

## Growth of Semi-insulating GaN Layer by Controlling Size of Nucleation Sites for SAW Device Applications

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Semi-insulating undoped GaN films were grown based on controlling the size of the nucleation sites through a special two-step growth method: First, 16 nm LT-GaN was annealed at 950 ° with a ramping time of 4 min, then the GaN was grown at this temperature for 1 min. Second, the growth temperature was increased to 1020° with a ramping time of 2 min and the GaN layer finally grown at 1020 ° for 40 min. The film grown by this sequence exhibited sheet resistance of up to 10<sup>9</sup> Ω/sq with mirror-like surface morphology. By slow ramping to 950° in the initial phase of growth, smaller grain sizes and higher nuclei densities were formed and the columnar growth mode along the c direction was dominant. The observation of higher resistance in two-step growth is believed due to the increased misorientation of nuclei when the growth proceeds during temperature ramping to 1020°. The fabricated saw filter on semi-insulating GaN exhibited a high velocity of 5342 m/s at center frequencies of 133.57 MHz and an electromechanical coupling coefficient( $k^2$ ) of about 0.763 %, which was enhanced due to the improvement of surface morphology with high sheet resistance by the two- step ramping technique.

### 1 Introduction

GaN and related III-nitrides are very promising materials for the fabrication of optoelectronic devices [1] and high power and high temperature electronic devices [2] [3]. AlGaIn/GaN heterostructure field effect transistors (HFETs) have also exhibited great potential for high-frequency and high-power applications due to such advantages as a high breakdown voltage and high electron peak velocity. III-nitride materials are also suited for application in high temperature piezoelectronics, pyroelectric sensors, and surface acoustic wave (SAW) devices due to their inherently strong lattice polarization effects [4] [5] [6] [7]. Since an undoped GaN layer exhibits a high electrical conductivity resulting from residual donors unintentionally introduced during growth, there are a few reports on SAW devices fabricated on epitaxially grown undoped GaN thin films revealing an increased insertion loss, which is a fatal disadvantage to realizing good SAW devices. Semi-insulating GaN films for electronic devices are usually

doped with Mg, Zn, C, and Fe to compensate for any free carriers [8] [9] [10]. A successful preparation of highly resistive undoped GaN films was recently reported based on simply adjusting growth parameters such as the III/V ratio, growth pressure, growth temperature, and annealing time [11] [12] [13]. Accordingly, the current study also reports on the growth of semi-insulating undoped GaN films based on controlling the size of the nucleation sites through a special two-step growth method. A high electromechanical coefficient was demonstrated in the fabricated SAW filter on semi-insulating undoped GaN.

### 2 Experiments

Undoped GaN films were grown on a (0001) sapphire substrate using an EMCORE D125 low-pressure rotating-disk metal organic chemical vapor deposition (MOCVD) reactor. Trimethylgallium (TMGa) and high-purity ammonia (NH<sub>3</sub>) were used as the precursors for the Ga and N, respectively. High purity hydrogen (H<sub>2</sub>) was used as both the carrier gas and the flow to supple-

ment the total flow rate required for maintaining a well-matched laminar flow pattern in the reactor. The total gas pressure during the growth was set at 300 Torr and the spinning rate of the substrate was about 1000 rpm. Prior to the epilayer growth, the wafers were cleaned by  $H_2$  ambient at  $1020^\circ$ , then a 16 nm-thick low temperature GaN buffer layer was grown at  $550^\circ$ . After the growth of the buffer layer, the substrate temperature was annealed at three different temperatures of 950, 1020, and  $1050^\circ$  with a ramping time of 4 min when increasing the temperature from  $550^\circ$  to each of the annealing temperatures. Five different undoped GaN samples were grown on a 16 nm-thick LT-GaN buffer at temperatures of 950, 980, 1000, 1020, and  $1050^\circ$  for 40 min. During the growth, the typical flow rates were maintained at 9/12 slpm for  $NH_3/H_2$  and 170  $\mu\text{mol}/\text{min}$  for TMGa. The surface morphology of the layers was measured by both atomic force microscopy (AFM) and scanning electron microscopy (SEM). The electrical and optical properties were measured by a Hall-effect measurement and photoluminescence (PL), while the crystal quality of the films was determined by high-resolution X-ray diffraction (HR-XRD). The frequency response of the SAW filters fabricated on the undoped GaN layer was measured using an HP 8753D network analyzer.

