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Remote plasma-enhanced atomic layer deposition of gallium oxide thin films with NH_3 plasma pretreatment

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Abstract: High quality gallium oxide (Ga_2O_3) thin films are deposited by remote plasma-enhanced atomic layer deposition (RPEALD) with trimethylgallium (TMG) and oxygen plasma as precursors. By introducing *in-situ* NH_3 plasma pretreatment on the substrates, the deposition rate of Ga_2O_3 films on Si and GaN are remarkably enhanced, reached to 0.53 and 0.46 Å/cycle at 250 °C, respectively. The increasing of deposition rate is attributed to more hydroxyls (–OH) generated on the substrate surfaces after NH_3 pretreatment, which has no effect on the stoichiometry and surface morphology of the oxide films, but only modifies the surface states of substrates by enhancing reactive site density. Ga_2O_3 film deposited on GaN wafer is crystallized at 250 °C, with an epitaxial interface between Ga_2O_3 and GaN clearly observed. This is potentially very important for reducing the interface state density through high quality passivation.

Key words: Ga-oxide; RPEALD; passivation; NH_3 plasma

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1. Introduction

Oxide thin film is a kind of essential materials in semiconductor devices. Silicon dioxide, as a kind of dielectric material, has been playing a vitally important role in Si-based devices and the well-developed integrated circuit (IC) industry due to its excellent properties and the perfect matching with Si for interface passivation[1]. However, it is still a bottleneck for compound semiconductors, particularly for wide band gap semiconductors, to find a suitable dielectric material in their device applications. For example, the two critical issues with GaN-based high electron mobility transistor (HEMT) devices are current collapse and gate leakage, for which the root cause is considered as high interface state density at dielectric/GaN interface, resulted from poor passivation on GaN surface and defects at the interface[2]. For GaN based device, Ga_2O_3 should be the first choice to play a similar role as SiO_2 in Si-based devices, but so far it is unsuccessful in practice. There are many reasons for that, but one of them is the difficulty in formation of high quality Ga_2O_3 thin film with low impurity and defect densities. Actually, native gallium oxides generally exist at the interface between GaN and dielectric layer (such as Al_2O_3 or Si_3N_4), creating dense defects because of poor quality. Therefore, it may be a good solution to replace the native oxides by controllable growth of high quality Ga_2O_3. Ga_2O_3 is a oxide semiconductor or dielectric material with large band gap (4.9 eV)[3], good thermal and chemical stabilities, and high dielectric constant (10.6)[4, 5]. Ga_2O_3 thin film has been applied in a wide variety of applications, including optoelectronic devices[6], gas sensor devices[4], passivation or dielectric layers for III–V based microelectronics[7, 8]. Additionally, the latest technology of semiconductor fabrication requires a controllable deposition technique as well as low temperature process[9]. Therefore, various techniques have been developed to deposit Ga_2O_3 thin film, including pulsed laser deposition[10], electron beam evaporation[11], magnetron sputter deposition[12], molecular beam epitaxy[13], metal organic chemical vapor deposition (MOCVD)[14], vapor phase epitaxy[15], chemical vapor deposition (CVD)[16], atomic layer deposition (ALD)[17–19]. Among them, ALD is the most potential one to grow thin film materials with good thickness uniformity and composition control ability under low temperatures[20]. Actually, low temperature processes are more required in next generation devices, such as transparent and flexible material, in order to limit potential interlayer diffusion[21].

Plasma enhanced atomic layer deposition (PEALD) provides a number of advantages for preparing Ga_2O_3 thin film. It reduces the activation energies for the chemisorption of precursors at low-temperature growing films[22, 23], and additionally makes film compact and dense[24]. In our work, we adopt remote-enhanced plasma atomic layer deposition (RPEALD) technology for Ga_2O_3 thin film deposition, by taking its unique advantage of less interference between substrate and plasma source. It was reported that NH_3 plasma treatment before film deposition could remove impurities on substrate surface, reduce interface state density, and then improve the film quality remarkably[25–28]. Moreover, it also makes substrate surface activated, enhancing chemical adsorp-

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Table 1. Deposition recipe for Ga_2O_3 thin films.

