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Effect of Dry Thermal Oxidation on Bulk GaN Substrates Grown by HVPE during CMP

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Chemical mechanical polishing (CMP) of bulk GaN substrates via dry thermal oxidation is investigated in this paper. In this work, we study the effects of oxidation with respect to different thermal treatments, change in morphology and thickness on bulk GaN substrates. The results of the study show that a defect-free surface with roughness average (Ra) and material removal rate (MRR) of 0.377 nm and 51 μm/h respectively is achievable by CMP after thermal treatment at 800°C. However, for thermal treatments above 900°C, several pits and defects are observed with significant deformation of the surface likely due to the domination of diffusion-controlled reaction over interfacial reaction-controlled. The molar fractions of the chemical components remained on the polished GaN surfaces are characterized via X-ray photoelectron spectroscopy. It is found that the conversion rate from GaN to Ga_2O_3 is dependent on the real contact area between CMP pad and GaN substrate surface during the CMP.
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Gallium nitride (GaN) is a promising semiconductor, which has a wide direct bandgap (3.4 eV), high breakdown voltage, high electron mobility, and high thermal stability.[1–3] Due to its excellent properties, it can overcome the physical threshold of various materials by improving the efficiency of optical materials over that of Si-based materials, and it can be applied to a broad range of UV to IR optical devices to form AlN, AlGaN, and ternary solid solution.[4] In addition, GaN substrates can be applied to power electronic devices, which are suitable for miniaturization, high integration of devices, high power conversion efficiency, fast switching speed, and high power density.[5] In the aspect of substrate technology, mechanical polishing (MP) processes including slicing, lapping, and polishing are required to obtain high-quality single crystal substrates during the manufacturing process. However, during the MP of GaN substrates, damaged layers bearing several defects, such as pull-outs, scratches, and pits, can be generated on the surface, which can severely deform the substrate surface and extremely deteriorate the quality of epitaxial growth.[6] Chemical mechanical polishing (CMP) is the most effective technique for obtaining an undamaged surface. When CMP is conducted for the metal alloy compounds such as GaN and AlN, the chemical oxidizers and water molecules in the CMP slurry generate metal oxide compounds on the substrate surface, and at a certain pressure, the produced metal oxide compounds are removed by the abrasive particles in the CMP slurry.[7,8] However, since the GaN single crystal has high chemical inertness and hardness, it has a low material removal rate (MRR) during the CMP process.[3,9] Thus, several strong oxidizers that can induce a relatively high MRR and further oxidation on the substrate surface have been investigated.[10] However, even the CMP slurries, which have strong oxidizers, induce a low MRR; consequently, many types of slurry have been explored to obtain a defect-free surface. These investigations are considerably expensive and time-consuming. To overcome these problems, the formation of a thick Ga_2O_3 layer in GaN CMP has been recently studied and presented; the process using hydrogen peroxide with UV-treatment, by Jie et al.,[11] is one of the representative examples, and its success is evidenced in the obtained damage-free surface with a high MRR above 100 nm/h.[12] Another method for obtaining a relatively thick Ga_2O_3 layer and a relatively high MRR is a pre-annealing process, which was presented by Hideo et al.,[13] and this study indicated that the CMP process time for obtaining a damage-free surface was remarkably reduced from 150 to 44 h as the annealing temperature increased. However, inhomogeneous oxidation occurred via a diffusion-controlled reaction due to the non-uniform depth of the damaged layer and the inhomogeneity of the inherent defect in the GaN single crystal.[13] To inhibit the diffusion-controlled reaction and obtain a homogeneous damage-free layer with a relatively short process time, the proper ranges of the temperature and time and the analysis of the surface characteristics were required in detail. Recently, a comprehensive model for the thermal oxidation of GaN surfaces was presented by Yamada et al.,[14] and they explained the behavior of the Ga_2O_3 layers obtained when GaN was thermally oxidized in an oxygen atmosphere, and it was well schematically described.

In this study, the dry thermal oxidation for bulk GaN substrates at different temperatures was conducted to obtain sufficiently thick Ga_2O_3 layers bearing the extrinsic damages generated from MP and inherent defects in the GaN substrate. Furthermore, the performance of Ga_2O_3 on the GaN surface was discussed based on the experimental results. Afterward, CMP was performed at different times, and the variations in the surface characteristics were measured throughout the CMP process. To assess the final surface quality and the remaining chemical components on the surface of the GaN substrate, atomic force microscopy (AFM) and X-ray photoelectron spectroscopy (XPS) were used, respectively.

Experimental

Bulk GaN (0001) single crystal substrates (2 inch and 970 μm) that were grown by hydride vapor-phase epitaxy (HVPE) were prepared for the CMP process, which involved thermal treatment. β-Ga_2O_3 thick films with a thickness of 5.6 μm were grown on the bulk GaN single crystal substrates by furnace oxidation[15] and were employed for the MRR comparison between GaN and Ga_2O_3 with different pH values. The pH of the colloidal silica slurry was manipulated with a HNO_3 solution (Sigma Aldrich, 63.01 wt%), and the MRRs were characterized using an electronic balance (Ohaus, E02130, USA), which had an accuracy of 0.0001 g according to the weight loss. The MRR was calculated using Eq. 1:[16]

$$\mathrm{MRR} = \Delta m / \left(\rho \times r^2 \times t\right), \qquad [1]$$

where Δm (g) is the removal mass of the material, ρ (g/cm^3) is the density of GaN, r (cm) is the radius of the substrate, and t (h) is the polishing time. Prior to the CMP process, the thermal treatment was conducted at 700–1000°C for 5 h in ambient air in a home-made furnace to anneal the nitride single crystal. After thermal treatment, the thickness of the Ga_2O_3 layers was measured by scanning electron microscopy (SEM; JEOL, JSM-5900LV, Japan) and energy-dispersive X-ray spectroscopy (EDS; Oxford Instruments, X-Max, England), and

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Table I. GaN CMP conditions.

