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High-efficiency planarization method combining mechanical polishing and atmospheric-pressure plasma etching for hard-to-machine semiconductor substrates

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Abstract

A high-efficiency planarization technique for preprocessing before final polishing is needed for hard-to-machine wide-band-gap semiconductors, such as silicon carbide (SiC), gallium nitride, and diamond. We proposed a novel planarization method that combines chemical mechanical polishing (CMP) and atmospheric-pressure plasma etching (plasma chemical vaporization machining [P-CVM]) and developed a prototype of the basic type CMP/P-CVM combined processing system. This prototype has a mechanical polishing part for introducing a damaged layer on the convex part of the sample surface and a P-CVM part for efficient etching of the damaged layer. Process conditions for plasma generation were determined in order to minimize the optical emission intensity ratio of nitrogen to helium because nitrogen comes from circumstance air and should not exist in the plasma region. Process conditions for mechanical polishing were determined in order to efficiently generate a damaged layer only on the convex part of the sample surface. The combined process was performed using a SiC substrate on which the mesa structure was fabricated as a sample. As a result, we found that the convex parts of the mesa structure were preferentially removed and the surface of the sample was planarized. We also found that the decreasing rate of the peak-to-valley value of the mesa structure obtained by CMP/P-CVM combined processing was approximately seven times greater than that during mechanical polishing.

Key words : Hard-to-machine materials, Atmospheric-pressure plasma, plasma etching, Mechanical polishing, Damaged layer, Planarization, Silicon carbide

1. Introduction

Wide-band-gap semiconductors, such as silicon carbide (SiC), gallium nitride (GaN), and diamond, have favorable properties, including high thermal conductivity, high breakdown electric field strength, and high thermal stability and provide suitable substrates for high-temperature and high-power applications. When power transistors are fabricated on such substrates, they can be operated with lower power loss compared with conventional silicon power transistors because of a higher breakdown in the electric field (Kimoto, 2015). However, because of their hardness and chemical stability, there are few efficient machining methods, and this results in an increasing wafering cost. Although there are

some reports concerning final polishing techniques and an atomically flat surface was reported (Kato et al., 2007; Xu et al., 2003), there are few reports on a high-efficiency planarization technique for preprocessing before final polishing.

Plasma chemical vaporization machining (P-CVM) is a plasma etching technique that uses atmospheric-pressure plasma (Mori et al., 1993, 2000). In an atmospheric-pressure plasma, a high processing rate is expected because of the high density of radicals. A damage-free processed surface can also be obtained because of the very low amount of ion energy in the plasma due to the short mean free path of gas molecules (about 0.1 μm). Using P-CVM, a high removal rate of more than 1 $\mu\text{m}/\text{min}$ has been observed even for SiC (Sano et al., 2009) and GaN substrates (Nakahama et al., 2008). However, the P-CVM etching is isotropic, and there are no means to planarize the surface. Figure 1 shows a planarization model of P-CVM. Although sharp asperity is dulled by the chemical etching process, the process takes a long time and needs substantial removal for planarization because both the concave and convex parts of the work piece are removed. High-efficiency planarization could be achieved if the topmost site of the surface could be removed preferentially by P-CVM.

Thus, we propose a chemical mechanical polishing (CMP)/P-CVM combined processing method (Doi et al., 2014), as shown in Fig. 2. This method consists of two parts: mechanical polishing, where the convex part of the work surface is converted to the damaged layer; and P-CVM, where the damaged convex area is efficiently removed. The P-CVM removal rate was confirmed to be increased on damaged layers of both SiC and GaN (Sano et al., 2014, 2015), which is an essential phenomenon for the method. We developed the basic type CMP/P-CVM combined processing system (Type A) and demonstrated the principle of the method (Shiozawa et al., 2014). However, planarization data has not been presented yet. In this report, we describe the basic planarization characteristics of the method with brief introductions of the processing system and the process conditions.

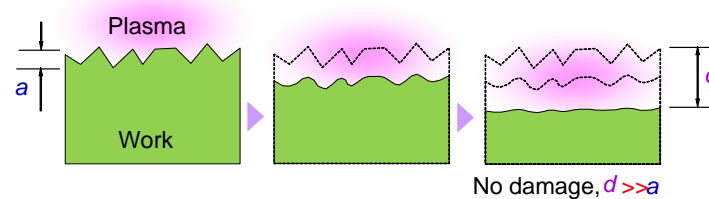


Fig. 1 Planarization model of P-CVM. Both concave and convex parts of the work piece are removed during P-CVM because of isotropic etching.