Parameter	Pretreatment condition	Deposition condition
Working pressure (hPa)	10	10
Deposition temperature (°C)	room temperature	100, 200, 250, 300, 400
Gas flow rate (sccm)	150 (NH_3 plasma), 40 (N_2)	150 (TMG), 50 (O_2 plasma), 50 (N_2)
Pulse/purge time (s)	NH_3 plasma (13), N_2 (6)	TMG (0.1), N_2 (6), O_2 plasma (13–20), N_2 (6)
Plasma generator power (W)	2000 (NH_3 plasma)	2800 (O_2 plasma)

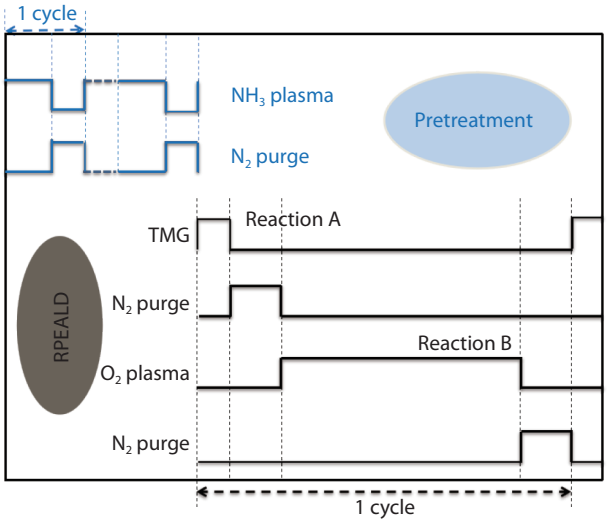

Fig. 1. (Color online) Schematic diagram of Ga_2O_3 thin film deposition process.

tion of precursors[29]. Here we propose a method for deposition of Ga_2O_3 thin films by *in-situ* NH_3 plasma pretreatment of Si and GaN substrates, and the subsequent deposition of Ga oxide films by RPEALD. And focus on the interface properties to explore the possibility of GaN surface passivation by epitaxial Ga_2O_3 thin film.

2. Experiment

Ga_2O_3 thin films were deposited by RPEALD (PICOSUN R-200 Advanced, Finland) using TMG (5N) as gallium source, ultra-pure O_2 (6N) plasma as oxygen reactant with a base pressure of 10 hPa. N_2 (5N) is used as carrier gas of TMG and purge gas of residues. Before the film deposited, 2-inch n-Si (100) wafers were first prepared with ultrasounds of acetone, isopropyl alcohol, and de-ionized (DI) water for 5 min each in sequence, then, cleaned in diluted HF solution (HF : H_2O = 1 : 30) for removing native oxide, followed by DI water rinsing and pure N_2 drying. GaN epi wafers (~1 × ~1 cm^2) were cleaned similarly as Si substrates, except using NH_4OH (25%–28%) instead of HF solution to remove intrinsic oxidation layer. After the *ex-situ* cleaning, Si wafers and GaN epi wafers were placed in the RPEALD chamber. The *in-situ* NH_3 plasma pretreatment was conducted in the RPEALD reactor at room temperature (RF power 2000 W, 13 s of NH_3 plasma and 6 s of N_2 purge, 10–70 cycles of treatments). Then in each cycle, TMG, N_2, O_2, and N_2 were introduced into the reactor in sequence, and the flow rates are 150, 50, 50, 50 sccm, respectively, as illustrated in Fig. 1. Ga_2O_3 films were deposited for 250 cycles on these substrates at a selected temperature from 100 to 400 °C. The detailed recipe is listed in Table 1, where one cycle consists of 0.1 s TMG (precursor temperature 0 °C) / 6 s N_2 purge/13–24 s O_2 plasma (RF power 2800 W)/6 s N_2 purge.

The thickness, crystal quality and surface morphology of Ga_2O_3 thin films were determined by ellipsometry (SE, M2000DI, J. A. Woollam), high resolution transmission electron microscope (HR-TEM, Tecnail G2 F20 S-Twin, FEI) and atomic force microscope (AFM, Dimension ICON, Burker). X-ray photoelectron spectroscopy (XPS, PHI 5000 Versa Probe II, ULVAC-PHI), together with time-of-flight secondary ion mass spectroscopy (TOF-SIMS 5, IONTOF), were employed to characterize the film chemical composition, bonding states and impurities from the surface to interface.

