebih tinggi semasa pemendapan pertumbuhan berlebihan mendorong penyatuan permukaan yang lebih baik. Suhu bilik Raman mendedahkan bahawa suhu pertumbuhan 1080 ° C menggambarkan regangan mampatan bebas berbanding dengan suhu pertumbuhan yang lain. Berdasarkan hasil ini, suhu pertumbuhan yang menjanjikan 1080 ° C dapat digunakan lebih lanjut di masa hadapan bagi aplikasi peranti optoelektronik.

Kata kunci: gallium nitride; satah-m; MOCVD; semi-polar; evolusi permukaan

INTRODUCTION

III-nitride semiconductors have been widely used to grow optoelectronic devices such as light emitting diodes (LEDs) and laser diodes (LDs) [1-3]. However, the c-plane GaN-based LEDs are greatly affected in the quantum efficiency towards longer emission wavelength [3-5]. This is mainly due to the optoelectronic devices suffering the undesirable quantum-confined Stark effect (QCSE) owing to their strong spontaneous and piezoelectric polarization issues [4-8]. There is a strong field induced-polarization that also causes the electrons and holes to separate from one another [9, 10]. This in turn leads to a rise in the recombination efficiency of restricted carrier, red-sifted emission as well as the strength of the reduced oscillator. In order to reduce the polarization effect, a method to produce non- and semi-polar GaN on m-plane sapphire was presented as an alternative [10].

To develop non-polar GaN, namely (11-20) a-plane or (10-10) m-plane orientation, significant achievements have been accomplished [3, 11-14]. However, the growth of the non-polar is challenging due to anisotropic surface characteristics leading to high defect density in the crystal structure. There is a possibility to either reduce or eliminate the polarization effect when the GaN was grown along the semi-polar plane [2, 13]. Unlike the non-polar plane, semi-polar plane considered easier to obtain over a wide range of the growth condition [13, 15-17]. Up to now, semi-polar GaN including the orientation of (11-22), (10-11) and (10-13) have been grown on different substrates such as spinal substrate [18], semi-polar GaN substrate [19], and m-plane substrate [20]. Among those approaches, m-plane substrates are the most suitable choice due to the preferred size (enabling larger wafer growth sizes) as well as cost effectiveness (considerably cheaper substrates) [21]. There are several approaches to produce semi-polar (11-22) GaN on m-plane sapphire substrate; Ki-Ryong et. al. has introduced a high temperature of three-step growth technique, which consisted of GaN seed film growth, I-STEP, and lateral growth step [20]. Other than that, Tim et. al. reported the growth of semi-polar GaN films on (10-10) m-plane sapphire substrate by MOVPE, comprising a two-step growth technique that yielded semi-polar GaN (2-1-12) oriented [21]. On the other hand, Ploch et. al. has acquired a polycrystalline semi-polar GaN on m-plane sapphire also using MOVPE by optimizing the nitridation time and temperature during growth growth [5]. All the different approaches regarding semi-polar GaN (11-22) showed that they achieved the semi-polar GaN growth. However, the variation of the overgrowth temperature and the V/III ratio towards the grain size distribution has yet to be studied in detail. In this work, a three-step growth of a semi-polar GaN (11-22) consisting of, GaN-nucleation layer (NL), GaN-buffer layer (BL) and overgrowth GaN epilayer (OG) is presented. The optimization of V/III ratios were investigated during the deposition of NL. Finally, the effect of the OG temperature of semi-polar GaN towards the improvement of crystal quality and surface morphology was investigated, portraying enhancement in the grain size distribution.