### 3 Results and Discussion

Figure 1 shows SEM and AFM images of the as-grown and annealed LT-buffer GaN at different temperatures. The as-grown LT-buffer layer is shown in Figure 1(a), while three subsequent LT-buffer layers were annealed at (b)  $950^\circ$ , (c)  $1020^\circ$ , and (d)  $1050^\circ$ . The SEM and AFM images show a gradual increase in the polycrystalline island size with an increasing annealing temperature. The values of the root mean square (rms) of the surface roughness were (a) 2.4 nm, (b) 7.1 nm, (c) 22 nm, and (d) 24 nm, respectively. With a relatively thin LT-buffer layer thickness (16 nm), Ga species become more mobile on a sapphire surface with an increasing temperature and form larger polycrystalline islands [14]. Figure 2 shows the HRXRD diffraction data for the same samples presented in Figure 1. The FWHM values for the X-ray rocking curve were (a) 13786 arcsec, (b) 8420 arcsec, (c) 2714 arcsec, and (d) 2310 arcsec, respectively. A broader rocking curve was observed for the as-deposited sample and sample ramped up to  $950^\circ$  based on 4 min. However, when the annealing temperature was increased above  $1020^\circ$ , the single-crystalline GaN quality was improved. Figure 3 shows the PL spectra at 10 K for the samples presented in Figure 1. As mentioned above, no near band edge peak at 365 nm was observed for the as-grown sample and sample annealed up to  $950^\circ$  based on 4 min, yet when the annealing temperature was increased, the intensity of

the near band edge emission peak also increased. As such, these results indicate a change in the crystallinity of the LT-GaN layer, from amorphous to single crystal, during the ramping process.

Figure 4 shows the results of the sheet resistance and electron mobility for undoped GaN films grown at various temperatures for 40 min. The value of the sheet resistance greatly increased from  $\sim 10^3$  to  $\sim 10^6 \Omega/\text{sq}$  with decreasing growth temperature, while the electron mobility also significantly decreased from 250 to 4  $\text{cm}^2/\text{Vs}$ . Free carrier concentration was reduced from  $5 \times 10^{16} \text{ cm}^{-3}$  ( $\sim 5 \times 10^{12} \text{ cm}^{-2}$ ) for the 1.7  $\mu\text{m}$ -GaN films grown on sapphire substrate at  $1020^\circ$  to  $1 \times 10^{16} \text{ cm}^{-3}$  ( $\sim 1 \times 10^{12} \text{ cm}^{-2}$ ) for the sample grown at  $950^\circ$ . When the ramping temperature was slowly increased from 550 to  $950^\circ$  based on 4 min, smaller grain sizes and a higher nuclei density were observed, as shown in Figure 1(b). Since the recrystallization sites are not large enough and misorientation of the nuclei occurs in the direction of the a or c axis with a slower and lower ramping temperature, an HT-GaN layer grown under these conditions contains high density dislocations and stacking faults [15]. Thus, the high sheet resistance was believed to be due to generation of various point defect centers at the coalescence boundaries as a result of a small nucleation size in the initial growth stage. In contrast, larger grain sizes and a lower nuclei density, as shown in Figure 1c and Figure 1d, allowed more room for individual islands to develop before coalescence. Although the larger intermediate islands resulted in a rougher surface morphology, the increased average volume of defect-free columnar domains and lower density of coalescence boundaries eventually produced improved structural and electrical properties [15], as shown in Figure 4. When using a typical single-step growth method, a high sheet resistance was obtained at a relatively low growth temperature near  $950^\circ$ , yet the surface morphology of the resultant film was shown to be 3-D in fashion, as shown in Figure 5. However, when the epilayer growth temperature was increased above  $1000^\circ$ , a smooth surface resulted from the high thermal energy. Thus, to obtain a smooth surface with a high sheet resistance, a special two-step growth was implemented to resolve the problem encountered with single-step growth.

Figure 6 shows the two-step growth procedure. First, 16 nm LT-GaN was annealed at  $950^\circ$  with a ramping time of 4 min, then GaN was grown at this temperature for 1 min. Second, the growth temperature was increased to  $1020^\circ$  with a ramping time of 2 min and the GaN layer was finally grown at  $1020^\circ$  for 40 min to improve the crystal quality. The film grown using this sequence turned out to be semi-insulating ( $> 10^9 \Omega/\text{sq}$ )

with a mirror-like surface morphology (rms: 8 nm), as shown by the inset in Figure 6. The observation of high resistance is believed to be due to this initial sequence forming deep trap levels in the band gap [16] [17].