3. Results and discussion

To explore the growth parameters of Ga oxide, effects of TMG dose, O_2 plasma duration, and temperature on the growth rate were studied. The TMG dose is represented by its pulse duration, which is found no effect on the deposition rate when it changes from 0.1 to 0.2 s, indicating that the shortest duration of 0.1 s is enough to reach a saturation. Fig. 2(a) shows the deposition rate of Ga oxide on Si as a function of O_2 plasma flow duration, with fixed TMG dose of 0.1 s and growth temperature of 250 °C. The growth rate increases with O_2 plasma duration and saturates when it exceeds 18 s. In order for the reaction to be sufficient, we chose 20 s as O_2 plasma duration. As Fig. 2(b) shows, the deposition rate and chemical ratio of the films are almost independent to growth temperature in a wide range, similar to other reports[18, 20]. With these optimized parameters, the film thickness shows a linear correlation with the cycle numbers, as shown in Fig. 2(c), demonstrating the self-limiting characteristic of ALD method. In addition, it is clear that the deposition rate of Ga oxide is lower on GaN epi wafer than on Si substrate, meaning initial surface conditions plays a significant role on the growth rate. All of these behaviors are the typical features of ALD technique, although the growth rate of Ga oxides is slightly lower than that of other reports[4, 18], as shown in Fig. 2(d).

It has been reported[28, 30] that plasma treatment may modify the surface bonding states, or increase reactive sites on the substrate. For example, N_2/NH_3 plasma can eliminate surface-adsorbed ligands, forwarding the chemical adsorption of precursor in the next precursor supply step[29, 31]. *In-situ* NH_3 plasma cleaning process was also applied to eliminate carbon contamination from GaN surface, maintain a relatively steady oxygen cover and may passivation the defect state about N-vacancy. To further explore the mechanism of NH_3 plasma pretreatment, we first focus on its effect on the deposition rate of gallium oxide films, as shown in Fig. 3. For the films of 250 cycles deposition, the growth rate increases from 0.44 to 0.53 Å/cycle with 10 cycles of NH_3 pretreatments on the substrate (Si), and saturates afterwards. Similar effect is also observed for the oxide growth on GaN epi wafer, which increases from 0.24 to 0.46 Å/cycle.

All Ga oxide films grown on Si and GaN, with and without

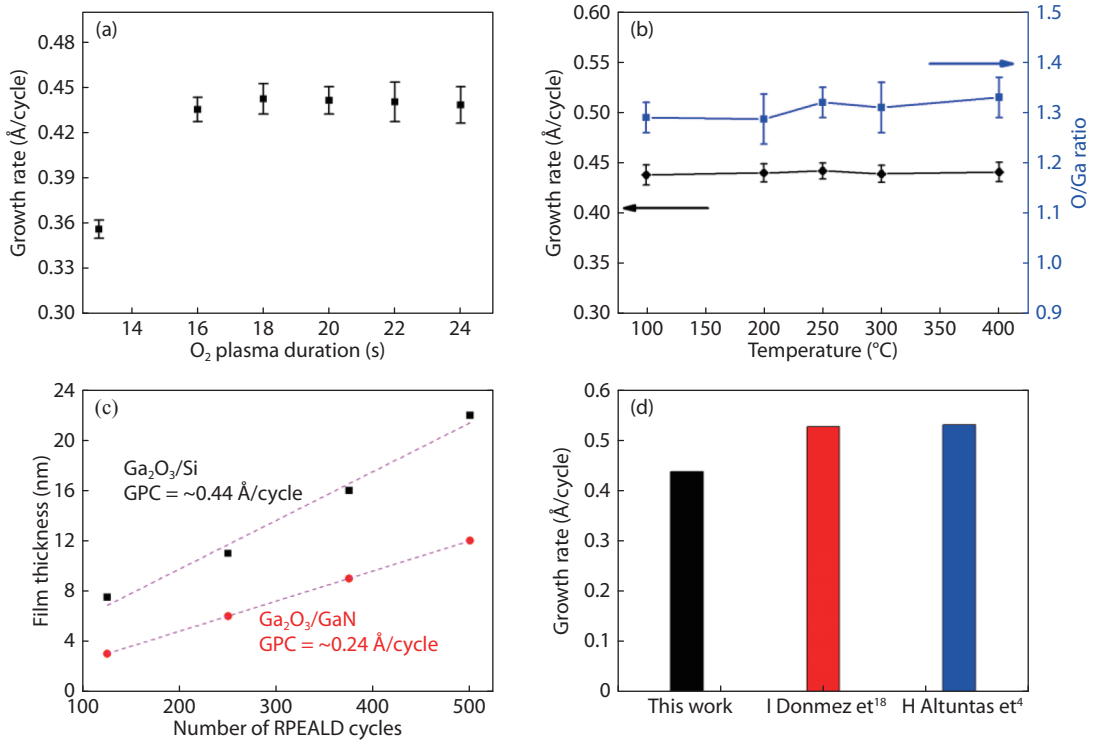

Fig. 2. (Color online) (a) Growth rate of Ga_2O_3 films on Si as a function of O_2 plasma flow duration at 250 °C. (b) Growth rate and O/Ga ratio of Ga_2O_3 films on Si as a function of temperature. (c) The Ga_2O_3 thin films thickness as a function of the number of RPEALD cycles. (d) A comparison of Ga_2O_3 deposition rates on Si between this work and the reported literatures.