Name	Unit	Quantity
Platen rotation speed	RPM	30
Carrier rotation speed	RPM	30
Applied pressure	kg/cm^2	0.16
Feed rate of the slurry	ml/min	35
Polishing pad type	-	Suba-600
Slurry type	-	Colloidal silica
Abrasive particle size	nm	40
Particle concentration	%	30
pH	-	5.5
Polishing time	min	120

the changes in the GaN surface were observed via AFM (Park systems, XE-100, Korea). Afterward, the CMP process was conducted on a polishing machine (EK-3801D, Engis, Japan) with the colloidal silica slurry, at pH 5.5, on a SUBA-600 pad, and the detailed CMP conditions are shown in Table I. After performing the CMP process several times, the as-polished substrates were rinsed ultrasonically with acetone, ethanol, and deionized water for 15 min severally, and the substrates were subsequently dried in pure N_2 atmosphere. During the CMP process, the variation of each surface morphology was observed by white light interferometry (Nano System, Nano View, Korea), and the final surface morphology was assessed by AFM. The chemical changes in the polished surfaces after thermal treatment were analyzed using an XPS instrument (Thermo Fisher Scientific, K-alpha plus, USA) installed at the Hanyang LINC+ Analytical Equipment Center (Seoul).

Results and Discussion

Comparison of the MRRs for GaN and Ga_2O_3 with different pH values.—Figure 1 shows the comparison of the MRRs for GaN and Ga_2O_3 using colloidal silica slurry with different pH values. GaN had a higher MRR of 40.2 nm/h under an acidic condition than under neutral and basic conditions. The MRR during the CMP of GaN without thermal treatment could be obtained. For Ga_2O_3, the MRR, which was almost 1000 times higher than that for GaN, was observed under all conditions, and the highest MRR of 51.0 μm/h was observed under the acidic condition. This result indicated the MRR of the thermally treated GaN, and it showed that the acid condition exhibited the highest efficiency in the CMP process for both of them.[17,18] Thus, to obtain the shortest process time, the colloidal silica slurry in the acid condition at pH 5.5 was used during the CMP process.

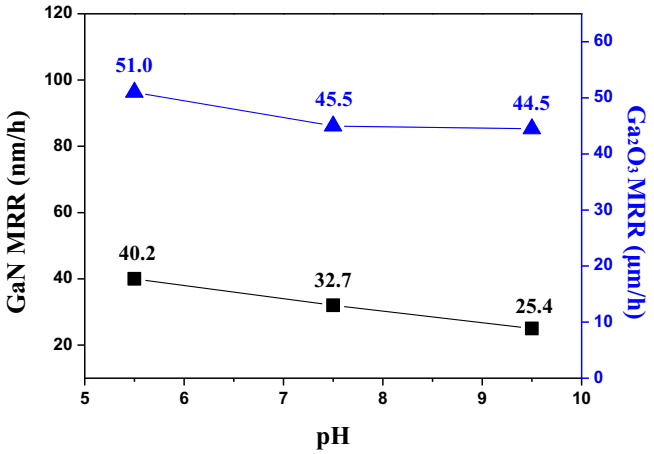

Figure 1. MRR of the GaN and Ga_2O_3 with different pH values.

Performance of the Ga_2O_3 compounds.—The surface morphology and roughness average (Ra) value of the GaN substrates after thermal treatment at different temperatures are shown in Fig. 2. Figure 2a shows the relatively low surface Ra of 0.510 nm and flattened shape although several defects including micro-scratches could be observed on the polished surface. Figure 2b shows the surface became slightly irregular, where the scratches generated from the MP process were remarkably revealed with a Ra of 7.286 nm. The surface depth and roughness increased around the micro-scratches formed in the MP process with a Ra of 8.422 nm as shown in Fig. 2c. Figure 2d shows the overall irregular surface, which was attributed to the formation of several small grains and the surface deformation with a Ra of 67.164 nm. The relatively large grains and significant surface deformation were clearly observed on the surface with a Ra of 112.305 nm as shown in Fig. 2e. Generally, when GaN is oxidized at high temperatures above 800°C in ambient air or an oxygen atmosphere, it reacts with oxygen atoms to form Ga_2O_3 compounds and nitrogen oxides according to Eq. 2[14] regardless of the crystallinity or crystal facet.

$$GaN + O_2 \rightarrow GaOx + NOx \quad [2]$$

GaN was subjected to thermal treatment to form Ga_2O_3 compounds with the desorption of nitrogen oxides, and the Ga_2O_3 compounds could migrate to the vicinity of the GaN surface defects and accumulate to form nuclei around the surface defects.[15] The migration of Ga_2O_3 compounds increased as the temperature increased. In addition, the migrated Ga_2O_3 compounds started forming crystalline grains at 900°C, and the formed crystalline grains could densely populate the surface at 1000°C.[14,15] Thus, the surface morphology became less uniform as thermal treatment temperature increased.

Thickness of the Ga_2O_3 layers on the GaN surface.—To measure the thickness of the oxide layers and the surface morphology according to the thermal treatment temperature, the cross-section of the thermally treated GaN substrates were analyzed via SEM and EDS, and the results are shown in Fig. 3. When we observed the changes in the thickness with different temperatures, an oxide layer with a thickness of about 2 μm was formed at 700°C, and a significantly thick oxide layer of about 70 μm thickness was formed at 800°C. During thermal treatment at 900°C, the thickness of the oxide layer increased by about 12 μm of that during thermal treatment at 800°C. However, the thickness of the oxide layer reduced drastically, and a rough and distinct oxide film with a thickness of 5.6 μm was confirmed at 1000°C. As described in Fig. 2, it can be determined that the reaction of GaN with oxygen atoms and the formation of the Ga_2O_3 layer hardly occurred below 800°C, and the substantial formation of Ga_2O_3 compounds started at 800°C. Therefore, it was considered that the thickness of the Ga_2O_3 layer and the oxide intensity increased as the amount of migrated Ga_2O_3 compounds increased with increasing temperature.[15] However, the decrease of overall thickness of the Ga_2O_3 layer and the highest oxide intensity was observed at 1000°C due to the densification and crystallization of Ga_2O_3.[19]