To investigate the growth procedure of highly resistive GaN, figure 7 shows SEM and AFM images of a sample (a) grown at 1020° for 3 min based on typical one-step growth and a sample (b) grown at 950° for 1 min, then at an increasing temperature up to 1020° for 2 min based on the proposed two-step growth. The rms values for the surface roughness were 36 nm for the one-step growth and 2.67 nm for the two-step growth. When compared to the one-step growth, the surface roughness of the two-step growth was improved due to the small and numerous grain sizes developed during the initial growth at a relatively low temperature. This implies that smaller grain sizes were formed and the columnar growth mode along the *c* direction was dominant during the initial low temperature ramping and low temperature growth. Thereafter, misorientation of the nuclei in the direction of the *a* or *c* axis was promoted due to GaN growth during the temperature ramping from 950 to 1020°. Figure 8 shows cross-sectional TEM images under  $g=0002$  two-beam conditions proven sensitive to screw threading dislocation centers for samples grown a) by one-step process at 1020° and b) by two-step process from 950 to 1020°. The screw threading dislocation density near the interface between the low-temperature buffer and overlayer was estimated to be roughly  $10^9$  cm<sup>-2</sup> for the one-step growth and roughly  $10^{10}$  cm<sup>-2</sup> for the two-step growth, respectively. Thus, the observed threading dislocation density of the two GaN samples was not high enough to capture all of the calculated average carrier density of  $\sim 10^{12}$  cm<sup>-2</sup>. However, by considering multiple electron traps around a dislocation center and/or other electron traps incarnated into the GaN layers by the presence of the interfacial threading dislocations [16] [17], we speculate that, since the one-step sample with threading dislocation density of  $\sim 10^9$  cm<sup>-2</sup> indicates an average carrier density of  $\sim 10^{12}$  cm<sup>-2</sup> resulting in a sheet resistance of  $10^6$  Ω/sq, the two-step sample with a dislocation density of  $\sim 10^{10}$  cm<sup>-2</sup> would also result in a high resistance over  $10^9$  Ω/sq. Well-oriented investigations for the origin of probable electron traps should be made to systematically produce this highly resistive GaN layer.

Figure 9 shows the PL spectra at 10 K for the samples grown based on one-step growth and two-step growth for 3 min, as shown in Figure 7. The PL intensity of the one-step sample increased approximately 17 times compared to that of the two-step sample, indicating that the two-step procedure generated nonradiative

recombination centers. In the case of epilayer growth for 40 min, the PL intensity of the one-step sample also increased approximately 10 times compared to that of the two-step sample, as shown by the inset in Fig 9. As such, these results show that the semi-insulating GaN was acquired from compensating free carriers resulting from the many acceptor-like point defects (associated with Ga vacancy) generated at the relatively low ramping and growth temperature and change in growth temperature from 950 to 1020° based on 3 min for epilayer growth [16] [17].

Figure 10 shows the frequency response of a 1.7 μm-thick undoped GaN SAW filter with an interdigital transducer (IDT) including 82 pairs of single electrodes ( $\lambda = 40$  μm) [7]. The wave propagation velocities for the undoped GaN film grown at 1020° with a relatively low sheet resistance ( $\sim 3 \times 10^3$  Ω/sq) and semi-insulating GaN films grown based on the proposed two-step growth with a very high sheet resistance ( $> 10^9$  Ω/sq) were calculated to be 5286 and 5342 m/s at center frequencies of 132.15 and 133.57 MHz, respectively. The calculated electromechanical coupling coefficient [7],  $K^2$  was about 0.049 % for the undoped GaN with a relatively low sheet resistance, whereas for the semi-insulating GaN film grown based on the proposed two-step growth,  $K^2$  increased to about 0.763 % due to a significantly reduced insertion loss. The good performance of fabricated SAW device could be explained by the improvement of surface morphology, which is important for contacts on the electromechanical devices with high sheet resistance through special two step growth.