MATERIALS AND METHODS

The growth of semi-polar GaN (11-22) on m-plane sapphire (10-10) was carried out by horizontal metalorganic chemical vapor deposition (MOCVD) (SR-2000, Taiyo Nippon Sanso, Japan). Trimethylgallium (TMG_1) and ammonia (NH_3) were used as the precursors for gallium as well as nitrogen sources, respectively. As it can be seen from the schematic diagram in Figure 1, prior to the growth, the m-plane (10-10) sapphire were exposed to a heat treatment at 1125°C in the ambiance of H_2 to remove contamination (step 1). The initial step of the growth was nitridation, followed by the deposition of the low temperature GaN-NL at 400°C (step 2). The V/III ratios were varied from 2000 to 4000 by changing the NH_3 flow rate, while TMG_1 was maintained along the epitaxial growth. Then, a recrystallization process was performed, followed by the deposition of the GaN-BL at 800°C with a pressure of to 25 KPa (step 3). The GaN-BL thin film was grown, followed by the growth of uid-GaN with a varied temperature of 1030°C to 1080°C (step 4). To investigate the crystal quality high-resolution x-ray diffraction (HR-XRD), including 2θ-ω and X-ray rocking curve (XRC) scans. Room temperature Raman spectroscopy was utilized to further analyze the structural properties of semi-polar GaN-OG, mainly the strain relaxation effect and the phase purity. Finally, atomic force microscopy (AFM) and field emission scanning electron microscopy (FESEM) were employed to analyze the surface morphology as well as demonstrate the grain size evolutions.

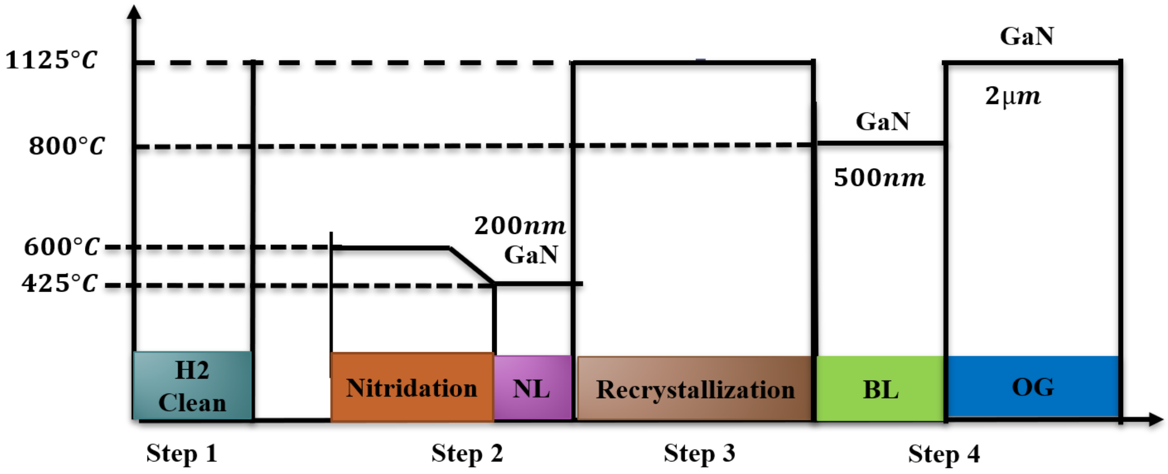

Figure1: Flowchart of the growth condition with schematic illustration of layer-by-layer deposition

RESULTS AND DISCUSSION

A. Nucleation layer

Figure 2 (a, b) shows the HR-XRD 2θ-ω of semi-polar GaN-NL for different V/III ratios. HR-XRD 2θ-ω confirms the semi-polar GaN epilayer along (11–22) plane grown on m-plane (10–10) sapphire substrate. The peak at 68.2° is corresponding to semi-polar (11-22) GaN, while the peak at 63,4 is assigned to semi-polar GaN (10-13). During the nitridation process, the (10-13) plane is suppressed, so that a single crystalline semi-polar (11-22) thin film is produced. Based on previous studies, it was seen that when the nitridation process is not accurately applied to the m-sapphire, the as-grown GaN often exhibits both (11-22) and (10-13) orientation called twinned grains [22]. Due to such occurrence, the nitridation process will break the symmetry of the m-plane surface; hence, the boundaries of the grain will prevent a complete coalescence [17, 23]. Consequently, one peak related to the semi-polar GaN (11-22) oriented is observed with high V/III ratio (4000), while the peak of (10-13) is suppressed. However, the samples with lower V/III ratios, namely 2000 and 3000 exhibits polycrystalline structure, in which the diffraction peak of semi-polar (10-13) GaN exists at 63.4°. This is mainly due to the twining grains formation, enduring polycrystalline structure [5, 17].

