

Effects of Rapid Thermal Annealing on the Structural Properties of GaN Thin Films

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Abstract—Effects of rapid thermal annealing (RTA) on the structural properties were investigated in undoped GaN film grown by rf-plasma-assisted molecular beam epitaxy (MBE). Detailed characterizations of the photoluminescence (PL), high-resolution X-ray diffraction and low-frequency noise were conducted on both the as-grown and annealed films. PL and X-ray diffraction measurements showed that the crystallinity of the films improved with RTA at 800 °C with significant reduction in the yellow emission. Annealing at 900 °C and 1000 °C resulted in an increase in the FWHM of the X-ray diffraction, indicative of thermal decomposition of the materials. The results are in excellent agreement with our study of low-frequency noise, which demonstrates similar trend in the magnitudes of the Hooge parameters as a function of the annealing temperature. The temperature dependence of the voltage noise power spectra $S_v(f)$ was examined from 400 K to 80 K in the frequency range between 30 Hz and 100 kHz. At the low-frequency range the fluctuation is dominated by $1/f^\gamma$ noise, and for $f > 1$ kHz the noise is dominated by G-R noise processes. Our experimental results show that 800 °C is the optimal temperature for RTA, which results in substantial improvements in both the optical, structural and noise properties for the material, whereas annealing at 1000 °C is found to result in significant material degradation.

Index Terms—Gallium nitride, rapid thermal annealing (RTA), low-frequency noise.

I. INTRODUCTION

THE III-V nitrides have been under intense research because of their important applications in optoelectronic and electronic devices such as blue light-emitting diodes [1], blue laser diodes [2], UV photodetectors, and high-temperature and high-power devices [3]–[5]. The lack of GaN substrates remains to be the biggest technological hurdle for the growth of high-quality GaN epitaxial layers. Presently, sapphire is the most commonly used substrate for GaN epitaxial growth due to its relative low price, availability of large-area wafers with good crystallinity, and stability at high temperatures. The lattice mismatch between GaN and sapphire is about 14%. Such huge mismatch in the lattice constants results in poor crystal quality if GaN films were to be grown directly on the sapphire. Typically,

GaN thin films exhibit wide X-ray rocking curves, rough surface morphologies, high intrinsic electron concentrations, and significant yellow luminescence [6]–[8]. A low-temperature buffer layer is typically used for improving the crystal quality of the epitaxial film. The role of the buffer layer has been studied extensively. It is found that the AlN or GaN buffer layers serve to enhance two-dimensional growth and the density of nucleation for the epitaxial films. This is because the interfacial energy for the GaN/buffer system is found to be significantly lower than the GaN/sapphire system.

It has been known for a long time that the structural quality of the epitaxial films plays a crucial role in determining the optoelectronic properties of the material. One of the most undesirable characteristics is the yellow luminescence peaking at around 2.2 to 2.3 eV. This is a universal feature in both doped and undoped GaN epilayers grown by various techniques such as molecular beam epitaxy (MBE), and metalorganic chemical vapor phase epitaxy (MOVPE). A number of possible origins have been postulated to account for the observed yellow luminescence, including Ga vacancy (V_{Ga}), or complexes involving V_{Ga} and a carbon impurity occupying the nitrogen site, or extended defects like dislocations and grain boundaries [9]–[11]. The presence of mixed cubic and wurtzite phases, due to stacking faults, is also one of the major factors affecting the crystal quality, which has been shown by Lin *et al.* [12] to be partly responsible for the yellow luminescence. Previous studies also showed that yellow luminescence intensity is quite inhomogeneous over the entire film. Cathodoluminescence performed on undoped GaN hexagonal crystallites indicates that while the band edge emission originates in the bulk of the GaN hexagonal crystallites, the yellow luminescence, on the other hand, is associated with the crystalline boundary and appear to correlate with the presence of defects in the material [13], [14]. Since the yellow luminescence significantly affects the operations of the device in the visible regime, it is important to determine the growth conditions to minimize it.