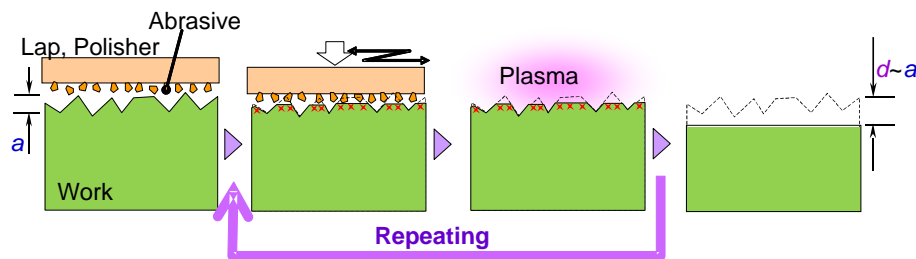


Fig. 2 Concept of CMP/P-CVM combined processing, which is the combination of mechanical polishing to introduce a damaged layer on the topmost site of a surface and atmospheric-pressure plasma etching to preferentially remove the damaged layer.

2. Basic type CMP/P-CVM combined processing system

We developed a prototype of the basic type CMP/P-CVM combined processing system; it includes a mechanical polishing part and a P-CVM part (Fig. 3), and a sample holder can move between these two parts. A sample is placed on the undersurface of the rotating sample holder and pushed onto the rotating polishing plate in the mechanical polishing part. A certain gap distance is maintained from the electrode to apply RF voltage for plasma generation in the P-CVM part. There is no device for work-surface cleaning between the mechanical polishing part and the P-CVM part. The main part exists in a ventilated enclosure to prevent active species generated in the plasma from diffusing outside.

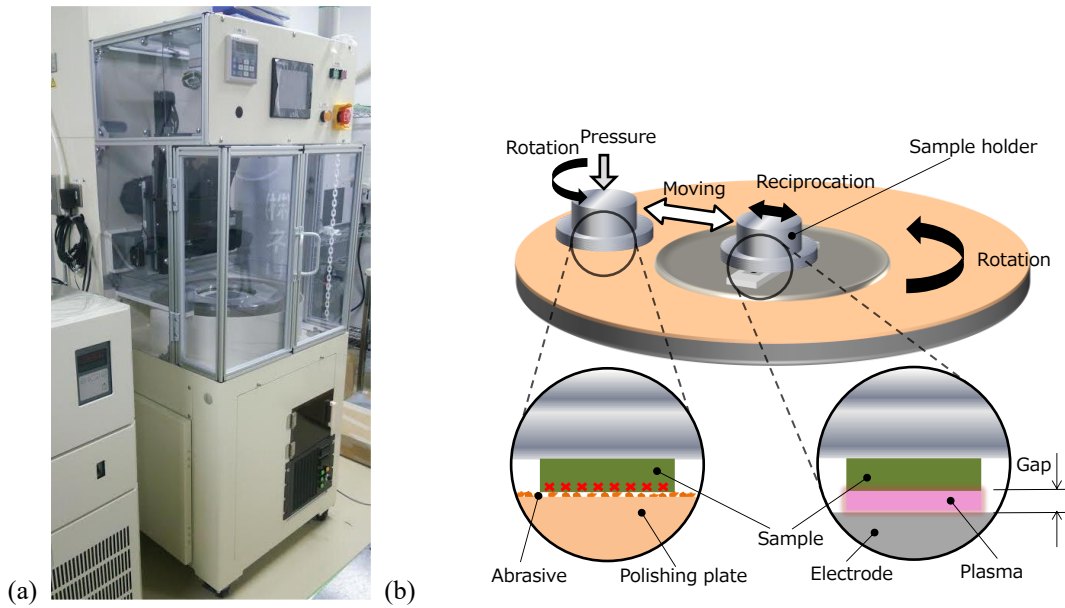


Fig. 3 (a) Photograph of the developed basic type CMP/P-CVM combined processing system, and (b) schematic drawing of the main part of the system, which has a mechanical polishing part and an atmospheric-pressure generating part.

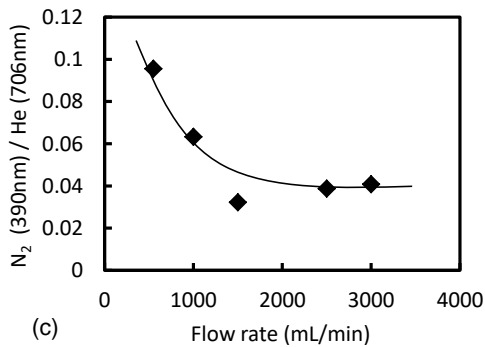
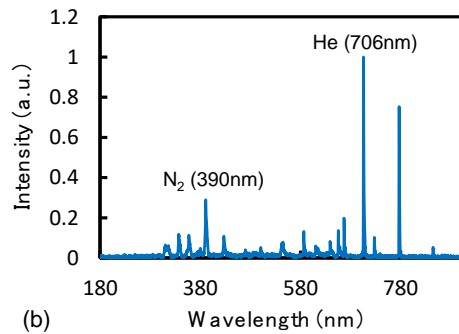
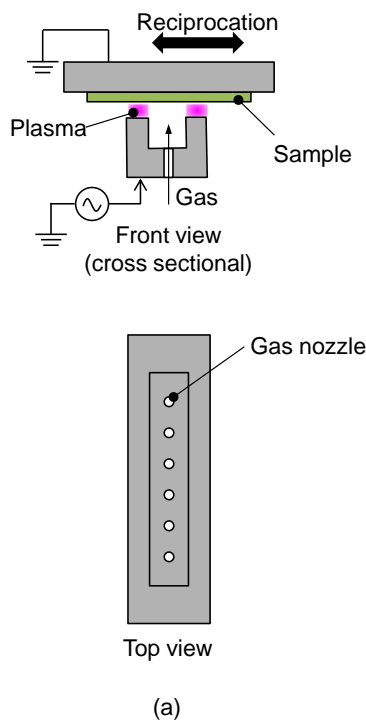


