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Paper:

Surface Finishing of Single-Crystal SiC and GaN Wafers Using a Magnetic Tool in H_2O_2 Solution

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To remove the microroughness and subsurface damage on the SiC and GaN surface efficiently, a surface finishing technique using a magnetic tool holding iron particles in a hydrogen peroxide solution is developed. This technique utilizes OH radicals generated from the iron catalytic particles in a hydrogen peroxide solution, and can be used to preferentially remove the topmost convex part on the surface, resulting in an atomically smooth surface. We employed this polishing technique to finish the surfaces of 2-inch SiC and 2-inch GaN wafers. The surface roughness before and after finishing was measured by scanning white light interferometric microscopy and atomic force microscopy. In addition, the material removal rate was calculated by weight loss due to the finishing process. The results show that the surface roughness on the SiC and GaN wafers is markedly improved. Moreover, the surface waviness and flatness of these wafers before and after finishing did not deteriorate. Atomic force microscope images indicate that an atomically flat SiC surface with a roughness value below 0.1 nm RMS and a GaN surface with atomic step and terrace structures were achieved. Our proposed finishing technique is effective in improving the surface microroughness of SiC and GaN wafers.

Keywords: SiC, GaN, surface smoothing, OH radical, abrasive Fe

1. Introduction

Silicon carbide (SiC) and gallium nitride (GaN) have excellent properties, such as wide band gap, high electron mobility, and high thermal conductivity. Therefore, these materials attract much attention as the optimum semiconductors for high-power electronics [1]. To fabricate the device using these materials, atomic-scale smooth and damage-free SiC and GaN substrates are needed to make a high-quality epitaxial layer. However, SiC and GaN substrates are relatively difficult to machine because of their high hardness and chemical inertness.

To manufacture SiC and GaN wafers, wire-cutting of bulk ingots and multiple lapping and polishing methods were conducted in a step-by-step manner. Generally, the SiC and GaN wafers were lapped using diamond and/or alumina abrasives. The surface roughness and crystallographic damage can be reduced effectively with the lapping process. However, this material removal is based on mechanical actions. Therefore, a damaged layer with numerous scratches and cracks is left on the polished surfaces [2, 3]. Additionally, mechanical lapping/polishing techniques require a long machining time, and SiC and GaN wafers cannot be easily obtained with high flatness. Recently, a grinding technique has been developed to planarize the SiC wafer in a short time [4, 5], resulting in mirror surfaces that can be manufactured with high efficiency and low subsurface damage [6]. To remove the subsurface damage and microroughness due to the mechanical polishing/grinding, chemical mechanical polishing (CMP) methods using a hard polishing pad with abrasives, such as colloidal silica, alumina, and ceria abrasives, have been developed. Atomically flat and damage-free SiC [7–11] and GaN [12–17] surfaces have been reportedly obtained. However, as the wafer size becomes larger, planarization of wafers on a large scale becomes more challenging because of the difficulty in controlling the polishing parameters and maintaining proper machine stiffness and motion accuracy. Therefore, it is difficult to obtain highly smooth, large-sized SiC and GaN wafers in CMP.

In our previous study, we proposed a polishing technique utilizing hydroxyl radicals (OH radicals) generated on the surface of an iron catalyst in hydrogen peroxide (H_2O_2) solution [18–22]. This planarization technique is performed as follows. First, Fe catalyst is immersed in H_2O_2 solution and ionized to ferrous iron (Fe^{2+}). Fe^{2+} then reacts with the H_2O_2 in the following reaction:

$$Fe^{2+} + H_2O_2 \rightarrow OH\cdot + OH^- + Fe^{3+}. \quad (1)$$

Although OH radical is a strong oxidant, the lifetime of OH radicals is very short. Therefore, the generated OH radicals react with the uppermost area of the SiC substrate to form an oxide layer when the Fe catalyst comes into contact with the SiC surface, as shown in the reaction:

$$SiC + 4OH\cdot + O_2 \rightarrow SiO_2 + 2H_2O + CO_2. \quad (2)$$

The oxide layer (SiO_2) produced on the SiC substrate is preferentially removed, resulting in a smooth and