Low-frequency noise is strongly affected by crystalline defects [15], [16]. It is an important figure-of-merit for semiconductor devices as it represents the smallest signal a device can process. Studies of flicker noise and G-R noise in semiconductor devices show that both processes originate from thermal activation of carriers to localized states within the bandgap [17]. Defect density that varies smoothly with energy will lead to $1/f^\gamma$ noise, whereas G-R noise arises from defects that peak at one single energy level. This model for low-frequency excess noise in GaN thin films is supported by recent results on the characterization of $1/f^\gamma$ noise of GaN thin films grown with the application of indium surfactant [18]. The results show that application

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of indium surfactant lead to substantial reduction in defect density as well as the level of $1/f^\gamma$ noise, strongly indicating defect origin of flicker noise. Thus, improvement in the crystal quality is the crucial step toward the improvement of the optical and noise properties of GaN-based devices.

In this paper, we report a systematic study on the effects of rapid thermal annealing (RTA) on the optical and structural properties of undoped GaN epilayers grown by rf-plasma-assisted MBE. The films were examined by photoluminescence (PL) spectroscopy, high-resolution X-ray diffraction (HRXRD), and low-frequency noise. The work provides a basis for further elucidation of the effects of RTA on the optical and structural properties of GaN films and the feasibility of the technique for improving the quality of GaN thin films.

II. EXPERIMENT AND RESULTS

Undoped GaN epilayers were grown on single-side-polished (0001) sapphire substrates by an SVTA BLT-N35 MBE system. The backsides of the substrates were coated with molybdenum to improve the effectiveness of radiation heating. The substrates were cleaned by organic solvents, followed by etching in hot $\text{H}_2\text{SO}_4\text{:H}_3\text{PO}_4 = 3 : 1$ solution and rinsing in deionized water. The sapphire substrates were then placed in the preparation chamber for outgassing at 850 °C for 30 min. The substrates were then transferred to the growth chamber and were maintained at 550 °C while being exposed to plasma activated nitrogen for the nitridation. It was shown that this process changes the surface layer of the sapphire substrate to AlN, leading to significant improvements in the quality of the epitaxial layers. Subsequently, 200 Å of low-temperature GaN buffer layer was deposited at around 550 °C [19]. The substrate temperature was then raised to 770 °C for the growth of the epilayer. High purity metallic Ga was evaporated by an EPI SUMO cell and N_2 gas was excited by an EPI UNI-Bulb nitrogen plasma source. Typical growth rate was 0.9 $\mu\text{m}/\text{h}$ and the final thickness was about 860 nm. The as-grown GaN film was divided into four pieces. Three of them were annealed in N_2 ambient for 30 s at 800 °C, 900 °C, and 1000 °C, respectively, with dummy GaN films placed on top of the samples during the RTA process.

The crystallinity of the films was examined by high-resolution X-ray diffraction (HRXRD) experiment, which was carried out with $\text{CuK}\alpha_1$ line radiation ($\lambda = 1.54056 \text{ \AA}$), monochromated by a four-crystal Bartels monochromator. Fig. 1 presents the experimental results for the FWHM of the GaN (0002) X-ray rocking curve. Here we observe that the FWHM decreases drastically as a result of RTA at 800 °C. It is interesting to note that a slight increase in the FWHM is observed for RTA conducted at temperatures higher than 800 °C. This shows that RTA at 800 °C results in substantial improvement in the crystallinity of the sample. However, degradation in the material may have taken place for samples annealed at higher temperatures.

The HRXRD results are corroborated by the experimental data for room-temperature PL, which was measured using a 15 mW HeCd laser as the excitation source. The luminescence was dispersed in a 1/8 m spectrometer and detected by a photomultiplier tube. The results for the as-grown film as well as

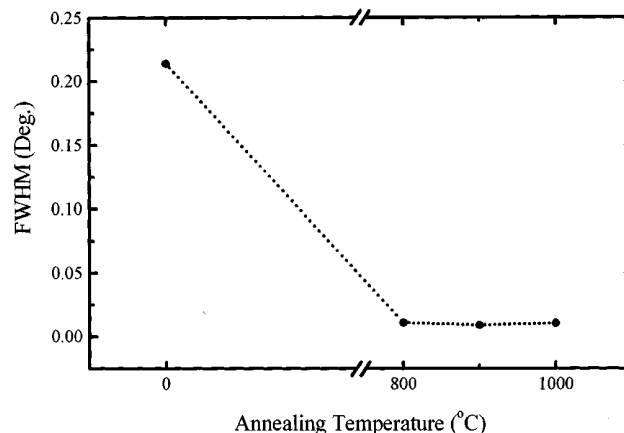


Fig. 1. Full-width-half-maximum of high-resolution X-ray diffraction of samples as a function of annealing temperature.