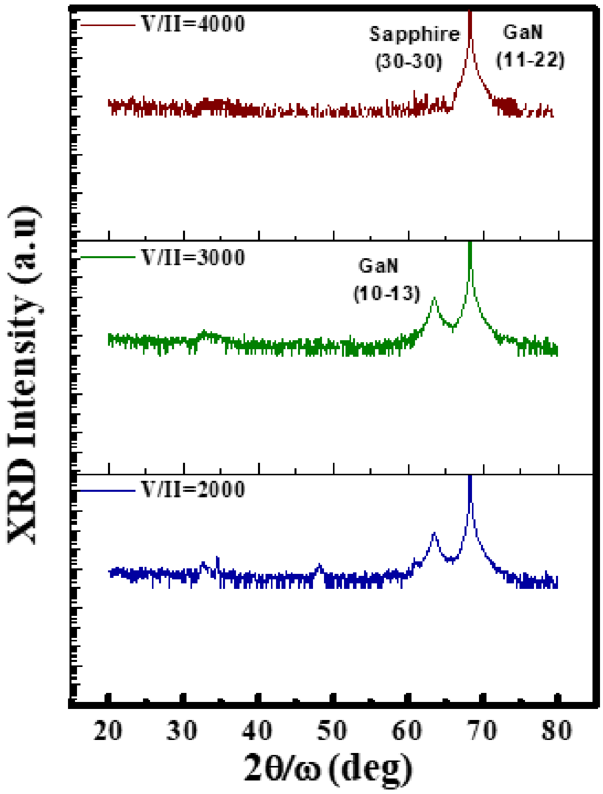

Figure 2: 2θ -ω scan profile of the symmetric plane of semi-polar GaN-NL on for different V/III ratios

Figure 3 (a-f) shows the surface analysis of semi-polar GaN-NL using different V/III ratios of 2000, 3000, and 4000, respectively. Figure 3 (a-c) demonstrates images of top-view FESEM of semi-polar GaN-NL of 2000, 3000, and 4000, respectively. While, Figure 3 (d-f) illustrates the typical 5 × 5 µm AFM 2D AFM images of semi-polar GaN-NL of 2000, 3000, and 4000, respectively. It can be seen from the FESEM analysis that the finest coalescence is obtained using the V/III ratio of 4000, while Figure 3 (a, b) exhibited irregular surface features. Thus, this can be indicated by the crystal defects, resulted from the rough surface. However, upon increasing the V/III ratio (c), the crystal irregularities were reduced, leading to semi-polar (11-22) GaN formation as it can be seen in Figure 3 (c). In addition, AFM analysis portrayed that the slate-like patterns which aligned with the c-plane direction occurred at lower V/III ratios (2000 and 3000) as shown in Figure (d) and (e). On the contrary, the 4000 of V/III ratio exhibited slate-like patterns aligned to the orientation of the semi-polar (11-22) orientation. Thus, a smoother structure is obtained due to the dominant orientation of (11-22) with strip-like grains