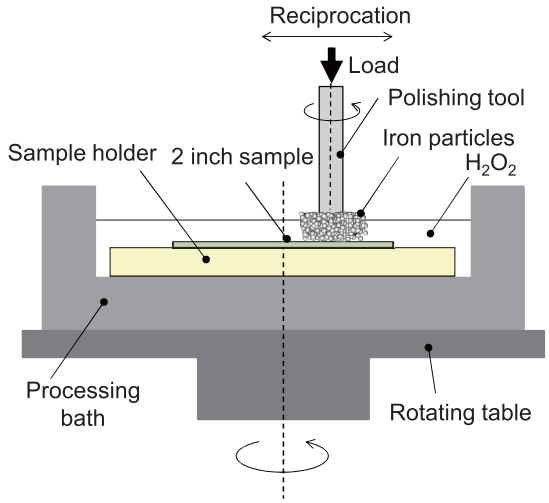

Fig. 1. Schematic drawing of the experimental setup.

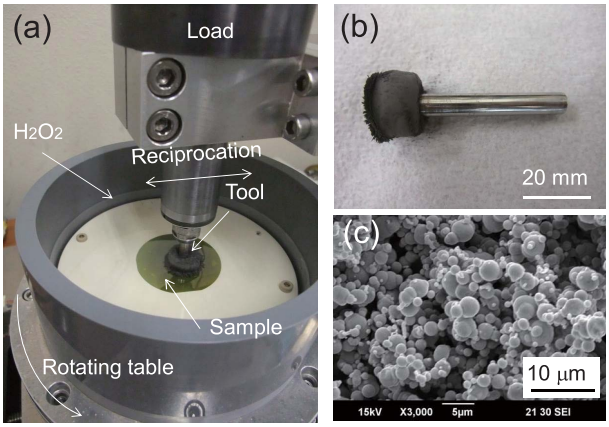

Fig. 2. (a) Experimental setup, (b) polishing tool, and (c) SEM image of iron.

damage-free SiC surface [20]. However, this polishing technique, which utilizes a slurry (iron particles and H_2O_2 solution) and an Fe lapping plate, requires considerable time to flatten and smooth the SiC wafer because of the undulation of the wafer surface before polishing [21].

In this work, to realize the smoothing of the entire surfaces of the SiC and GaN wafers using this removal mechanism, we have attempted to develop a tool-polishing technique [22] as an easy, simple, and low-cost method that can remove the surface irregularity induced by mechanical polishing/grinding. By tracing the surface of SiC and GaN wafers with a magnetic polishing tool that adsorbs iron particles in H_2O_2 solution, the stable contact between the iron catalyst particles and the wafers could be realized, resulting in highly smooth, large-sized SiC and GaN wafers. We evaluated the feasibility of removing and smoothing 2-inch SiC and GaN wafers in comparison with 0.25-μm diamond-lapped SiC and GaN wafers.

2. Experimental Procedure

Polishing of the SiC and GaN wafers was performed with a magnetic polishing tool, holding the iron particles in H_2O_2 solution. **Fig. 1** shows the schematic drawing of the experimental setup. The processing bath, filled with an H_2O_2 solution, was fixed to a rotating table. A sample holder was arranged in the center of the processing bath. The sample was fixed to the sample holder with electron wax. **Fig. 2(a)** shows a photograph of the polishing setup. The polishing tool, which adsorbs iron particles, applied a pressure against the surface on a sample. **Fig. 2(b)** shows a photograph of the polishing tool. The polishing tool was made of neodymium magnet, and its diameter was 10 mm. The iron particles were attached to the magnetic tool. **Fig. 2(c)** shows an SEM image of the iron particles. As can be seen in **Fig. 2(c)**, iron particles exhibited a relatively spherical shape, with sizes of less than a few microns.