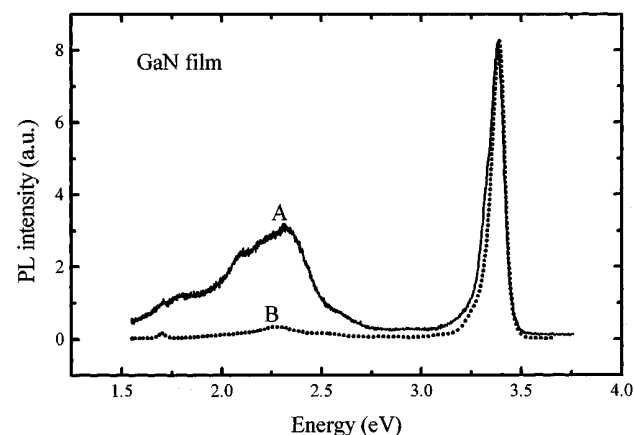


Fig. 2. PL of the as-grown film (Curve A) and a sample annealed at 800 °C (Curve B).

the sample annealed at 800 °C are shown in curves A and B of Fig. 2, respectively. The as-grown sample exhibits strong band edge emission at 3.40 eV with a 93.3 meV FWHM and substantial yellow luminescence at around 2.3 eV. Upon annealing at 800 °C, the FWHM for the edge emission at 3.4 eV is found to reduce to 66.6 meV, indicative of improved optical properties for the sample. Moreover, the yellow emission for the annealed film is found to decrease drastically. This clearly demonstrates significant improvements in the optical properties for GaN films as a result of RTA at 800 °C.

To examine the low-frequency noise of GaN thin films, cross-bridge resistive structures were fabricated by reactive ion etching. The ion energy is kept at a low level by using low rf power density of approximately 200 W to minimize RIE-related damages [20]. The etch rate is found to be approximately 200 Å/min. Furthermore, in our case, the damages resulting from the RIE process are limited to the sidewalls. The contact area was completely protected by photoresist during the etching process and should suffer no damage at all. Thus, the effects of damages due to RIE in our devices should be minimal. The

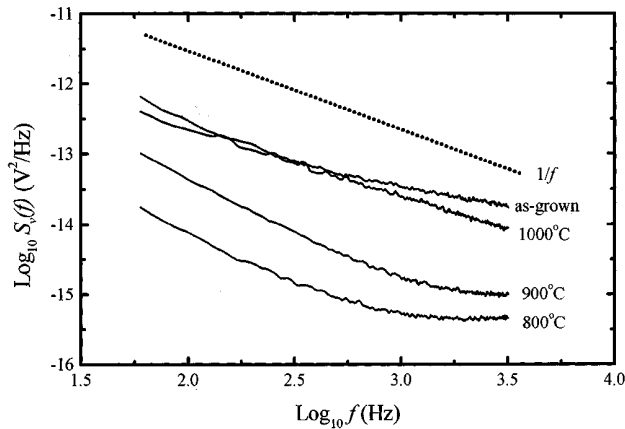


Fig. 3. Voltage noise power spectra $S_v(f)$ for samples annealed at different temperatures.

cross-bridge resistive structure facilitates the measurements of I – V characteristics, Hall mobility, and four-probe measurement of low-frequency noise over the entire temperature range of our experiment. Ohmic contacts were achieved by sputter deposition of Ti/Al bilayers. The contact resistance was determined using the standard multiple-contact, two-terminal contact resistance measurement technique and was found to be less than $10^{-4} \Omega\text{cm}^2$. The I – V characteristics of the samples were examined over the entire temperature range from 80 K to 400 K. The experimental results indicate well behaved I – V curves for the samples over the entire range of temperatures at which the noise was investigated. This shows that the contacts are of good quality and have relatively low potential barriers.