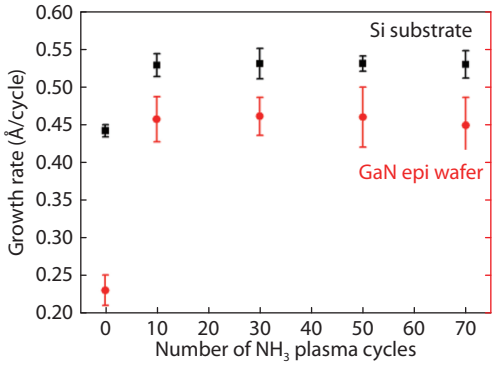

Fig. 3. (Color online) Growth rate of Ga_2O_3 thin films as a function of NH_3 plasma cycles.

NH_3 pretreatment, were characterized by XPS to analyze their composition and chemical states. The O1s and Ga3d core levels were collected from the as-grown surfaces with an emission angle of 45°, and calibrated by C1s peak (assigned at 284.8 eV) to align the binding energies of all peaks. In Fig. 4, the Ga3d (a) and O1s (b) peaks of Ga oxide films deposited by different processes are overlapped and fitted by using CasaXPS software. For Ga3d, the peak with binding energy of ~20.5 eV is attributed to Ga–O bonding[32, 33], while peaks located at 18.5 and 23.7 eV are assigned to Ga-rich phase and O2s core level, respectively. Correspondingly, the O1s peak can be fitted to the O–Ga with a peak of 531.2 eV and the O–H with a peak of 533.3 eV. The binding energies of Ga3d main peak (~20.5 eV) and O1s main peak (~531.2 eV) match well with the reported binding energies of Ga_2O_3[13, 18, 34]. The film's stoichiometry is determined by the intensity ratio of O/Ga, which is ~1.3 for all the samples. In addition, this ratio is nearly the same in a wide temperature window from 250 to 400 °C, as shown in Fig. 2(b). Since there is no notable difference among these Ga3d peaks, as well as O1s peaks, we believe that the stoichiometry of Ga oxide deposited on Si or GaN remain the same at a growth temperature of 250 °C or above, regardless of NH_3 pretreatment.

One of the ALD technique advantages is high conformity and uniformity. The surface morphology of Ga_2O_3 thin films deposited on Si and GaN were characterized by AFM, as shown in Fig. 5. The surface of Ga_2O_3 film on Si is smoother than that of Ga_2O_3 on GaN, which shows clearly step flow as following the features of GaN epi wafer. The surface roughness of the two Ga_2O_3 thin films are 0.12 and 0.20 nm respectively. Comparing with the results reported by others[7, 20, 21], we achieved the smallest values of RMS for Ga-oxide films deposited with similar conditions. With NH_3 pretreatment, the surface morphology of Ga_2O_3 thin films on GaN shows no difference. Therefore, we could infer that NH_3 pretreatment has effect on oxide growth rate but on the chemical stoichiometry and surface morphology, meaning it only modifies the surface states of substrate by enhancing reactive site density.

Therefore, it is likely that the substrate surface condition was modified somehow by the plasma. We conducted TOF-SIMS depth profiling to check the interfaces of oxides and substrates (Si and GaN). As shown in Fig. 6, with 250 cycles deposition, the oxide film thicknesses are increased on pretreated Si substrate and GaN epi wafer respectively. Meanwhile, the counts of H^- and OH^- secondary ions are obviously increased at the interfaces after NH_3 pretreatment. The 3D depth profile images of SIMS data reconstructions, as the inserts in Fig. 6(b), clearly represent the distribution of OH^- on the oxide surfaces and at the oxide/substrate interfaces. To further confirm the difference in the density of OH^- components, XPS was conducted on GaN epi wafer to investigate the GaN surfaces before