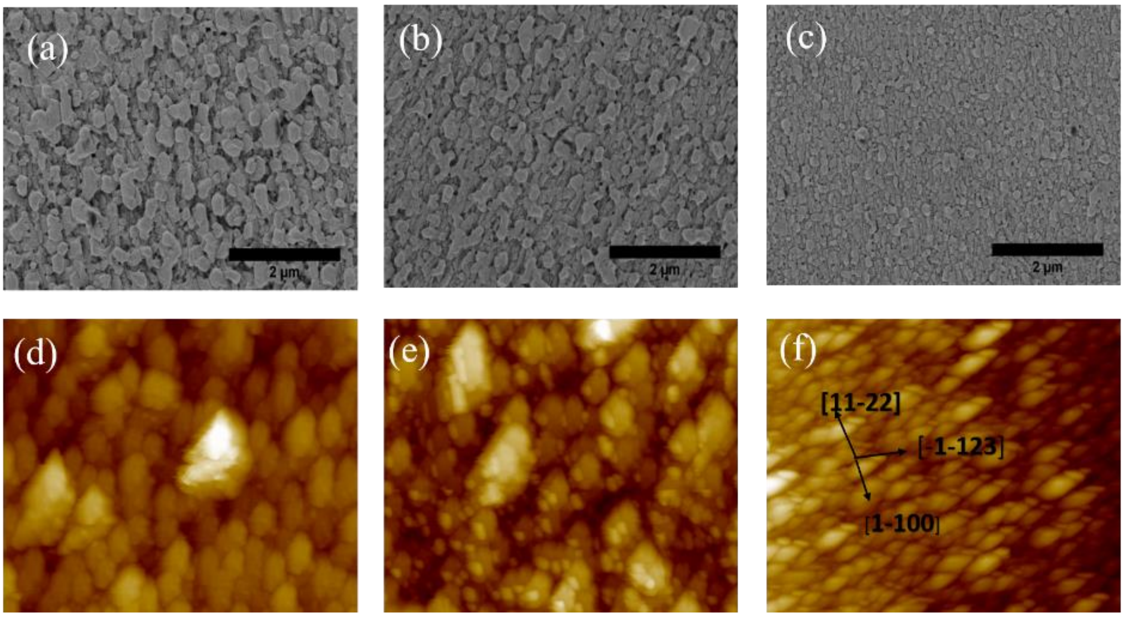

Figure 3: Surface analysis of the semi-polar GaN for V/III ratio of (a, d) 2000 (b, e) 3000 and (c, f) 4000.

It is presumed that in case of dominant orientation of (10-13), twinning can be observed leading to rough surface, inducing a correlation between the surface analysis and HR-XRD scan. Furthermore, it can be clearly seen that the epitaxial layers grown using low V/III ration exhibit~~s~~ a distinct structural difference as compared to the corresponding epitaxial layer using high V/III. Therefore, it is evidenced that a high V/III ratio is beneficial to improve the crystal quality, which in turn reduces the closely packed grain distribution. It can be noted that the root mean square (RMS) roughness was as low as 8.3 nm when the V/III was increased up to 4000. RMS can be defined as the standard deviation in relation to the height of average surface that is utilized to measure either the spatial difference or the temporal changes of the surface morphology. The arithmetic average (R_a) and the peak-to-valley (P2V) value were also decreased when the V/III ratio is increased. However, the samples with lower V/III ratios, namely 2000 and 3000 (d, e) promoted rough structure and polycrystalline structure, which is correlated with HR-XRD results. Finally, to have a better view of surface morphology of semi-polar GaN-NL, the values of RMS roughness, R_a and P2V of nucleation layer are tabulated in Table 1.

Table 1: RMS roughness, R_a and peak-to-valley of different V/III

V/III	**RMS Roughness (nm)**	**Peak to Valley (nm)**	**R_a (nm)**
4000	8.3	46.5	6.7
3000	18.4	73	13.4
2000	27.9	81	14.7

B. Overgrowth

Raman analysis was employed to investigate the phase purity as well as strain relaxation effect of semi-polar (11-22) GaN for different overgrowth temperature as shown in Figure 4. Three obvious sapphire peaks occurred in all samples, located at 377.5, 415.8, and 742.2 cm-1 [17, 39], denoted as (*). Raman analysis illustrates presence of peaks of E_2-high, and E_1 (TO) phonon line, which are well-correlated with the previous studies [24-27]. The Raman spectra are acquired from the back-scattering configuration, in which the light of the laser is incident along the normal direction of the growth. The Raman peaks of the substrate are designated by the symbol (*). From the Raman spectra, E_2-high mode can be utilized to analyze the in-plane strain and the crystal quality of the semi-polar GaN film. It can be clearly observed that the peak of E_2-high mode for the overgrowth temperature of 1080°C shows higher intensities compared to lower overgrowth temperatures. This is owing to the sensitivity of the strain to the GaN film; whereas, the overgrowth temperature of 1080°C shows an enhanced crystallinity as compared to the lower overgrowth temperature. Thus, it is obviously discerned that the high intense peak of E_2-high mode can be achieved when the semi-polar GaN is grown at 1080°C. In addition, the position of the E_2-high peak is at 568.8 cm^{-1}, by which the strain relaxation effect is confirmed [25, 28]. However, lower growth temperature exhibits compressive strain, portraying the peak position of E_2-high mode at 567.0 cm^{-1}.