#### 4 Conclusions

To obtain semi-insulating GaN films, a two-step growth method was developed based on controlling the nucleation sizes. The films grown using this sequence were shown to be semi-insulating ( $> 10^9$  Ω/sq) with a mirror-like surface morphology. During the initial low temperature ramping and low temperature growth, smaller grain sizes and higher nuclei densities were formed and the columnar growth mode along the *c* direction was dominant. The observation of a high resistance was believed to result from the misorientation of the nuclei in the direction of the *a* or *c* axis that occurred when the GaN film grew during the temperature ramping from 950 to 1020° during 3 min due to the initial sequence formed deep trap levels in the bandgap. A SAW filter fabricated using the semi-insulating GaN exhibited highly promising properties, including a high velocity of 5342 m/s at a center frequency of 133.57 MHz and electromechanical coupling coefficient ( $k^2$ ) of about 0.763 %. These superior SAW characteristics are believed to be due to both

the high resistance and the improvement of surface morphology by the two-step ramping method.

## ACKNOWLEDGMENTS

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## FIGURES

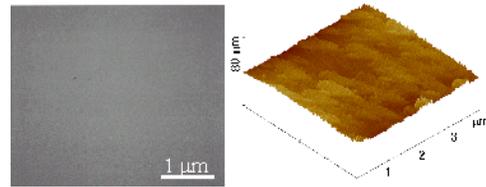


Figure 1a. SEM and AFM images of as-grown and LT-buffer GaN for an annealing temperature of initial buffer at 550° (RMS: 2.4 nm).

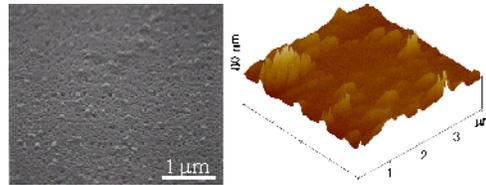


Figure 1b. SEM and AFM images of as-grown and LT-buffer GaN for an annealing temperature of 950° (RMS: 7.1 nm).

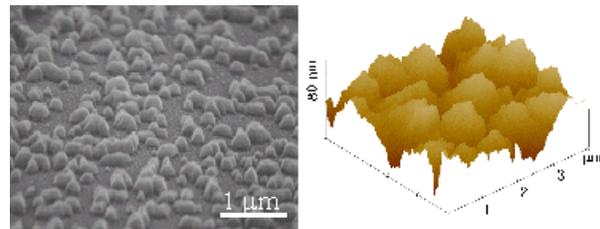


Figure 1c. SEM and AFM images of as-grown and LT-buffer GaN for an annealing temperature of 1020° (RMS: 22 nm).

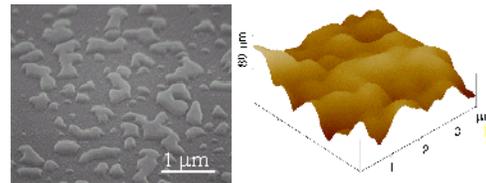


Figure 1d. SEM and AFM images of as-grown and LT-buffer GaN for an annealing temperature of 1050° (RMS: 24 nm)

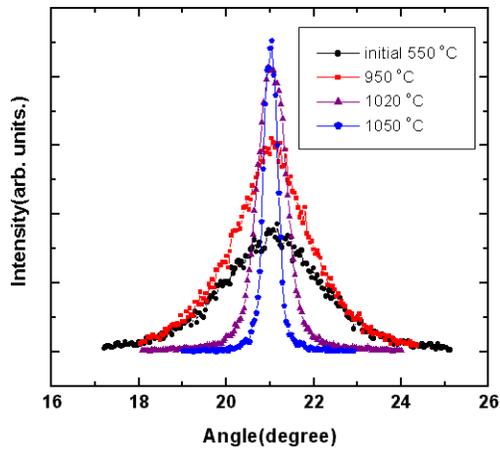


Figure 2. HRXRD diffraction data for as-grown and LT-buffer GaN at different annealing temperatures: initial buffer at 550°, 950°, 1020°, and 1050°

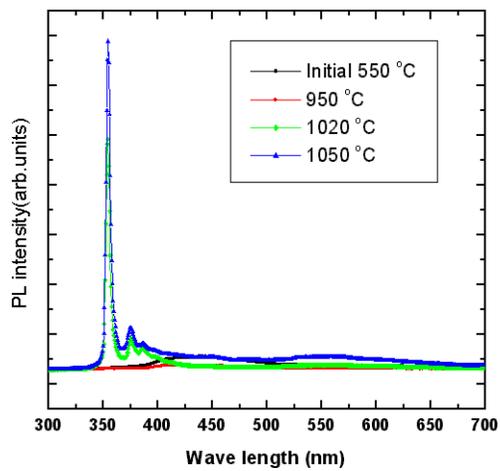


Figure 3. PL spectra at 10 K for as-grown and LT-buffer GaN at different annealing temperatures: initial buffer at 550°, 950°, 1020°, and 1050°