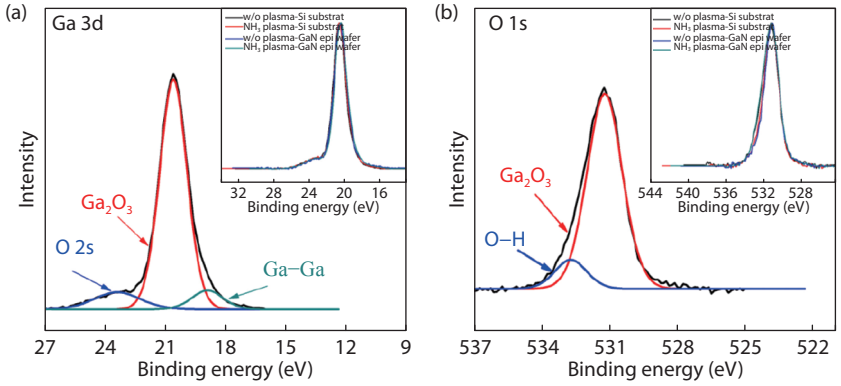

Fig. 4. (Color online) High resolution XPS spectra of (a) Ga3d core level and (b) O1s core level, in Ga_2O_3 films. The insets in (a) and (b) show the overlap of Ga3d and O1s peaks from the Ga oxide films deposited by different processes.

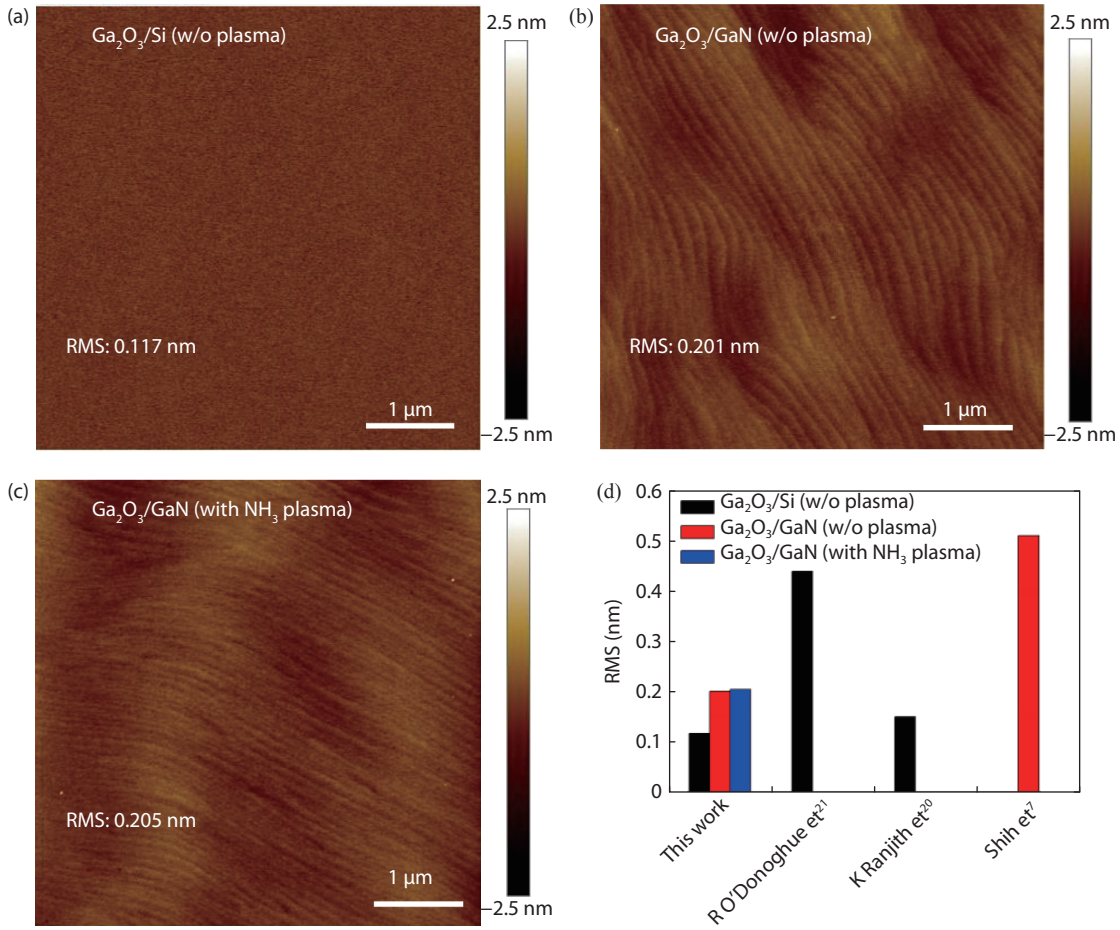

Fig. 5. (Color online) AFM images of (a) Ga_2O_3 film deposited on Si without NH_3 plasma treatment, (b) Ga_2O_3 film deposited on GaN without NH_3 plasma treatment, (c) Ga_2O_3 film deposited on GaN with NH_3 plasma treatment, and (d) a comparison of roughness between this work and previous reported literatures.