In this study, a 2-inch 4H-SiC wafer (4° off-axis) and a freestanding 2-inch GaN wafer, prepared by hydride vapor phase epitaxy, were used as samples. The polishing parameters were as follows: the rotating speed of the table was set to 30 min^{-1}. The rotating speed of the polishing tool was set to 250 min^{-1}. The feeding speed and reciprocating distance of the polishing tool were set to 3 mm/s and 20 mm, respectively. The polishing load was applied onto the magnetic tool by adding a weight of 2 kg. The polishing load can be adjusted by adding weights or counterweights as appropriate. The process times of Si-face SiC wafer, C-face SiC wafer, and Ga-face GaN wafer were 13 h, 4 h, and 17 h, respectively. Prior to the experiments, each sample was mechanically polished with 0.25-μm diamond abrasives. The surface roughness on the sample surface was measured with scanning white light interferometric (SWLI) microscopy and atomic force microscopy (AFM). To clarify the morphological improvement due to finishing, the same 24 points on the wafers were measured before and after polishing, with 6-mm intervals over the entire area. Moreover, the chemical bonding states of the sample surfaces were analyzed by X-ray photoelectron spectroscopy (XPS). The material removal rate (MRR) was determined by measuring the weight loss before and after polishing. The MRR was calculated as follows:

$$\mathrm{MRR} = \frac{\Delta m}{\rho S T} \quad \cdots\cdots\cdots\cdots\cdots\cdots \quad (3)$$

where Δm is the weight loss of the sample, ρ is the density of the sample, S is the area of the sample, and T is the polishing time.

3. Results and Discussion

3.1. Polishing of the 2-inch SiC Wafer

To examine the surface roughness before and after polishing, the surface roughness on the SiC wafer was measured by SWLI microscope. **Fig. 3** shows SWLI images

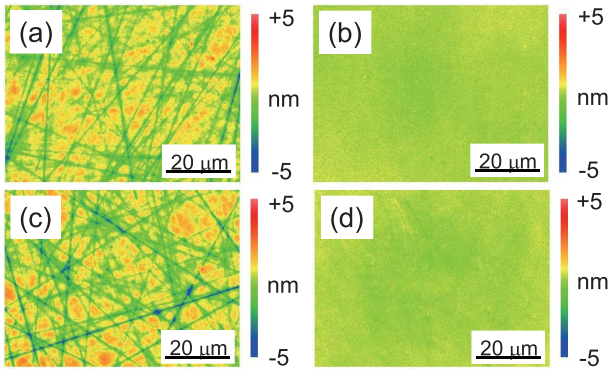

Fig. 3. Optical interferometer images of SiC surfaces. (a) Pre-polished Si-face 4H-SiC surface (PV: 7.941 nm, *Ra*: 0.591 nm, RMS: 0.773 nm), (b) polished Si-face 4H-SiC surface (PV: 1.731 nm, *Ra*: 0.124 nm, RMS: 0.156 nm), (c) pre-polished C-face 4H-SiC surface (PV: 15.673 nm, *Ra*: 0.755 nm, RMS: 0.988 nm), and (d) polished C-face 4H-SiC surface (PV: 1.800 nm, *Ra*: 0.141 nm, RMS: 0.176 nm).