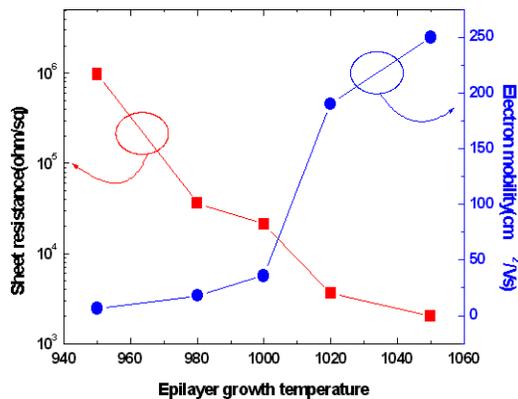


Figure 4. Sheet resistance and electron mobility of undoped GaN films grown at various temperatures: 950°, 980°, 1000°, 1020°, and 1050°

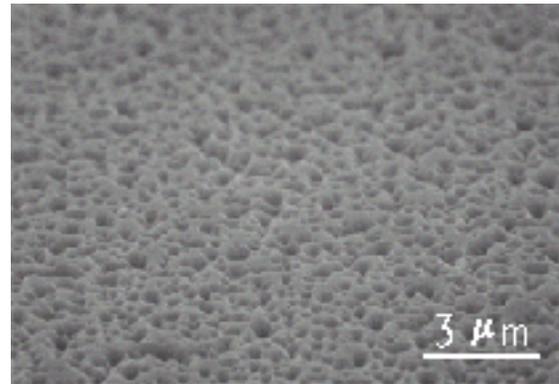


Figure 5a. SEM photographs of undoped GaN films grown at 950°.

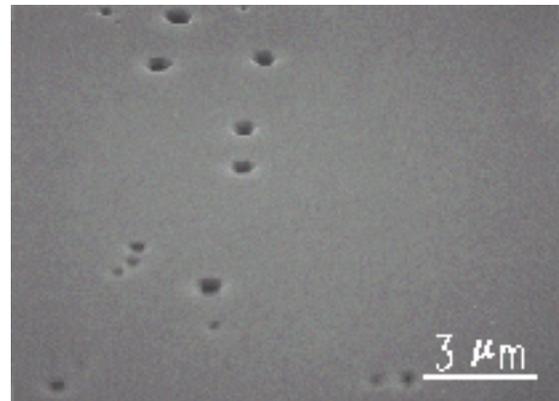


Figure 5b. SEM photographs of undoped GaN films grown at 980°.

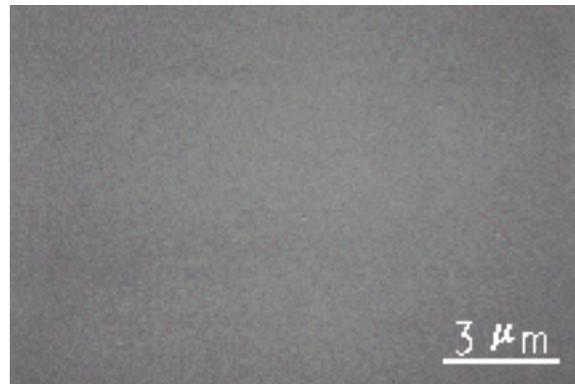


Figure 5c. SEM photographs of undoped GaN films grown at 1000°.

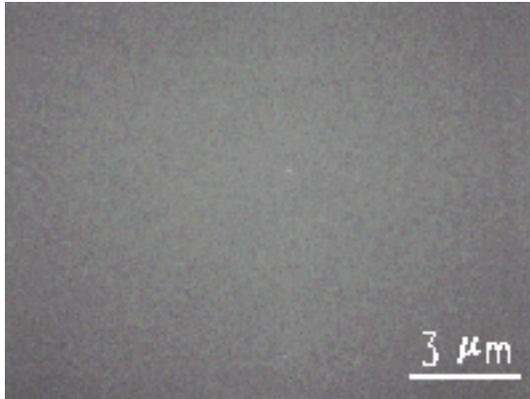


Figure 5d. SEM photographs of undoped GaN films grown at 1020°.