Low-frequency noise was studied over a wide range of temperatures from 80 K to 400 K, over the frequency range of 30 Hz–100 kHz. The devices were current biased using a passive current source, which was constructed by placing a metal film resistor, with a resistance at least 30 times that of the device, in series with the battery source. The noise voltage was amplified by a PAR113 preamplifier, and the voltage noise power spectral density was measured by an HP3561A dynamic signal analyzer. For low-temperature measurements, the samples were placed inside a continuous-flow cryostat. Temperature control was accomplished using a Lakeshore 91C temperature controller with an Si diode temperature sensor. Detailed discussions of the experimental setup are given in previous reports [21], [22]. Fig. 3 shows the voltage noise power spectral density, $S_v(f)$, in which flicker noise is the dominant fluctuation process for $f < 1$ kHz. Above 1 kHz, the noise is dominated by G-R processes. Similar results have been obtained by Misra *et al.* [23], who observed $1/f$ noise associated with contacts and surfaces and G-R noise from the trapping and detrapping of carriers by deep levels. Substantial reduction in the noise power spectral density is observed for the samples annealed at 800 °C. However, annealing at 1000 °C resulted in substantial increase in the magnitude of the voltage noise power spectrum, indicative of significant increase in the defect density. For $f > 1$ kHz, the voltage noise power spectra are characterized by Lorentzians indicating dominance by G-R noise. Typical voltage noise power spectra

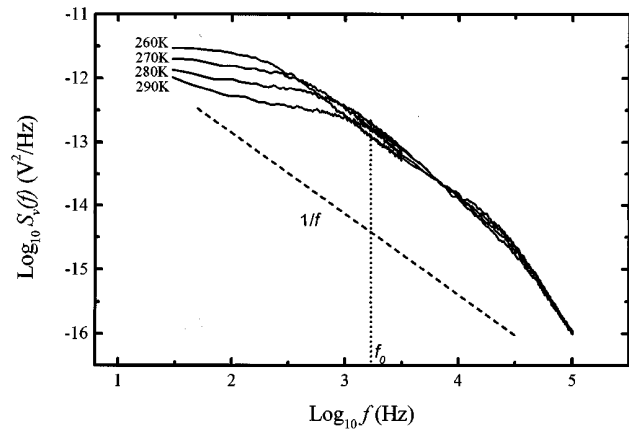


Fig. 4. Voltage noise power spectra for the as-grown film at high frequency measured at different device temperatures, demonstrating the shift of the corner frequencies of the Lorentzian bumps.

measured from the as-grown film are shown in Fig. 4. For the sample annealed at 1000 °C, only flicker noise was observed.

III. DISCUSSIONS

The experimental results shown above indicate that there are significant improvements in the trap density and the crystallinity as the films are annealed. The minimum value for the FWHM of the X-ray diffraction was obtained for films annealed at 800 °C. However, when the films are annealed at 900 °C or above there is an increase in the FWHM of the X-ray diffraction pattern, indicative of material degradation. The PL also exhibited significant reduction in the yellow emission when annealed at 800 °C. Improvement in the optical properties is also observed from the decrease in the FWHM of the PL, which decreases from 93.3 meV for the as-grown film to 66.6 meV for the film annealed at 800 °C. The corresponding carrier concentrations are $9.3 \times 10^{17} \text{ cm}^{-3}$ and $1.7 \times 10^{18} \text{ cm}^{-3}$, respectively. This contradicts with the recent results by Iliopoulos *et al.* [24]. Their experimental results exhibit broadening in the PL spectra with the increase in the carrier concentration. A possible reason for this discrepancy is that for Si-doped GaN films studied by Iliopoulos *et al.* high doping concentration leads to additional scattering by ionized donors and Auger processes resulting in the increase in the FWHM of the PL. In our case, undoped samples were being studied. The changes in the PL FWHM and the carrier concentration resulted from the thermal annealing process, which we have shown, from our HXRD results, to lead to improvements in the crystallinity of the material. It is, therefore, reasonable to expect a corresponding decrease in the FWHM of the PL. It is widely believed that free carriers in undoped GaN films are originated from nitrogen vacancies in the material. While our experimental results clearly demonstrate that RTA leads to the reduction in deep levels, it is not clear how the processes that give rise to the free electrons are being affected. Further study is needed to fully investigate the underlying mechanisms that affect the free carrier concentration by RTA.