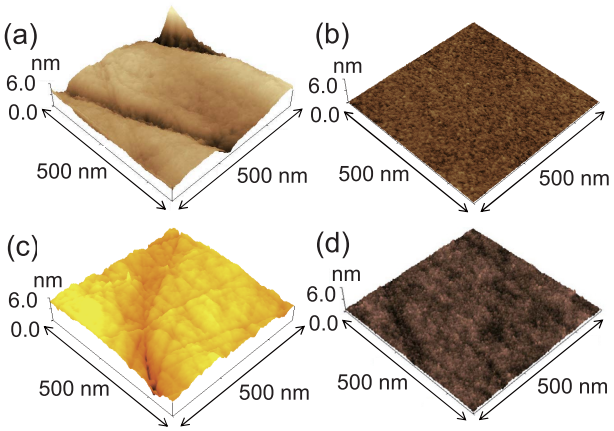

Fig. 4. AFM images of SiC surfaces. (a) Pre-polished Si-face 4H-SiC surface (PV: 7.052 nm, *Ra*: 0.782 nm, RMS: 0.995 nm), (b) polished Si-face 4H-SiC surface (PV: 0.575 nm, *Ra*: 0.043 nm, RMS: 0.054 nm), (c) pre-polished C-face 4H-SiC surface (PV: 7.299 nm, *Ra*: 0.613 nm, RMS: 0.791 nm), and (d) polished C-face 4H-SiC surface (PV: 1.582 nm, *Ra*: 0.122 nm, RMS: 0.155 nm).

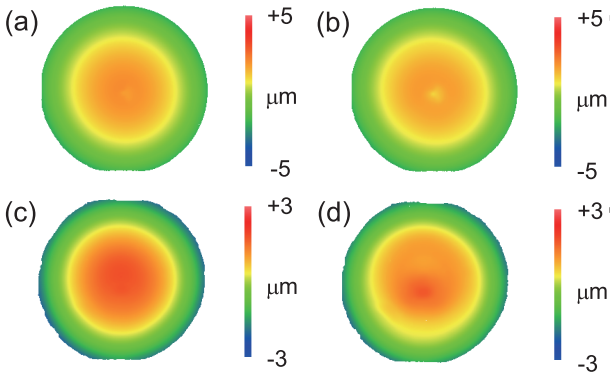

Fig. 5. Optical interferometer microscope images of 2-inch SiC wafers obtained by the stitching application. (a) Pre-polished Si-face 4H-SiC surface, (b) polished Si-face 4H-SiC surface, (c) pre-polished C-face 4H-SiC surface, and (d) polished C-face 4H-SiC surface.

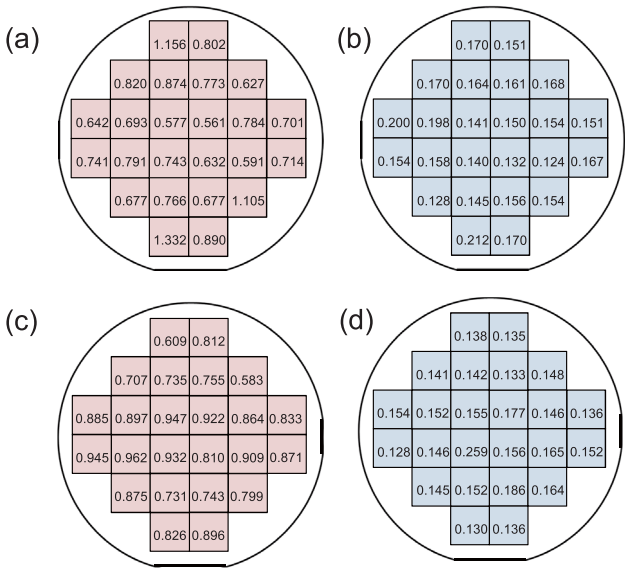

Fig. 6. Values of average roughness (*Ra*) at 6-mm intervals over the entire area of the polished 2-inch 4H-SiC 4° off-axis wafers, measured by optical interferometry (72 μm × 54 μm). (a) Pre-polished Si-face 4H-SiC surface, (b) polished Si-face 4H-SiC surface, (c) pre-polished C-face 4H-SiC surface, and (d) polished C-face 4H-SiC surface. The measurement included a total of 24 points.

of a surface on the Si-face and C-face of the SiC wafers before and after polishing. The measurement area was 72 μm × 54 μm. As shown in **Fig. 3**, many scratches on the Si-face and the C-face of the SiC surfaces were totally removed and atomically smoothed with an RMS roughness below 0.2 nm. **Figs. 4(a)** and **(b)** show AFM images of the Si-face SiC surface before and after polishing. The values of the surface roughness on the Si-face SiC surface before polishing were PV: 7.052 nm, *Ra*: 0.782 nm, and RMS: 0.995 nm. In contrast, those values after polishing were PV: 0.575 nm, *Ra*: 0.043 nm, and RMS: 0.054 nm. Numerous scratches on the pre-polished surface were remarkably removed to obtain an atomically flat and smooth surface by our proposed method. On the other hand, **Figs. 4(c)** and **(d)** show AFM images of the C-face SiC surface before and after polishing. We consider that its surface roughness follows the same trend as that of the Si-face SiC.