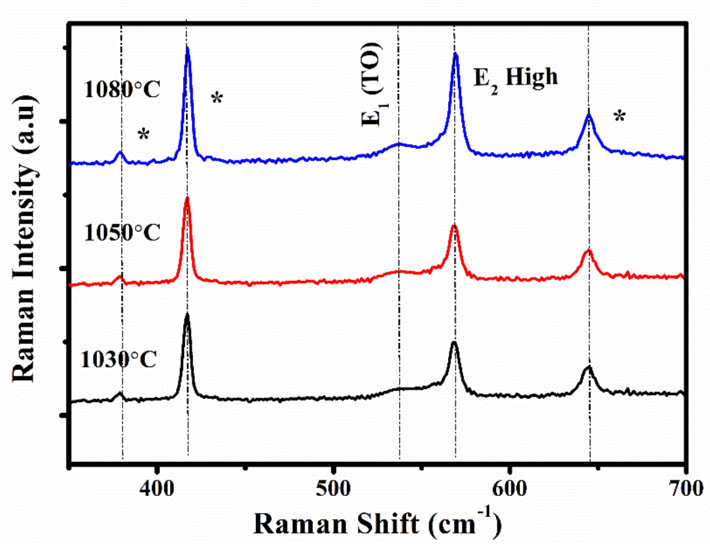

Figure 4: Raman spectra of different growth temperature for semi-polar (11-22) GaN

The measurement of XRC was implemented to reveal the comprehensive knowledge of the microstructure of semi-polar (11-22) GaN and the correlation to the growth dynamics. The measurement of XRC was accomplished for three different overgrowth temperatures along the symmetrical on-axis [11-22] direction as a function of overgrowth temperature as shown in Figure 5 (a-c). When the incident beam projection is parallel to the direction of [-1-123] GaN, the azimuth angle is defined as zero degree. The angle of rotation with the respect to the surface normal is φ, while the angle of sample tilted around the axis, which is formed by the intersection of the Bragg and scattering planes, is *x*. The φ angle was set at 0° and 90°, by which the incident beam directions of [-1-123] and [1-100], respectively as illustrated in Figure 5 (c). For the incident beam of [-1-123], the full widths at half maximum (FWHM) values for 2 µm overgrowth temperature of 1030°C, 1050°C and 1080°C were 0.86°, 0.52°, and 0.37°, respectively. While, the values of the FWHM for the incident beam of [1-100] were 1.2°, 0.51°, and 0.49°, respectively.