Variation of the surface morphology during the CMP process.—Figure 4 shows the morphological changes in the thermally treated GaN surfaces at different temperatures and CMP process times. For the GaN without thermal treatment, the micro-scratches that were not seen after the MP process were revealed after 1 h of CMP, and they were gradually removed through continuous chemical mechanical reaction. In the thermally treated GaN at 700°C, micro-scratches were revealed on the surface after 1 h of CMP. After 2 h of CMP, multiple small pits were observed along with the remaining scratches. In the thermally treated GaN at 800°C, the Ga_2O_3 compounds formed to around micro-scratches were completely removed after 1 h of CMP, and the surface uniformity and smoothness improved after 2 h of CMP. For the thermally treated GaN at 900°C, several pits were generated on the surface with uneven surface morphology after 1 h of CMP, and the depth of the pits significantly increased as the CMP time increased. For the thermally treated GaN at 1000°C, Ga_2O_3 crystalline grains were formed, and a substantial irregular surface morphology was observed

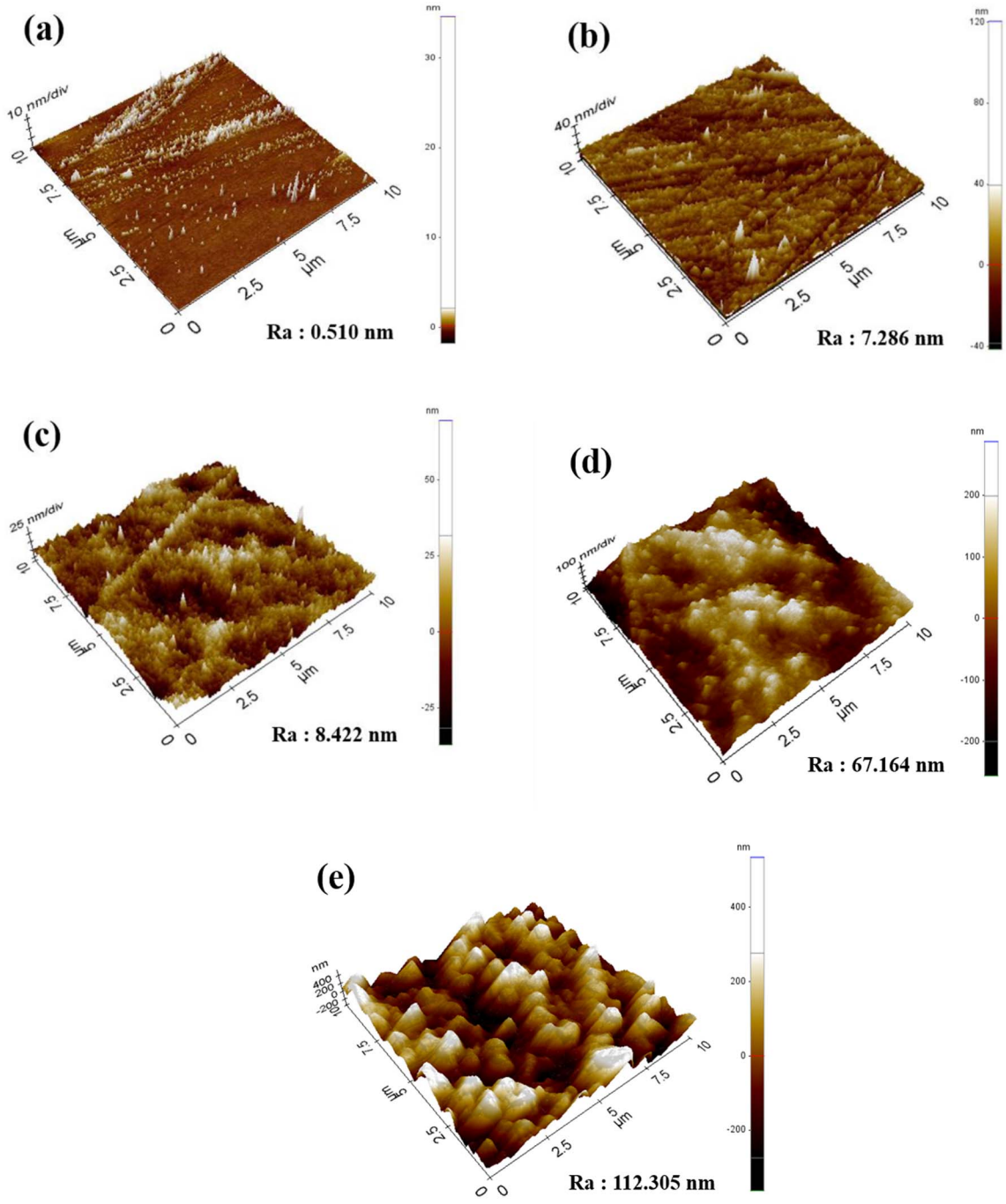

Figure 2. Three-dimensional AFM images of GaN surfaces after thermal treatment with different temperatures for 5 h (magnification × 10 μm). (a) after MP process, and after thermal treatment at (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

along with the crucial deformation of the surface. Similarly, the depth of the pits produced on the surface increased with increasing the CMP time. After the entire process was completed, AFM measurements were performed to analyze the more precise surface characteristics, and the results are shown in Fig. 5.

Assessment of the final surface quality.—The surface morphology and Ra of the polished GaN after thermal treatment are shown in Fig. 5. Figure 5a shows a Ra of 0.571 with micro-scratches and an irregular surface. It was determined that the damaged layer of the GaN surface was still remained, thus necessitating additional polishing to remove a sufficient amount of the damaged layer by chemical reaction with a silica abrasive. Figure 5b shows a relatively uniform surface with a Ra of 0.664 nm although various small pits are observed on the polished surface. A smooth and defect-free surface with a Ra of 0.377 nm is shown in Fig. 5c. Figure 5d and Figure 5e show the irregular surface with a Ra of 10.223 and 36.742 nm, respectively. The cross-sectional morphologies on the polished GaN surface are shown in Fig. 6. Figure 6a shows the micro-scratch generated from the MP process, which has a depth of 2.5 nm and a width of 0.5 μm. Figure 6b shows several small pits with a depth of 1.5 nm and widths of 1.5 and 2.5 μm, and multiple micro-pits caused by micro-scratches were observed on the surface. A defect-free and the most uniform surface was clearly displayed on the polished surface in Fig. 6c. In Fig. 6d, the bigger pits are shown with surface deformation, which have depths of 40 and 60 nm and widths of 1.5 and 3.0 μm, respectively. The biggest