To clarify the surface flatness and surface waviness before and after polishing, we utilized the SWLI microscope with a stitching application, a method utilized to expand the measured region. **Fig. 5** shows SWLI images of the Si-face and C-face SiC wafers before and after polishing, obtained by the stitching application. As seen in these figures, the wafer flatness and waviness before and after polishing were maintained, indicating that this method does not degrade surface flatness or waviness.

Figure 6 shows the RMS values for the entire area of

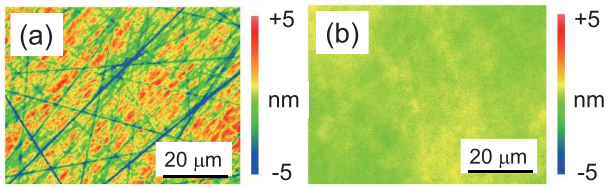

Fig. 7. Optical interferometer images of GaN surfaces. (a) Before polishing (PV: 31.007 nm, *Ra*: 1.250 nm, RMS: 1.639 nm) and (b) after polishing (PV: 1.957 nm, *Ra*: 0.182 nm, RMS: 0.227 nm).

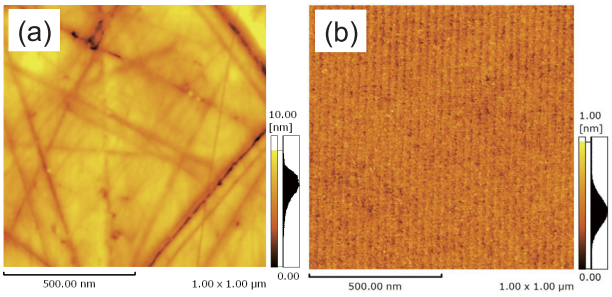

Fig. 8. AFM images of GaN surfaces. (a) Before polishing (PV: 11.254 nm, *Ra*: 0.991 nm, RMS: 1.299 nm) and (b) after polishing (PV: 1.040 nm, *Ra*: 0.087 nm, RMS: 0.109 nm).

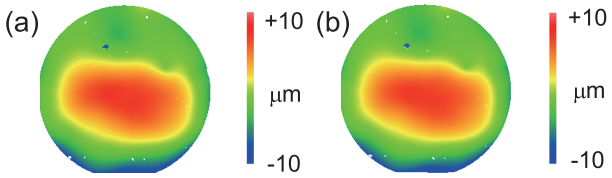

Fig. 9. Optical interferometer microscope images of 2-inch GaN surfaces. (a) Pre-polished Ga-face GaN wafer and (b) polished Ga-face GaN wafer.

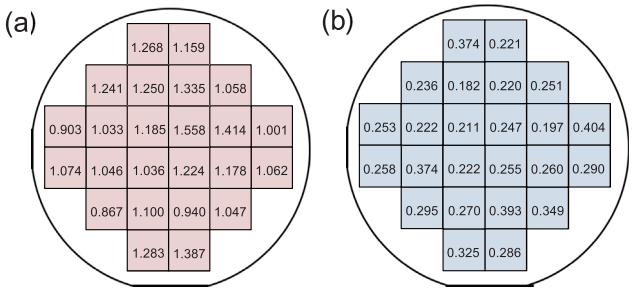

Fig. 10. Values of the roughness average (*Ra*) at 6-mm intervals over the entire area of polished 2-inch GaN wafers, measured by optical interferometry (72 μm × 54 μm). (a) Pre-polished Ga-face GaN surface and (b) polished Ga-face GaN surface. The measurement was 24 points.

the SiC wafer before and after polishing, measured by SWLI microscope (72 μm × 54 μm). Although the RMS values of the surface roughness on the points on the SiC wafer before polishing ranged from 0.5 nm to 1.4 nm, all of the points on the SiC wafer after polishing exhibited RMS surface roughness values less than about 0.2 nm. It is confirmed that none of the observed images displayed any scratches, as the surfaces were very smooth, showing images similar to **Figs. 3(b)** and **(d)**.