Figure 5e. SEM photographs of undoped GaN films grown at 1050°.

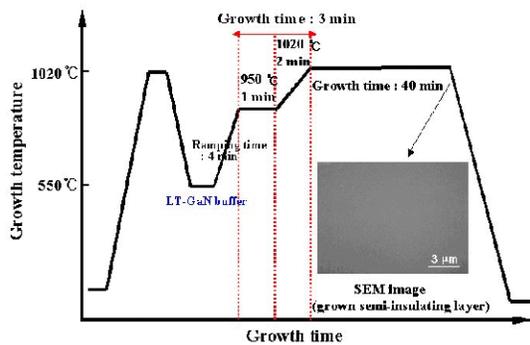


Figure 6. Proposed two-step growth procedure. Inset shows SEM photographs of semi-insulating GaN grown based on two-step growth.

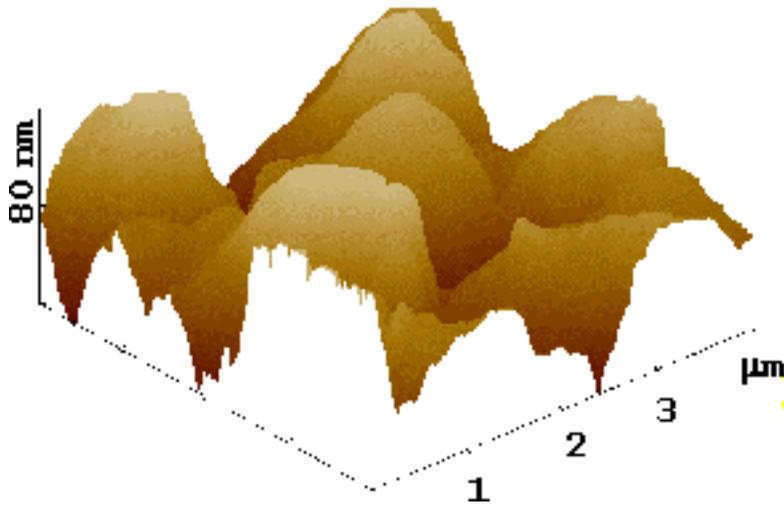
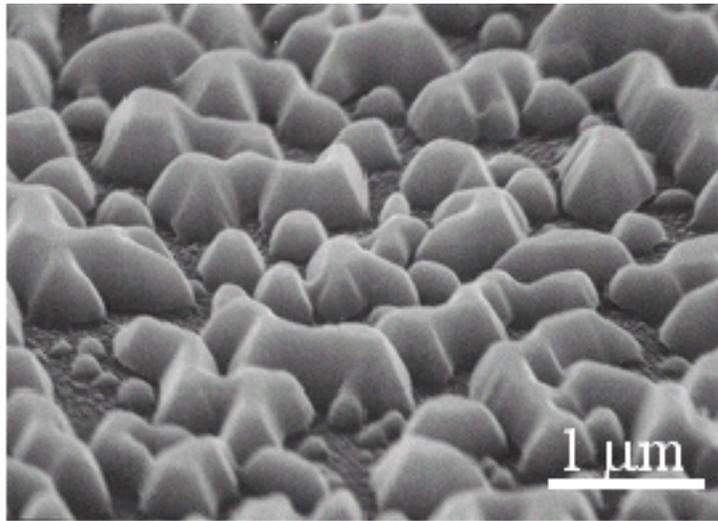


Figure 7a. SEM and AFM images of a sample grown at 1020 ° for 3 min based on typical one-step growth..

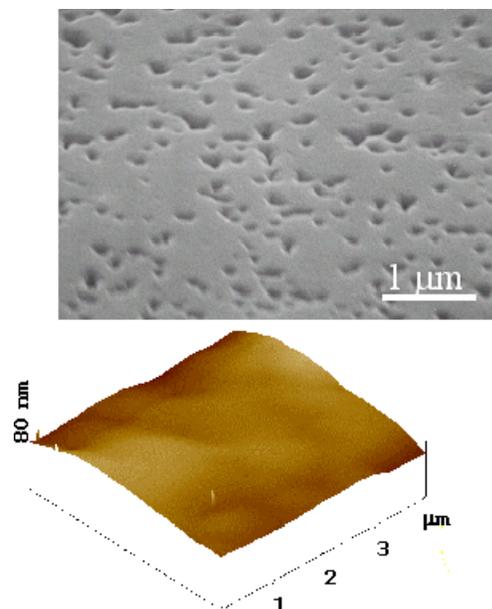


Figure 7b. SEM and AFM images of a sample grown at 950 °C for 1 min and at increasing temperature up to 1020 °C for 2 min based on proposed two-step growth.