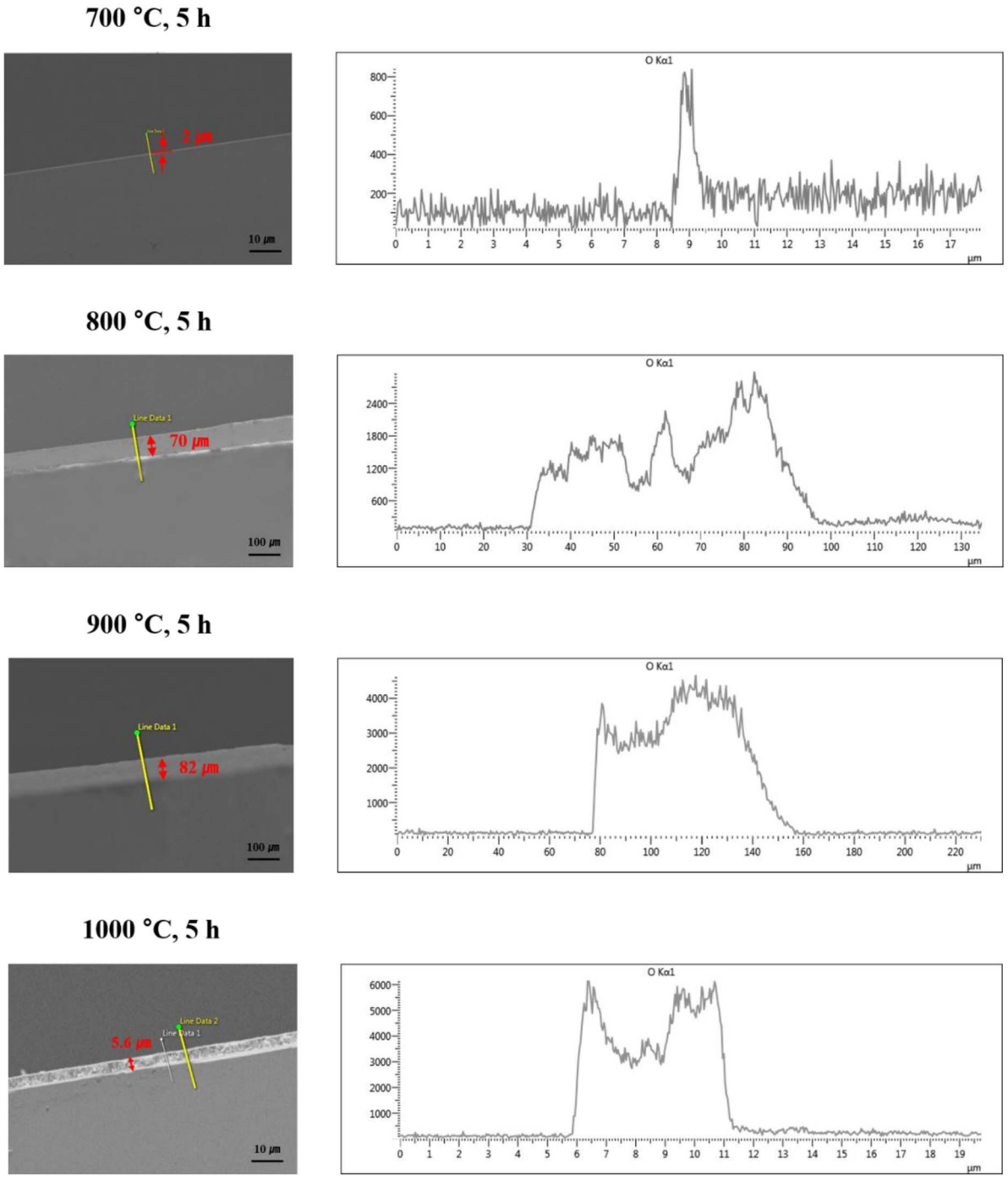

Figure 3. Cross-sectional SEM images and EDS graph of the thermally treated GaN with different temperatures for 5 h.

pit with a depth of 90 nm and a width of 6 μm was observed on the polished surface with intensive surface deformation as shown in Fig. 6e.

These results are related to the mechanism of the atomic migration on the GaN surface. This mechanism can be classified into interfacial reaction-controlled and diffusion-controlled reaction. The interfacial reaction-controlled is a reaction limited at the interface between Ga_2O_3 and GaN. In the case of the diffusion-controlled reaction, Ga atoms diffused from the subsurface to the surface with the internal decomposition of GaN. These reactions tend to alternate between an interfacial reaction-controlled and a diffusion-controlled reaction as the temperature increases, and this mainly occurs at temperature above 800°C.[20]

Therefore, for thermal treatment at 700°C, the GaN surface was slightly oxidized via the weak interfacial reaction-controlled, and it was shown that the oxidized inherent defects in strongly damaged layer were removed with generating multiple small pits and micro-pits on the surface throughout the CMP process. During thermal treatment at 800°C, the interfacial reaction-controlled was to be dominant and the sufficient damaged layer of GaN was transformed to Ga_2O_3 layers, resulting in a uniform surface morphology despite the chemical mechanical reaction.[14,21] However, for thermal treatment at 900°C, multiple pits and various defects were generated with surface deformation because the interfacial reaction-controlled and the diffusion-controlled reaction occurred simultaneously.[21] Thus, the pit size and depth were considered to increase as the CMP process progressed