3.2. Polishing of the 2-inch GaN Wafer

Polishing of the 2-inch GaN wafer was performed in a manner similar to the polishing of the 2-inch SiC wafer. **Fig. 7** shows SWLI images of a surface on the Ga-face GaN wafer before and after polishing. The measurement area is 72 μm × 54 μm. As shown in **Figs. 7(a)** and **(b)**, many scratches on the GaN wafer before polishing were evident, and these were completely removed by polishing. **Fig. 8** shows AFM images of the Ga-face GaN surface before and after polishing. The measurement area is 1 μm × 1 μm. The surface roughness values for the Ga-face GaN surface before polishing were PV: 11.254 nm, *Ra*: 0.991 nm, and RMS: 1.299 nm. On the other hand, the surface roughness values for the polished Ga-face GaN substrate were PV: 1.040 nm, *Ra*: 0.087 nm, and RMS: 0.109 nm. Moreover, a periodic atomic step and terrace structure on the polished GaN surface is clearly observed. The result shown in the AFM image implies that a damage-free GaN surface could be realized by our proposed method. **Fig. 9** shows SWLI images of a 2-inch Ga-face GaN wafer before and after polishing, obtained by a stitching application. As seen in **Figs. 9(a)** and **(b)**, wafer flatness and waviness before and after polishing were maintained, just as in the polishing of the SiC wafers. **Fig. 10** shows the RMS values for the entire area of the pre-polished and polished GaN wafer, measured by SWLI microscope (72 μm × 54 μm). Although the RMS values of the surface roughness on the points of the GaN wafer before polishing ranged from 0.8 nm to 1.6 nm, these values for all of the polished points were markedly improved.

3.3. Surface Modification of SiC and GaN Surface

To identify the layer produced on the SiC and GaN surfaces by the OH radicals generated from the chemical reaction in H_2O_2 solution, we analyzed the chemical bonding state on the SiC and GaN surfaces before and after polishing. Before the XPS analysis, we prepared three types of SiC and GaN samples. **Fig. 11** presents a flow chart of the sample preparation for the XPS measurement. First, samples A and B were treated with mechanical polishing with 0.25-μm-sized diamond abrasives, while sample C was treated with our proposed polishing technique. Then, all samples were cut to 10 mm × 10 mm size and cleaned with H_2SO_4/H_2O_2 solution for 15 min to remove the contamination on these surfaces. Afterwards, sample B was immersed in Fenton's reagent for 6 h. Fenton's reagent is a solution of H_2O_2 with ferrous iron as a catalyst that produces OH radicals. In this study, the mixed $FeSO_4/H_2O_2$ solution was used as Fenton's reagent. Finally, three samples were rinsed with pure water, followed by analysis by XPS to examine the chemical bonding state

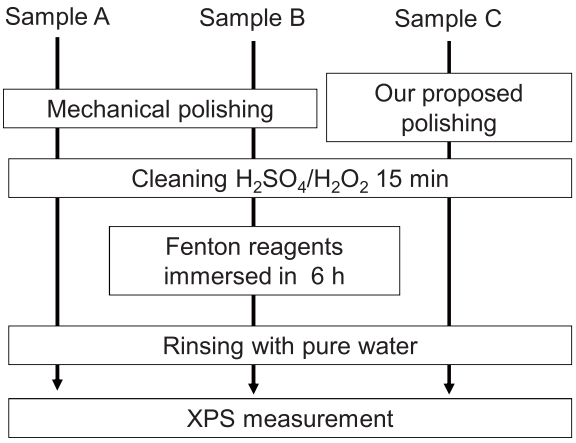

Fig. 11. Flow chart of the sample preparation for the XPS measurement.