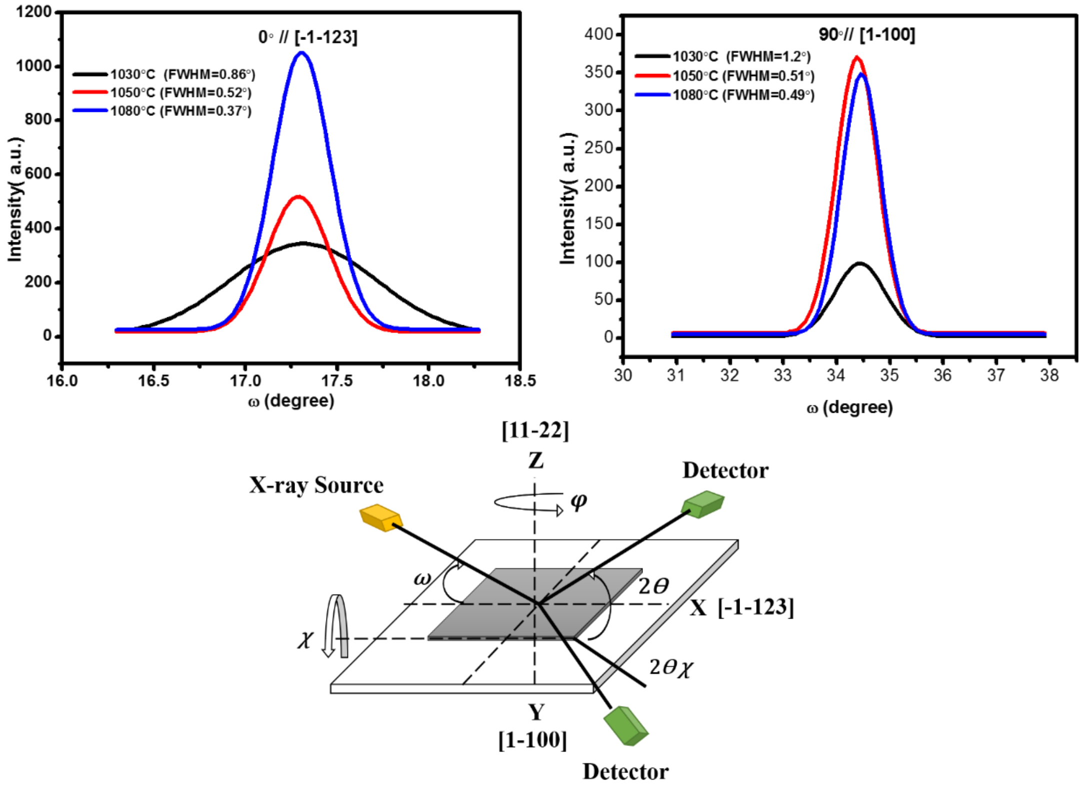

Figure 5: XRD ω rocking curves of semi-polar (11–22) GaN for different overgrowth temperatures in two azimuth angles: (a): 90°// [1-100] and (b): 0°// [-1-123]. (c) Geometry of the azimuthal dependent XRD.

The sample of 1080°C overgrowth temperature exhibits lower FWHM compared to other samples recorded in this study. It is deduced that the presence of the partial dislocation, larger mosaic tilt and/or a reduced coherent length, the peaks are broadened in lower overgrowth temperature [8, 17, 29]. Basically, it is evidenced that the crystal quality of semi-polar GaN was enhanced by changing the overgrowth temperature to 1080°C owing to coalescence of dislocation. Therefore, it is believed that the overgrowth temperature is a critical parameter to overcome the defect in the surface. The FWHMs of the semi-polar GaN films grown on free-standing semi-polar GaN substrates have lower value, by which it is difficult to compete [22, 23, 30]. However, the free-standing semi-polar GaN substrates are considered costly, in which it is impractical for mass production. Thus, it can be considered suitable for the performance of the nitride based-optoelectronic device.

Figure 6 (a-f) shows the surface analysis of semi-polar GaN-OG on m-plane sapphire substrate for different overgrowth temperature, namely 1030°C, 1050°C and 1080°C, respectively. In the face scanning of FESEM, Figure 6 (a, b), semi-polar (11-22) GaN-OG with a growth temperature has no flat surface, portraying polycrystalline 3-D structure. This is mainly due to the anisotropic surface structure; the structure might be affected by the crystallographic difference between semi-polar (11-22) GaN and m-plane sapphire [17, 31]. This is resulted from the incorporation probability and the diffusion length of surface atoms with different crystallographic direction [21, 28]. Nonetheless, when the semi-polar GaN-OG was at 1080°C, a coalescence was greatly enhanced as it can be observed in Figure 5 (c). The significant role to reduce the pronounced 3-D structure of the GaN epilayers is the growth temperature; whereas higher temperature promotes rate of higher GaN desorption. At lower temperature, it results in reduced surface atomic diffusion of the Ga and N atoms; hence, leads to structural defects [20].