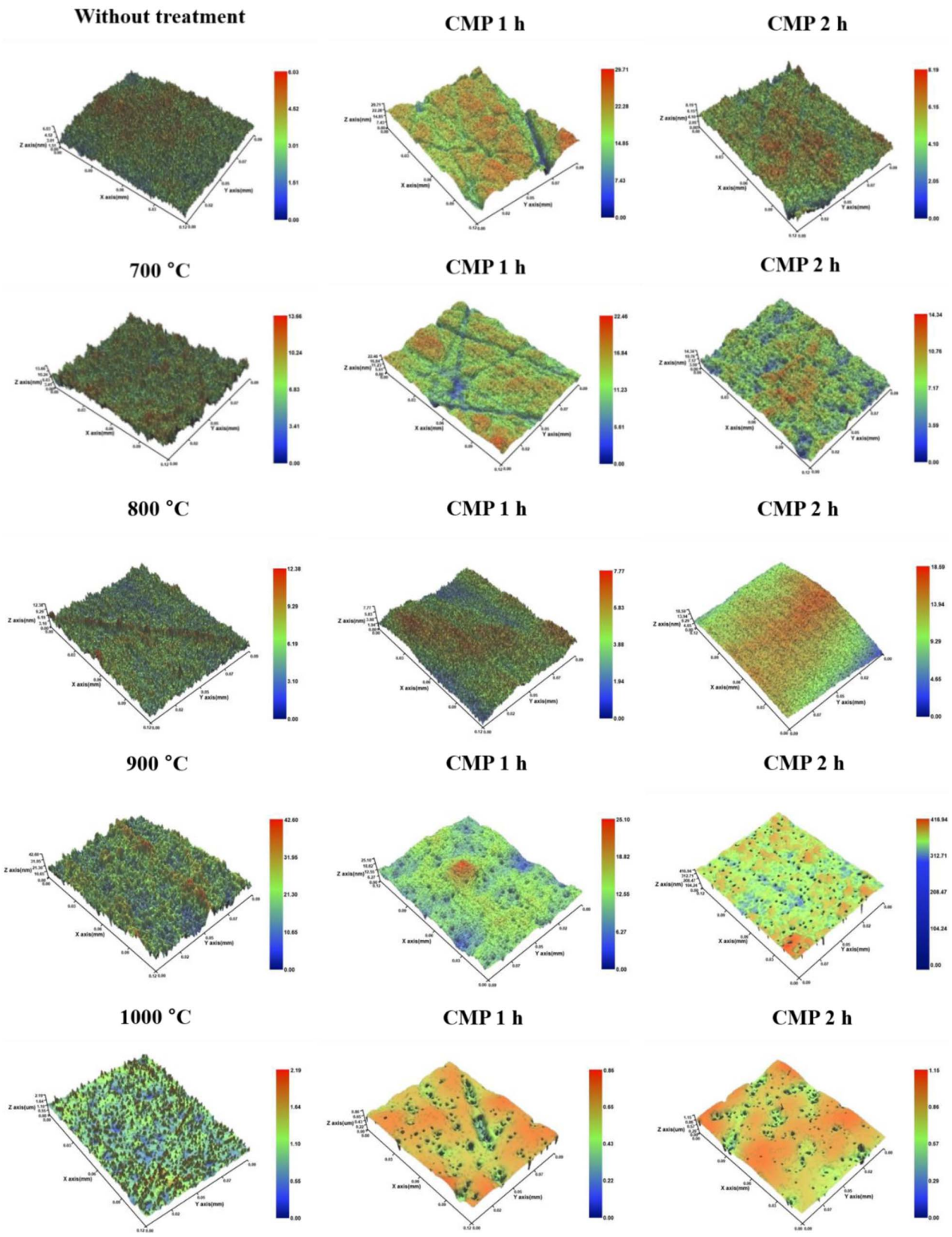

Figure 4. Three-dimensional white light interferometry images of the thermally treated GaN surfaces at different temperatures during the CMP process (magnification $\times$ 100 μm).