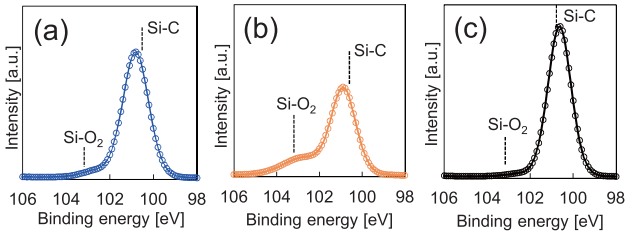

Fig. 12. (a) XPS Si2p spectra of SiC surface, (b) mechanically polished SiC sample before and after immersion in Fenton's reagent, and (c) SiC sample polished with our proposed polishing technique.

on the SiC and GaN surfaces.

Figure 12(a) presents the XPS Si2p spectrum of the mechanically polished SiC sample, **Fig. 12(b)** presents that of mechanically polished SiC sample after being immersed in Fenton's reagent, and **Fig. 12(c)** presents that of the SiC sample after our proposed polishing technique. The peak at 100.5 eV can be attributed to the Si-C bond, and the second peak at 103.2 eV can be attributed to the Si-O_2 bond [23–25]. From the results shown in **Figs. 12(a)** and **(b)**, the intensity of the Si-O_2 bond peak was increased after being immersed in Fenton's reagent. This result indicates that the SiO_2 layer was formed on the SiC surface by the OH radicals produced from Fenton's reagent. Moreover, as seen in **Fig. 12(c)**, a sharp peak is clearly observed, which corresponds to the Si-C bonding structure. From the results shown in **Figs. 3**, **4**, and **12**, we conclude that the oxidized layer on the SiC sample generated by the OH radicals was completely removed and smoothed by our proposed polishing technique.

On the other hand, **Fig. 13(a)** presents the XPS Ga3d spectrum of the mechanically polished GaN sample, **Fig. 13(b)** presents that of mechanically polished GaN sample after being immersed in Fenton's reagent, and **Fig. 13(c)** presents that of the GaN sample after our proposed polishing technique. The peak at 19.5 eV can be attributed to the Ga-N bond, and the second peak at 20.5 eV can be attributed to the Ga-O bond [26, 27]. From the results shown in **Figs. 13(a)** and **(b)**, the XPS Ga3d spectra exhibited almost the same shape, and the surface modification of GaN did not proceed much in Fenton's reagent. Moreover, compared with the results shown in **Figs. 13(a)** and **(c)**, it is clear that the intensity of the Ga-N bond peak in **Fig. 13(c)** was higher than that in **Fig. 13(a)**. We consider that this result is due to the improvement in the surface roughness and surface quality.

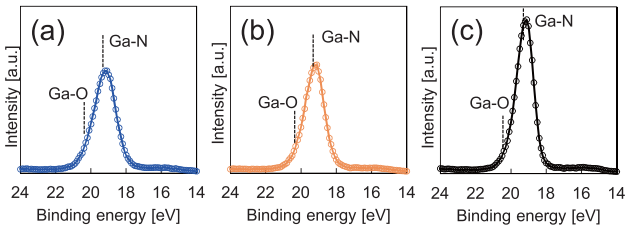

Fig. 13. (a) XPS Ga3d spectra of GaN surface, (b) mechanically polished GaN sample before and after immersion in Fenton's reagent, and (c) GaN sample polished with our proposed polishing technique.

3.4. Material Removal Rate of SiC and GaN Wafers

By measuring the weight loss due to polishing under the same conditions, the MRR of the Si-face and the C-face SiC wafers was calculated and found to be 10.7 nm/h and 130.1 nm/h, respectively. The MRR of the C-face SiC wafer is higher than that of Si-face SiC wafer. This is in agreement with the results obtained by the CMP process [7], which is considered to be due to the difference in the oxidation rates of the Si-face and C-face SiC samples. On the contrary, the MRR of the Ga-face GaN wafer was calculated to be 2.9 nm/h, an extremely low value. This is presumably because the surface modification (oxidation) by the OH radicals generated from the decomposition of H_2O_2 solution did not progress much during polishing.