and after NH_3 treatment, as shown in Fig. 7. The Ga3d and O1s core level peaks are fitted into related components, according to published references[8, 35]. No shift in the peak position (binding energies) of Ga3d and O1s peak is observed with NH_3 plasma pretreatment, but the portion of hydroxyl (–OH) bond in O1s peak are enhanced roughly by 85% after the treatment. Therefore, we conclude that the initial growth rate of Ga oxide could be enhanced by increasing hydroxide radicals on the substrate surfaces, and NH_3 pretreatment is an effective way to activate the surface bonding states.

In order to further verify the effect of NH_3 plasma on initial growth, we determined the thickness of very thin gallium oxides by using Angle-resolved XPS measurement. The Ga3d core-level spectra for the Ga_2O_3 film of 10 cycles growing obtained at different photoelectron collecting angle (θ) are shown in Fig. 8. Each Ga3d peak can be well fitted the peak of the Ga–N and Ga–O bonds. The thicknesses of the thin films are estimated to be 4.8 ± 0.1 Å for Ga_2O_3 film without NH_3 plasma and 7.9 ± 0.4 Å for Ga_2O_3 film with NH_3 plasma, based on Eq. (1) given as follows[36, 37],

$$\ln(1 + R/R_0) = d/\lambda_0 \cdot \sin\theta, \quad (1)$$

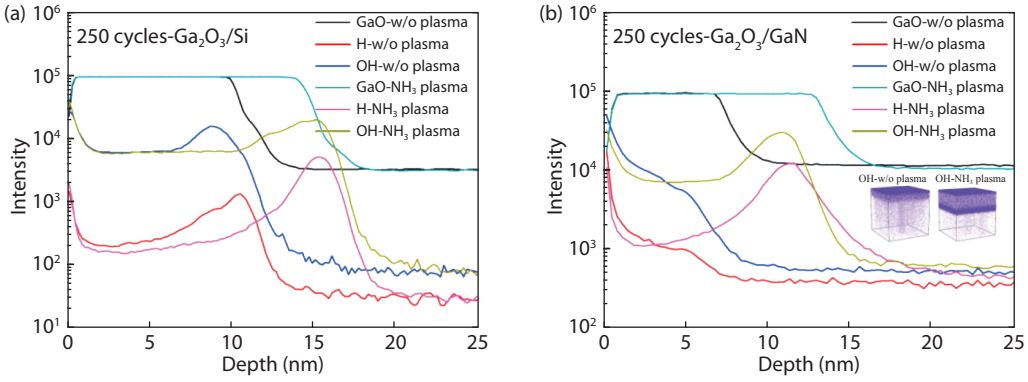

Fig. 6. (Color online) SIMS depth profiles of (a) Ga_2O_3/Si and (b) Ga_2O_3/GaN. The insets in (b) show 3D images of SIMS data reconstructions of Ga_2O_3/GaN.

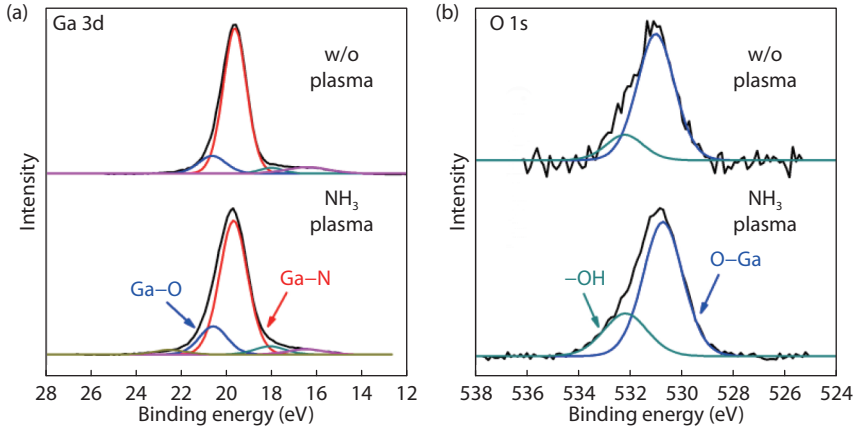

Fig. 7. (Color online) High resolution XPS spectra of (a) Ga3d core level and (b) O1s core level on GaN surface.