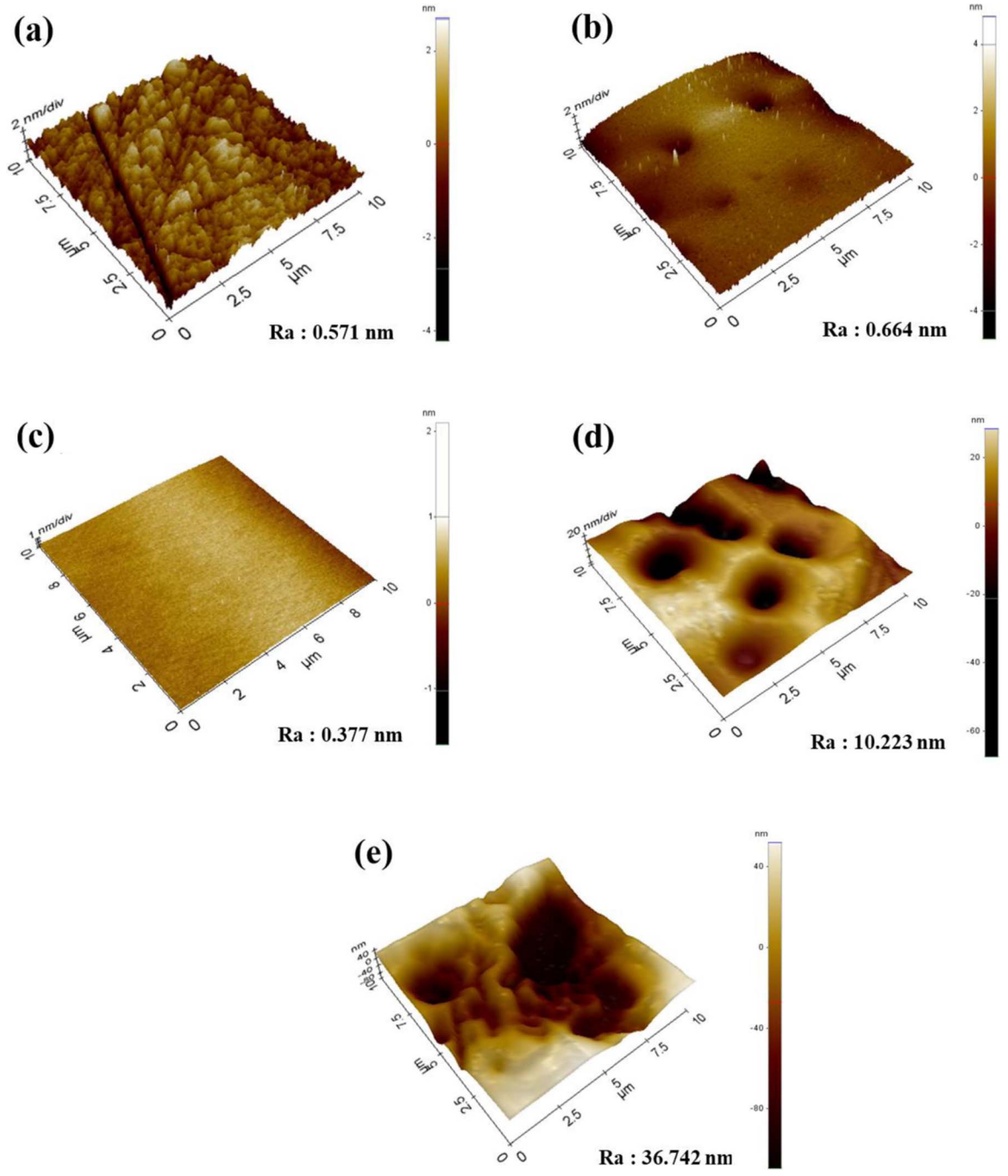

Figure 5. Three-dimensional AFM images of the polished GaN surfaces after thermal treatment for different temperatures (magnification × 10 μm). (a) GaN without treatment, and the thermally treated GaN at (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

with continuous chemical mechanical reaction.[3] In the case of thermal treatment at 1000°C, the diffusion-controlled reaction occurred dominantly with intensive surface deformation, resulting in the irregular surface morphology. Similarly, it was determined that the pit size and depth increased as the CMP time increased.

XPS analysis for surface chemistry.—The XPS analysis was performed to characterize the surface chemistry of the polished GaN surfaces after thermal treatment, as shown in Fig. 7. The Ga3d spectra peaks ranging from 18.6 to 19.6 eV were used to characterize the surface properties and can be categorized as Ga metal (18.6 eV), Ga_2O_3 (20.4 eV), and GaN (19.6 eV) depending on the chemical reaction and residual components. Eqs. 3–5[11] show the chemical reactions, which can occur throughout the CMP process.[18]

$$2GaN + 6OH^- + 6H^+ \rightarrow Ga_2O_3 + 3H_2O + N_2 \quad [3]$$

$$Ga_2O_3 + 6H^+ \rightarrow 2Ga^{3+} + 3H_2O \quad [4]$$

$$Ga_2O_3 + 6OH^- \rightarrow Ga_2O_3{}^{3-} + 3H_2O \quad [5]$$

Eq. 3 is the general oxidation procedure in GaN CMP and Eqs. 4–5 are the procedures for the decomposition of Ga_2O_3 compounds. In this process, the reaction expressed by Eq. 3 was replaced by that expressed by Eq. 2, and the GaN surface was transformed to Ga_2O_3 layers, which could be decomposed and flattened by chemical mechanical reaction with colloidal silica abrasives. Thus, the polished GaN surfaces were assessed by the molar ratio of the residual components via XPS

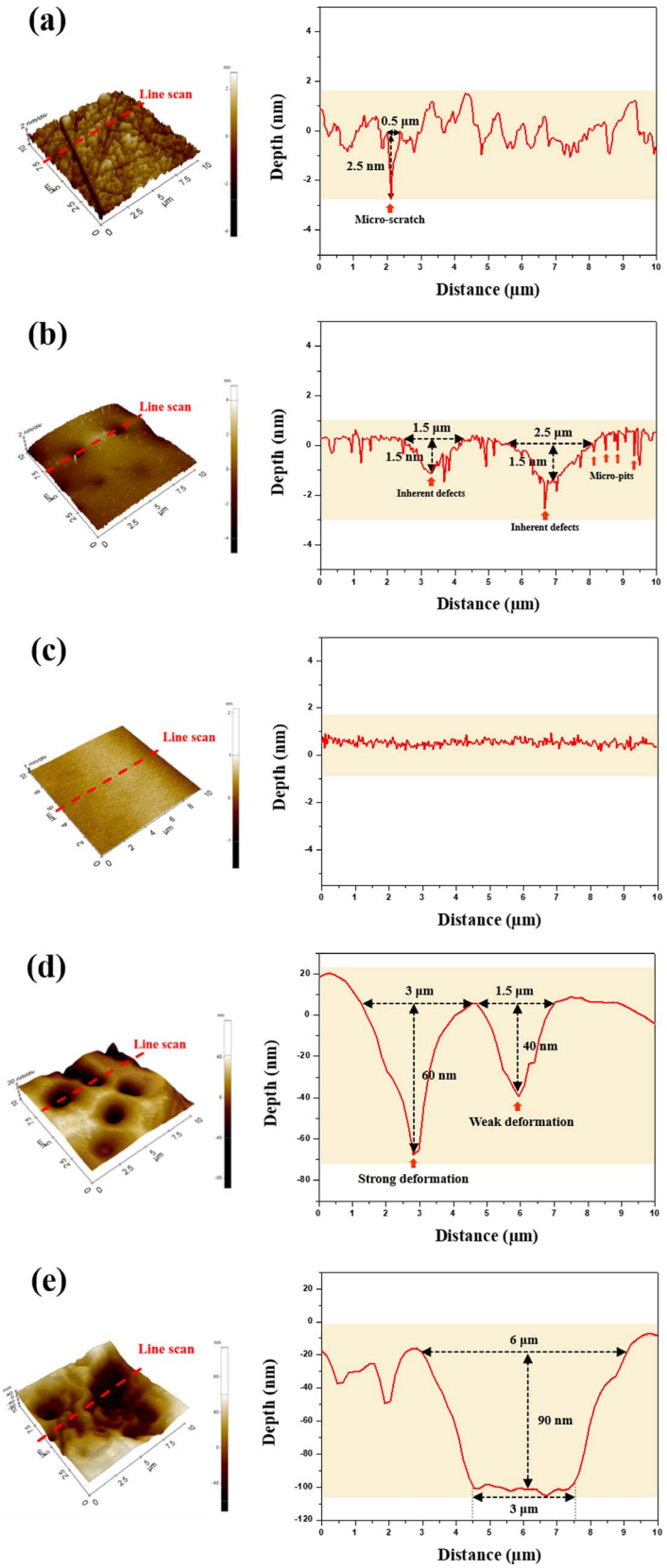

Figure 6. The cross-sectional morphologies on the polished GaN surface after thermal treatment for different temperatures. (a) GaN without treatment, and the thermally treated GaN at (b) 700°C, (c) 800°C, (d) 900°C, and (e) 1000°C.