Further studies will be needed to improve the MRR by determining the optimal process conditions, such as the rotating and reciprocating speeds of the magnetic tool, the pressure, and the size of the iron particles. Moreover, we would like to develop a magnetic polishing tool with a large diameter to increase the MRR by enhancing the contact area between the iron fine particles and the sample substrate during polishing.

3.5. Material Removal Mechanism

From these experimental results, we interpreted the surface removal mechanism in this polishing technique. **Fig. 14** shows the schematic drawing of the removal of surface roughness in this polishing technique. First, the OH radicals were generated by the decomposition of H_2O_2 solution near the surface of the iron particle. The OH radicals existed only near the surface of the iron particle because the lifetime of the OH radical was very short. Then, the generated OH radicals reacted with the topmost area of the workpiece surface to form an oxide layer

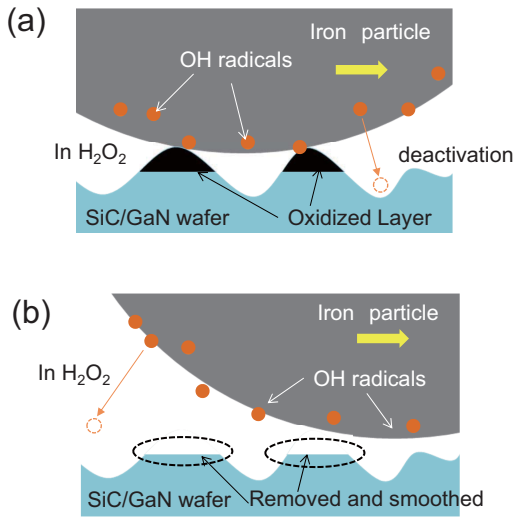

Fig. 14. Schematic drawing of the removal of surface roughness in this polishing technique.

when the iron particle came into contact with the workpiece surface (**Fig. 14(a)**). Afterwards, the oxide layer was preferentially removed, resulting in a smooth surface (**Fig. 14(b)**). In this polishing technique, spherically shaped iron particles, with the size of a few microns, were employed to smooth SiC and GaN wafers, as shown in **Fig. 2(c)**. As the waviness of the microscopic surface of the iron particles has a larger radius of curvature than that of the roughness on the workpiece surface to be removed, iron particles selectively interact with the topmost area on the workpiece surface. Therefore, the surface roughness with a spatial wavelength range on the order of a micron was removed uniformly and smoothed markedly. The removal mechanism of surface roughness is supported by the experimental results shown in **Figs. 3**, **4**, **6**, **7**, **8**, **10**, **12**, and **13**.

On the other hand, the surface waviness of the wafers was hardly improved in this polishing technique, as shown in **Figs. 5** and **9**. We speculated that the surface waviness was maintained because the magnetic cluster of the iron particles, adhering to the magnetic tool, was deformed in accordance with the surface waviness of the workpiece and was traced along the surface profile of the workpiece during polishing. From these experimental results of SiC and GaN polishing, we conclude that this polishing technique is effective for the removal of surface roughness and is not suitable for the removal of surface waviness on the SiC and GaN wafers. To obtain highly flat SiC and GaN wafers, another machining technique is needed to improve the wafer flatness and waviness prior to polishing.

4. Conclusion

In this study, we conducted polishing of 2-inch SiC and 2-inch GaN wafers with the use of a magnetic polishing tool that adsorbs iron particles in H_2O_2 solution. The experimental results show that the surface microroughness was improved markedly over the entire surface of the 2-inch SiC and 2-inch GaN wafers, and an atomically smooth and damage-free surface could be achieved after finishing. Moreover, wafer flatness and waviness did not deteriorate in this polishing technique. However, it was found that the MRR was very low. To realize larger values of MRR, it is necessary to optimize the process conditions, such as the rotating and reciprocating speeds of the polishing tools, the size of the iron particle, and the polishing tool diameter in future work.

Acknowledgements

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