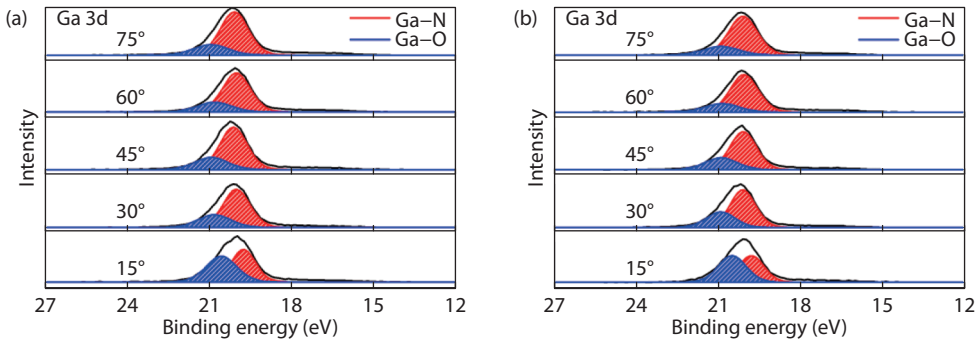

Fig. 8. (Color online) Ga3d core level spectra (a) w/o NH_3 plasma and (b) with NH_3 plasma, in Ga_2O_3 films.

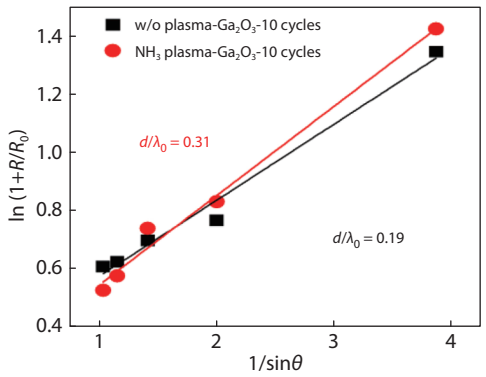

Fig. 9. (Color online) the linear fitting of ln $(1+R/R_0)$ against $1/\sin\theta$.

where R is the areal ratio of Ga–O component to Ga–N component under the Ga3d spectrum; R_0 is related to the bulk material properties of Ga_2O_3 and GaN, it is calculated according to material densities, formula weights and inelastic mean free path (IMFP); d is thickness of the Ga_2O_3 film; λ_0 is electron IMFP of Ga_2O_3 film. A plot of ln $(1 + R/R_0)$ against $1/\sin\theta$ is linear with a slope of d/λ_0, from which the thickness d can be decided, as shown in Fig. 9. The deposition rate of Ga_2O_3 film of the first 10 cycles was increased after NH_3 plasma treatment. It is noticed that the trend of growth rate change is consistent with the above result, which confirmed the initial growth are enhanced by increasing hydroxide radicals on the substrate surfaces, and NH_3 pretreatment is an effective way to activate the surface bonding states.

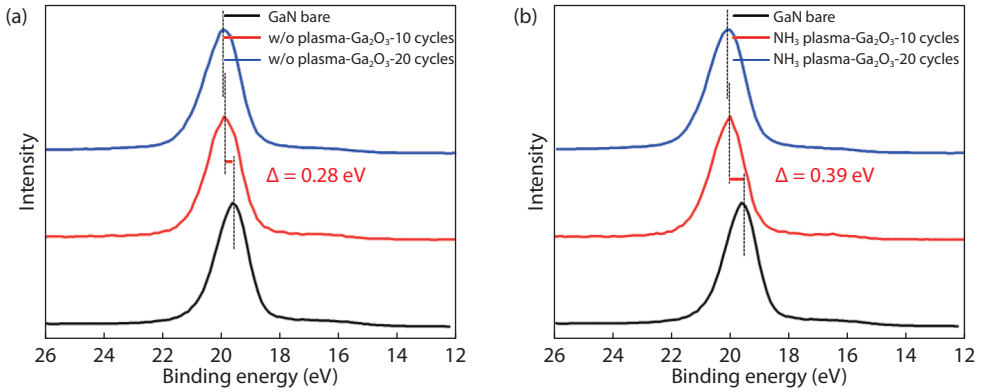

Fig. 10. (Color online) High resolution XPS spectra of Ga3d core level (a) w/o NH_3 plasma and (b) with NH_3 plasma, in Ga_2O_3 film.