analysis, and each molar ratio is displayed and explained in Fig. 8. When the molar ratios of the GaN components were compared, the molar fraction showed a reduction of about 0.03 from without thermal treatment to 800°C, while it increased to about 0.04 from 800 to 1000°C. In contrast, the molar fraction of Ga_2O_3 increased by about 0.04 from without thermal treatment to 800°C, and it decreased by about 0.07 from 800 to 1000°C. Ga metal showed a low molar fraction below 0.08 in the whole range, and it was found that the highest

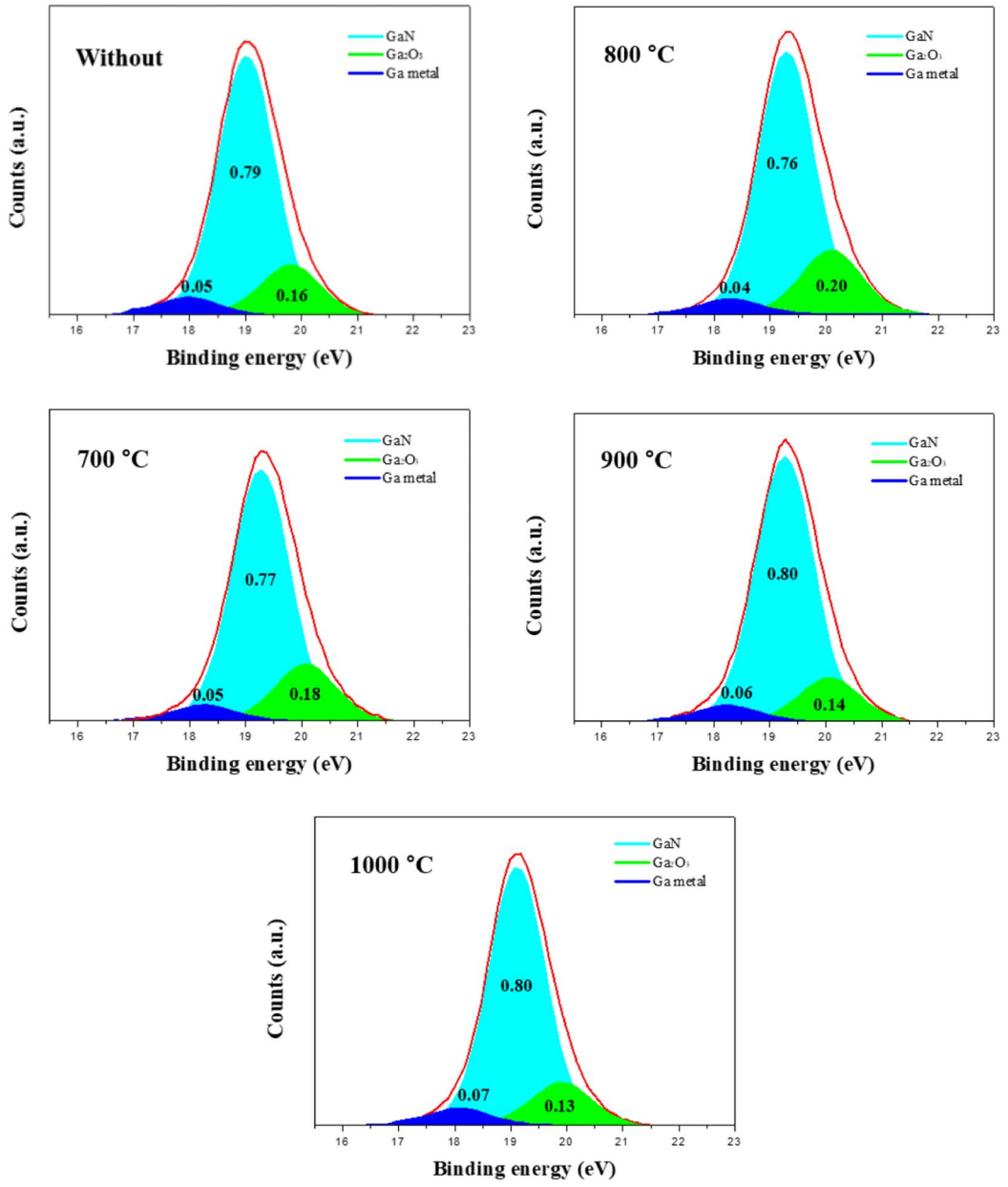

Figure 7. Binding energies of core level of Ga3d scan.

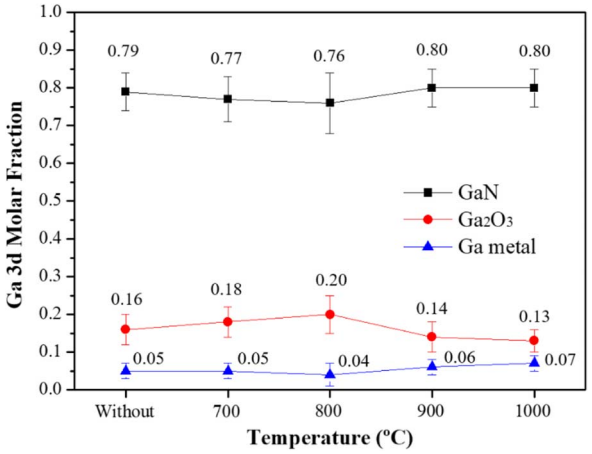

Figure 8. Ga3d core level peak of each component molar ratio.

value and the lowest value were about 0.07 at 1000°C and 0.04 at 800°C, respectively.