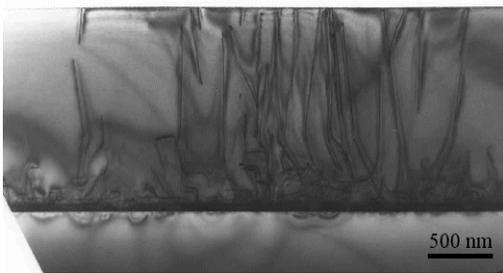


Figure 8a. Cross-sectional TEM images of typical one-step sample under 0002 two-beam.

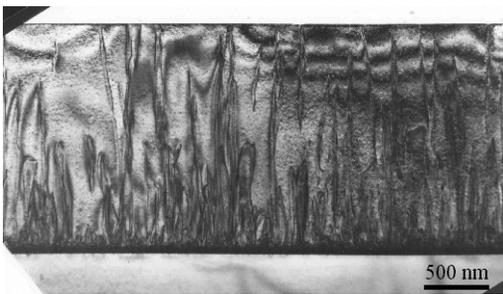


Figure 8b. Cross-sectional TEM images of special two-step sample under 0002 two-beam.

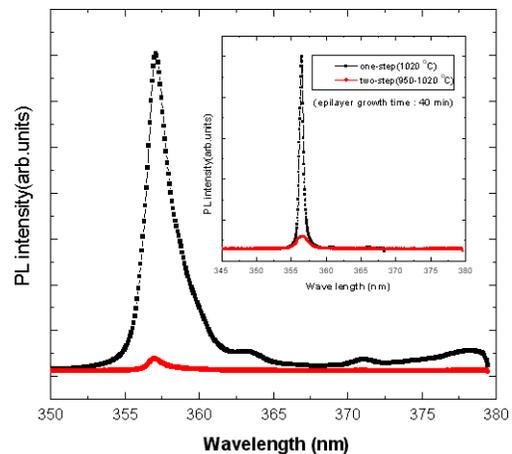


Figure 9. PL spectra for samples grown based on one-step growth and two-step growth for 3 min. Inset shows PL spectra for both samples grown for 40 min.

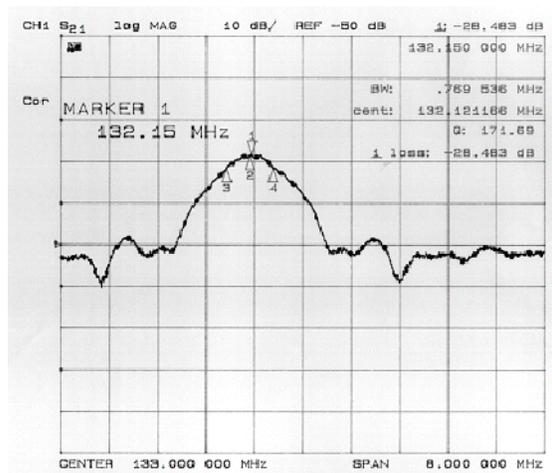


Figure 10a. Frequency response characteristics of fabricated GaN SAW filters with wavelength of 40 μm for undoped GaN film grown at 1020 °C.

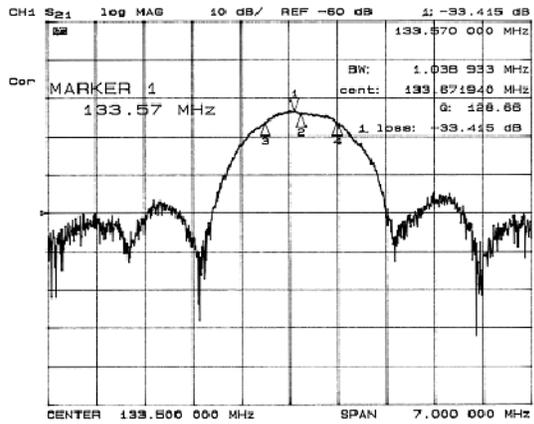


Figure 10b. Frequency response characteristics of fabricated GaN SAW filters with wavelength of 40  $\mu\text{m}$  for semi-insulating GaN film grown based on two-step growth.