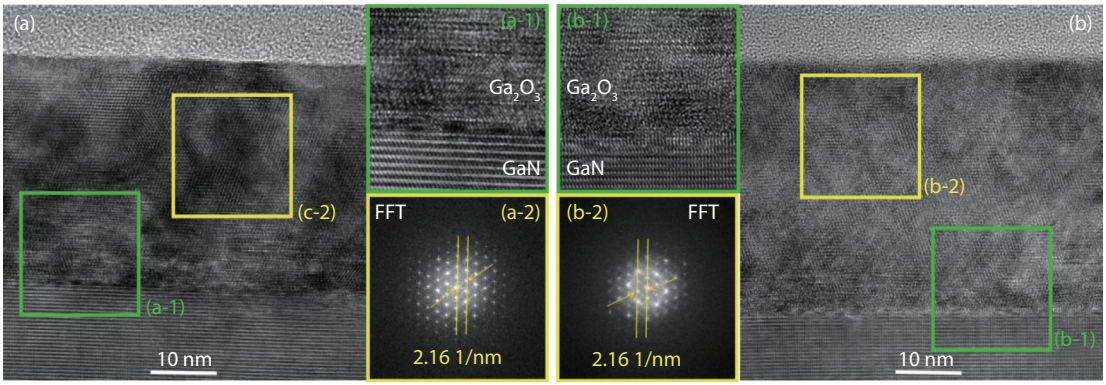

Fig. 11. (Color online) HRTEM analysis of Ga_2O_3 films on GaN deposited by RPEALD. (a) Cross-sectional TEM image of a Ga_2O_3/GaN structure without NH_3 plasma treatment. (a-1) Higher magnification views of the Ga_2O_3/GaN interface without NH_3 plasma treatment. (a-2) FFT of Ga_2O_3 films deposited on GaN without NH_3 plasma treatment. (b) Cross-sectional TEM image of a Ga_2O_3/GaN structure with NH_3 plasma treatment. (b-1) Higher magnification views of the Ga_2O_3/GaN interface with NH_3 plasma treatment. (b-2) FFT of Ga_2O_3 film deposited on GaN with NH_3 plasma treatment.

To further confirm the mechanism of initial growth, we check the chemical states of Ga_2O_3 films deposited with different cycles. As shown in the Fig. 10, the Ga3d peaks shift to higher binding energy with more Ga_2O_3 deposited. For the same deposition cycles, the amount of the peak shift in Fig. 10(a) is about 0.11 eV smaller than that in Fig. 10(b), meaning thicker Ga_2O_3 films are formed on the surface with NH_3 plasma pretreatment than that without the pretreatment. All the results prove that the NH_3 plasma pretreatment enhanced the initial growth rate of Ga_2O_3 thin layer, because more active sites are generated on the substrate surfaces.

High resolution TEM is a powerful technique for atomic structure analysis of thin film and interface. The as-grown films deposited on GaN exhibit ordered arrangement of atoms in a certain level, as shown clearly in Figs. 11(a) and 11(b). The FFT (fast Fourier transform) patterns in Figs. 11(a-2) and 11(b-2) demonstrate the crystallization of low temperature (~250 °C) growth of Ga_2O_3 film with an interplanar distance of 4.63 Å, corresponding to (−201) face of β-Ga_2O_3[10]. In addition, an epitaxial interface between Ga_2O_3 and GaN in Figs. 11(a-1) and 11(b-1) are also observed, which is potentially very important for reducing the interface state density through high quality passivation. However, the crystalline quality of epitaxial Ga_2O_3 film on GaN with NH_3 plasma pretreatment is slightly poorer than that without NH_3 plasma pretreatment, probably due to the damages or defects on GaN surfaces caused by NH_3 plasma. Therefore, to further improve the epitaxial quality, it is necessary to reduce the bombarding effect of the plasma somehow. Anyway, a low temperature epitaxial Ga_2O_3 thin film on GaN is potentially very important for high quality passivation of dielectric/GaN interfaces by reducing the interface state density.

4. Conclusions

In summary, high quality Ga_2O_3 thin films were deposited by remote plasma-enhanced atomic layer deposition within a wide temperature window. With *in-situ* NH_3 plasma pretreatment, the deposition rate of Ga_2O_3 is enhanced remarkably due to more hydroxyls generated on the substrate surface. This pretreatment has no effect on the stoichiometry and surface morphology of Ga_2O_3 films, indicating NH_3 plasma only modify the surface states of substrate by enhancing reactive site density. Ga_2O_3 films grown on GaN wafer is crystallized even at 250 °C, with an epitaxial interface between Ga_2O_3 and GaN. Those results open a new way to reduce the interface states in GaN based devices by passivating GaN surfaces with epitaxial Ga_2O_3 thin films.

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