In light of the correlation between the CMP process time and MRR, the Ga_2O_3 compounds produced by thermal treatment were almost removed after 2 h of CMP. In general GaN CMP model as shown in Fig. 9a and Fig. 9b, the water molecules in the CMP slurry penetrate the atomic bonds of GaN substrate surface, and it can convert GaN to Ga_2O_3 with formation of a thin Ga_2O_3 layer on the GaN surface. This thin Ga_2O_3 layer can be removed by the physical friction of the rotating platen with mechanical force. In the case of this process, the polished GaN surfaces can be differently varied according to the effects of thermal treatments. Thus, the real contact area between CMP pad and substrate surface can be varied with the surface roughness and morphology of the polished GaN after thermal treatment, and the real contact area rate indicates the conversion rate from GaN to Ga_2O_3.[22,23] As earlier mentioned, several defects and pits were generated at temperature of above 900°C by diffusion-controlled reaction. Thus, the surfaces of thermally treated GaN from 900 to 1000°C where the pits existed have the relatively weak and unstable dangling bonds of Ga

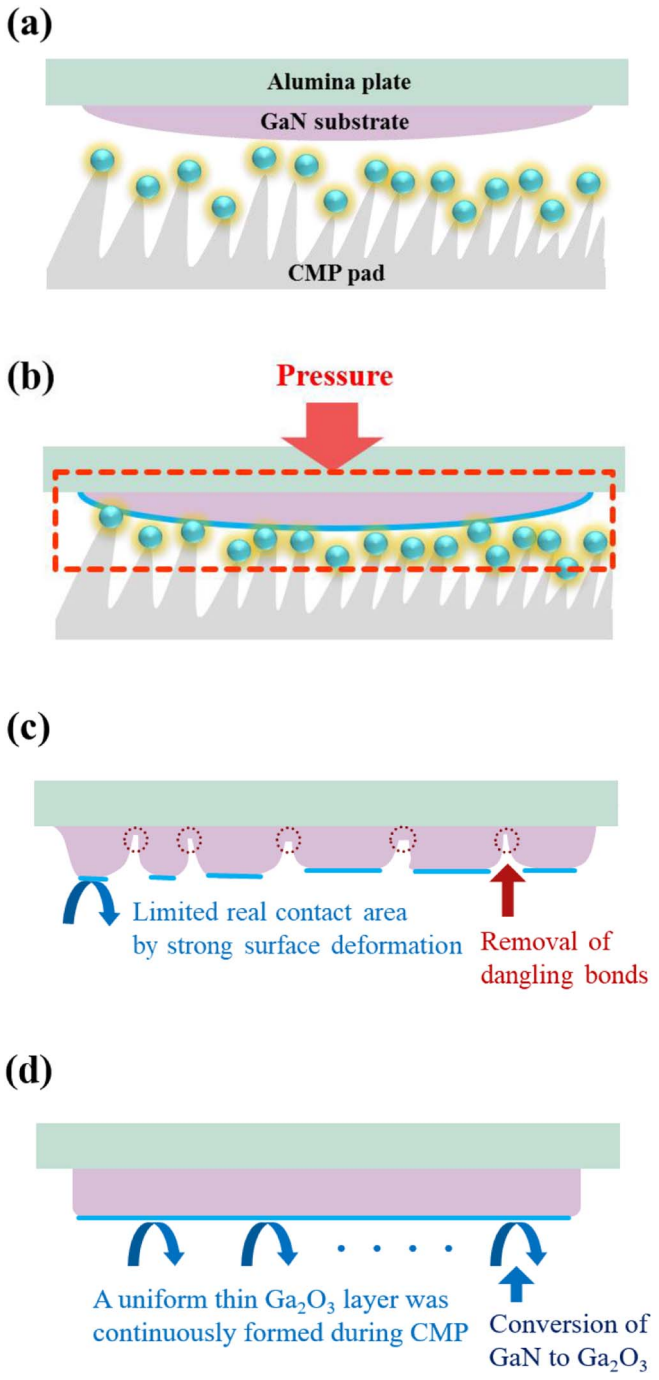

Figure 9. The GaN CMP model. Schematic diagram of (a) GaN CMP, (b) GaN CMP with applied pressure, and conversion and removal mechanism at temperature of (c) 900–1000°C, and (d) 800°C.

atoms, and these can be easily removed during CMP. However, since the surface area without pits remained with the stronger and stable atomic bonds of GaN, this area was shown in the irregular surface during CMP. As shown in Fig. 9c, therefore, the surface directly contacted with CMP pad can be composed of the limited real contact area. In contrast, in the case of the thermally treated GaN at 800°C, the entire polished surface can be contacted with CMP pad because the most uniform surface can be obtained on the GaN surface without any pits and defects as shown in Fig. 9d. Therefore, it was found that the highest molar fraction of Ga_2O_3 was obtained at temperature of 800°C. The molar fraction of Ga metal shows the extent of the damaged layers on the substrate surface in GaN CMP.[11] Therefore, for the molar fraction of Ga metal, the lowest value was obtained at 800°C with a defect-free surface, and the highest value was obtained at 1000°C, where the surface deformation and defects were mostly generated on the surface.

Conclusions

The dry thermal oxidation of bulk GaN substrates in ambient air was investigated at different temperatures, and the CMP process was conducted under the acidic condition at pH 5.5. The results showed that a defect-free surface with a Ra of 0.377 nm and high MRR of about 51 μm/h were obtained by CMP after thermal treatment at 800°C. The thermally treated GaN at different temperatures showed the different surface qualities according to mechanisms of the atomic migration on the GaN surface. During thermal treatment of GaN, the interfacial reaction-controlled mechanism was dominant at temperature of 800°C, and the diffusion-controlled reaction mechanism was to be dominant at temperatures above 900°C. Thus, the thermally treated GaN at temperatures from 900 to 1000°C showed several pits and defects on the surface throughout the CMP process, and the pit size and depths tended to increase as the CMP time increased. The molar fractions of Ga_2O_3 were measured to the polished GaN surfaces after different thermal treatments. The results showed that the highest molar fraction of 0.20 and the lowest molar faction of 0.13 were obtained at 800 and 1000°C, respectively. It was found that the conversion rate from GaN to Ga_2O_3 was proportional to the real contact area between CMP pad and GaN substrate surface during the CMP process.

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