The magnitudes of the $1/f$ noise power spectra follow the same trend as the FWHM of the X-ray diffraction. This shows that the observed $1/f$ noise has a strong correlation with the

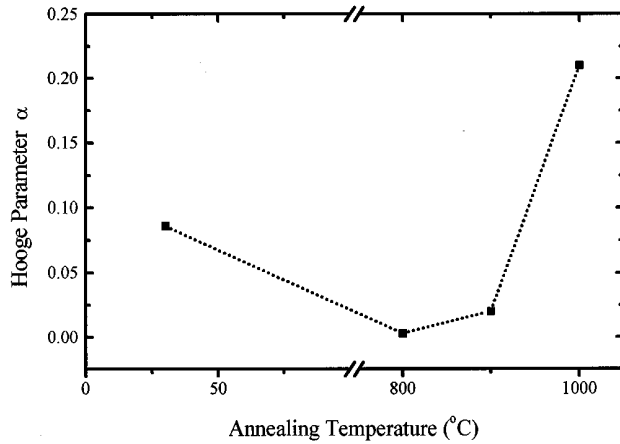


Fig. 5. Variation of the Hooge parameter α as a function of annealing temperature.

crystallinity of the material, clearly indicating that the noise arises from defects within the films. Flicker noise can be characterized by the dimensionless Hooge parameter α [25]

$$\alpha = S_v(f) f N / V^2 \quad (1)$$

where

N total number of carriers in the sample;
 V dc voltage applied to the device;
 $S_v(f)/V^2$ normalized voltage noise power spectral density.

The variation of α as a function of annealing temperature is shown in Fig. 5. The value of Hooge parameter α for the as-grown film at room temperature is 8.6×10^{-2} , which is much larger than other semiconductors such as Si ($\alpha \approx 10^{-8}$) [26] and 4H-SiC ($\alpha \approx 10^{-6}$) [27]. Such a high noise level was attributed to the large density of localized states in the bandgap. The large increase in α for the film annealed at 1000 °C is indicative of significant material degradation due to the high annealing temperature. This is consistent with our previous Rutherford Backscattering Spectroscopy characterization of p-type GaN thin films grown by MOCVD, which showed that annealing at 950 °C and above resulted in substantial changes in the microstructure of the films.

It is known that G-R noise is caused by the capture and emission of carriers by traps located at discrete energy levels [28]. From our experimental data, it is found that the Lorentzians are strongly temperature dependent. The fluctuation time constant is determined experimentally by $\tau = 1/2\pi f_0$, where f_0 is the cutoff frequency of the Lorentzian. Two distinct G-R processes are observed in the as-grown GaN film. However, only one Lorentzian is observed for GaN films annealed at 800 °C and 900 °C. No G-R noise is found for the sample annealed at 1000 °C in the temperature and frequency range of our experiment. The thermal activation energies for the fluctuation time constants, determined by the Arrhenius plots, are 65 meV and 360 meV for the as-grown GaN film, 100 meV for the sample annealed at 800 °C, and 120 meV for the sample