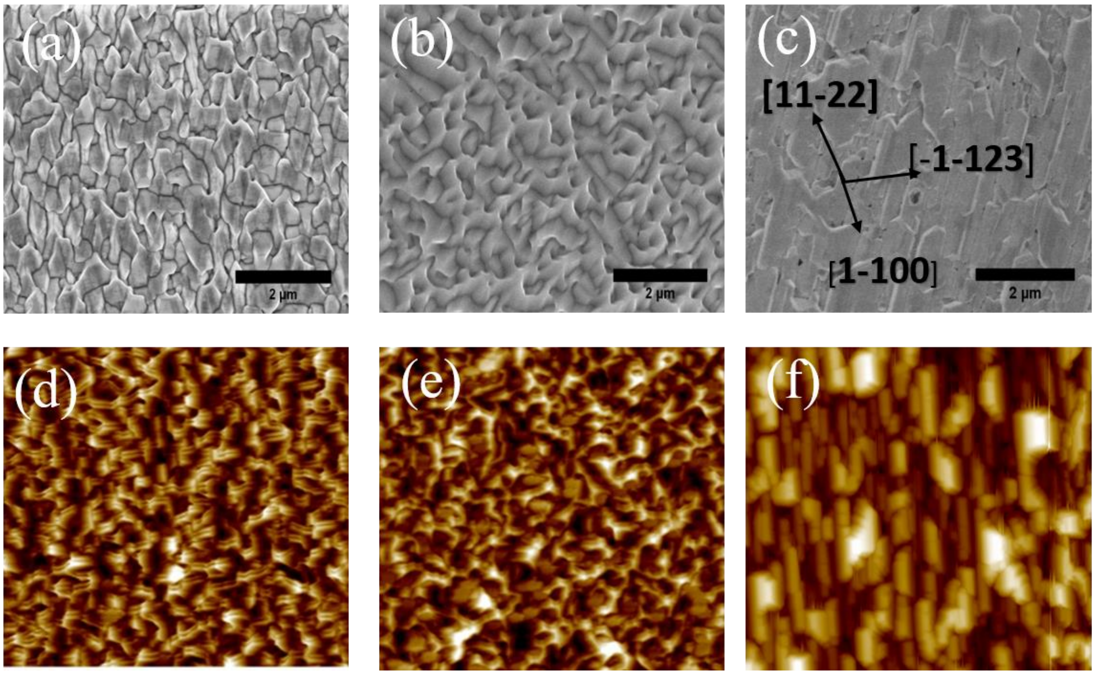

Figure 6: Surface analysis of the semi-polar GaN-OG for different overgrowth temperature at (a, d) 1030°C, (b, e) 1050°C and (c, f) 1080°C

Figure 6 (d-f) illustrates the 2D images of AFM measurement of semi-polar GaN-OG on m-plane sapphire substrate for different overgrowth temperatures. The surface morphology of semi-polar (11-22) GaN-OG suffered a rough surface owning to the anisotropic lattice mismatch between semi-polar (11-22) GaN and m-sapphire [21]. However, upon increasing the growth temperature, the surface morphology and the grain size distribution were enhanced, leading to closely packed feature as compared to lower growth temperature enhanced as it can be seen in Figure (f). The RMS roughness of semi-polar (11-22) GaN-OG was decreased from 66.02 to 22.9 nm by increasing the growth temperature.

CONCLUSION

A 3-step approach of semi-polar gallium nitride-epitaxial layers grown on m-plane sapphire has been accomplished via metalorganic chemical vapor deposition. The ammonia/gallim ratio was varied to improve the surface quality. The dominant orientations were (11-22) and (30-30), which are the semi-polar gallium nitride and m-plane sapphire orientations, respectively. Further, the atomic force microscopy images of the nucleation layer proved that the film of a high V/III was found to be more uniform and homogenous compared to others ratios, in which smallest grain size acquired (peak to valley value) is 46.5 nm. The RMS roughness of GaN-epitaxial layers was as low as 8.3 nm using 5×5 μm^2 AFM. In addition, the RMS roughness of the overgrowth temperature was decreased from 66.02 to 22.9 when overgrowth temperature was at 1080°C. The sample of overgrowth temperature at 1080°C has achieved an improved coalescence as shown via the FESEM measurement. Raman spectroscopy showed that the sample grown at 1080^{o}C portrayed strain relaxation state as compared to lower growth temperature. It should be also noted the overgrowth temperature of 1080°C can be further used in future work for optoelectronics devices.

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