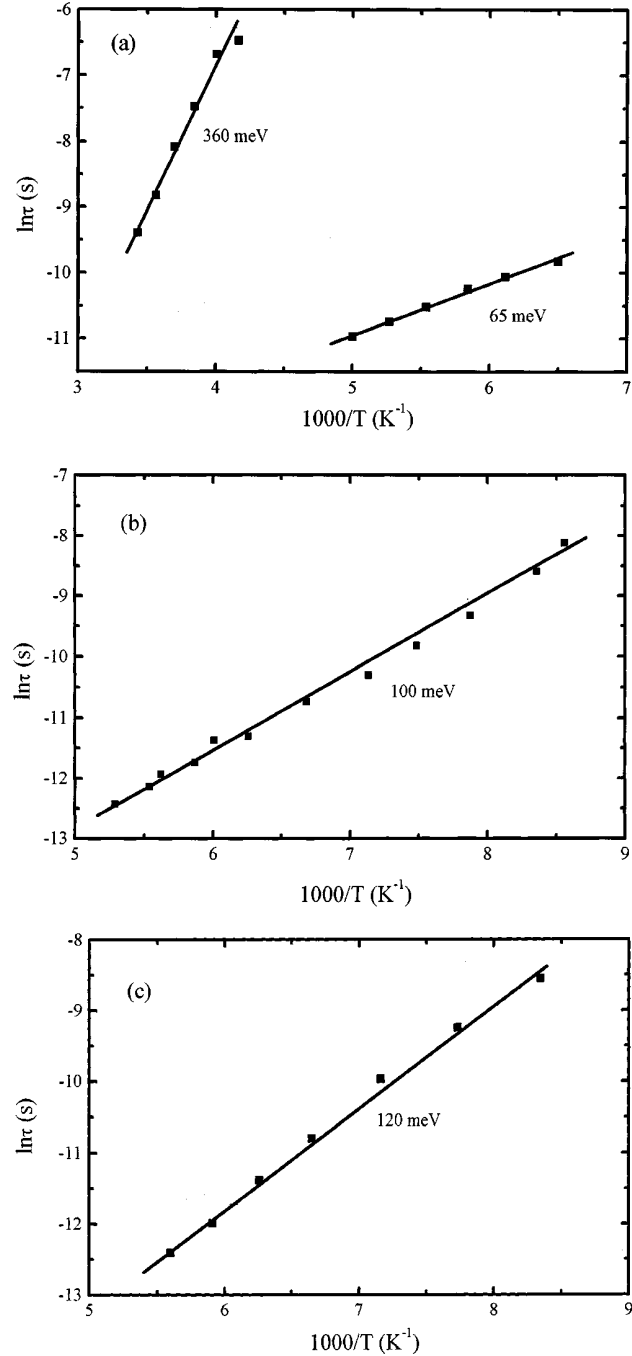


Fig. 6. Arrhenius plots of the fluctuation time constant, τ , (a) for the as-grown film and (b) for annealing temperature equals 800 °C and (c) 900 °C.

annealed at 900 °C, as shown in Fig. 6. Such pronounced changes in the $1/f$ noise and G-R noise properties stipulate corresponding significant changes in the crystallinity of the samples arising from the annealing processes. In particular, it is interesting to note that annealing at 1000 °C resulted in the domination by flicker noise in the sample, which is accompanied with a dramatic increase in α .

The experimental results can be explained by the defect model for low-frequency excess noise. In this model, both G-R

noise and $1/f$ noise originate from the localized states within the bandgap. Localized states at a specific energy level, E , will give rise to a noise power spectral density in the form of a Lorentzian, characterized by the fluctuation time constant τ given by

$$\tau = \tau_0 \exp\left(\frac{E}{k_B T}\right), \quad (2)$$

where E is the activation energy of the fluctuation time constant and τ_0 is taken as the inverse phonon frequency, typically of the order 10^{-14} s. If the defect density varies smoothly with energy then the total noise power spectrum can be evaluated as the superposition of all the individual Lorentzians resulting in a spectral form proportional to $1/f^\gamma$. If the trap density at some energy levels are substantially larger than the rest, then the associated Lorentzians will be significantly more prominent in the overall noise power spectrum. This gives rise to an $S_v(f)$ characterized by the superposition of $1/f$ noise and one or more bulging Lorentzian bumps. This is exactly what we observe in our experimental data. Fig. 4 indicates the presence of two Lorentzians. One Lorentzian has corner frequencies varying between 300 Hz and 2 kHz when the device temperature changed from 260 K to 290 K. The corner frequencies for the second Lorentzian are located between 15 kHz and 30 kHz. It is interesting to note that at the frequency range between the two corner frequencies, the voltage noise power spectral density roughly exhibit $1/f^\gamma$ relationship, with γ close to 1. Also, at frequencies beyond the corner frequencies for the second Lorentzian γ is much larger than 1. This clearly shows that $1/f$ noise can be obtained by simple linear superposition of a small number of Lorentzians. The corner frequencies of the Lorentzians are highly sensitive to temperature change, which shift toward the low-frequency end as the device temperature is reduced. This strongly indicates that the noise arises from a thermally activated process. The experimental results clearly show that certain traps have been effectively annealed out using an annealing temperature of 800 °C due to the disappearance of a Lorentzian subsequent to annealing the films at 800 °C. Furthermore, the disappearance of the Lorentzian bumps in $S_v(f)$ after annealing at 1000 °C can be explained by the substantial increase in the trap density in all different energy levels as a result of material degradation. Hence, the energy distribution of the localized states becomes smoothened out, leading to the absence of a dominating Lorentzian in $S_v(f)$.

IV. CONCLUSION

The effects of RTA on the structural, optical and electronic properties of GaN epilayers, grown by rf-plasma-assisted MBE, have been investigated systematically. Substantial improvements in the PL, especially in the yellow band emission, FWHM of the X-ray diffraction, and trap density was observed by performing RTA at 800 °C. Our results show that RTA is effective in improving the structural and optoelectronic properties of MBE-grown GaN films. However, when the films were annealed at 900 °C or above there is an increase in the FWHM of the X-ray diffraction and the magnitude of low-frequency

noise power spectral density, indicative of material degradation resulting from the excessive annealing temperatures.

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