Fig. 4 (a) Schematic of the electrode, which has gas nozzles for gas supply to the plasma region; (b) an example of optical emission spectroscopy of the plasma. A nitrogen peak is originated from environmental air. The relationship between gas flow rate and the optical emission intensity ratio of nitrogen to helium is shown in (c).

3. Process conditions

3.1 Process conditions for plasma generation

Figure 4(a) shows a schematic of the electrode. It has several nozzles to supply gas to the gap between the sample and the electrode. After purging initial air from the gap by helium gas, an atmospheric-pressure plasma can be

generated by supplying an RF (13.56 MHz) power to the electrode through an impedance-matching circuit. The whole area of the sample surface can be processed by reciprocal motion of the sample holder. Figure 4(b) shows an example of the optical emission spectroscopy of the plasma. As well as a helium peak, a nitrogen peak can be observed because nitrogen exists in the air surrounding the electrode. Since it is not desirable for the air to mix with the reactive gas (mixture of He and SF₆ in the case of SiC), we considered the experimental condition such as purge time, the distance between the electrode and the sample, and the gas flow rate to reduce the optical emission intensity ratio of nitrogen to helium. Although the ratio decreased drastically with increasing purge time for a period of several seconds, it became constant after several tens of seconds. We found that the distance between the electrode and the sample did not have much influence on the ratio; however, the ratio at a distance of 300 μm had the lowest value. The experimental results of the gas flow rate are shown in Fig. 4(c). The ratio decreased with increasing flow rate up to approximately 1.5 l/min and became constant thereafter. Thus, the conditions we chose were the following: distance between the electrode and sample of 300 μm, flow rate of 1.5 l/min, and purge time of more than several tens of seconds.

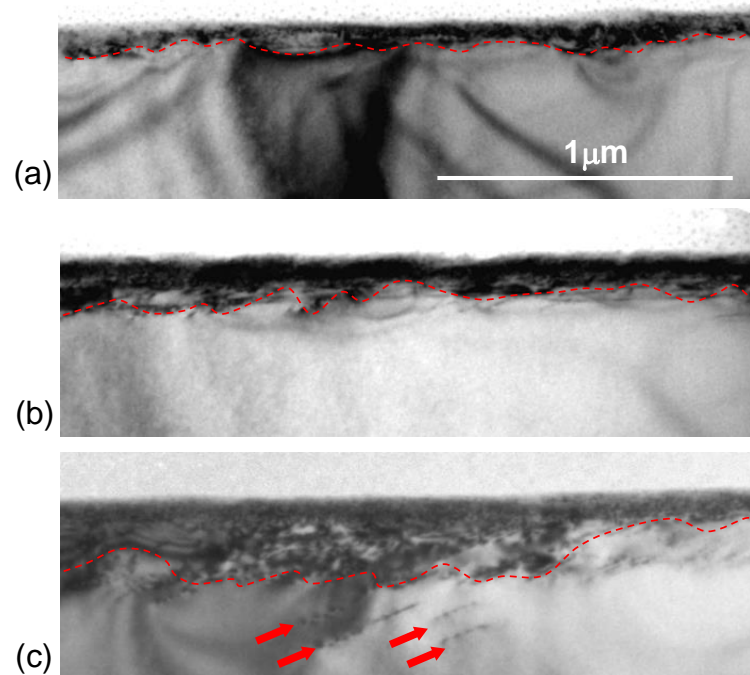


Fig. 5 Cross-sectional TEM images of the surface of mechanically polished SiC wafers under different processing pressure conditions: (a) 10 kPa, (b) 30 kPa, and (c) 90 kPa. The red dotted line roughly indicates the boundary of the damaged layer. Some belt-like black lines, especially in (a), are not dislocations but artifacts due to the thinness of the TEM sample fabricated by a focused-ion-beam method. These black lines move easily with changing TEM observation conditions.

3.2 Process conditions for mechanical polishing

In our proposed method, it is important to form the damaged layer only on the convex part of the surface of the sample. Thus, a free abrasive should not be used so that the concave part of the surface is not attacked. We decided to use a diamond film with a small fixed diamond abrasive (#8000). It is also very important to form the thicker damaged layer over a shorter time at the mechanical polishing stage, so we investigated the thickness of the damaged layer under various process conditions. The experimental detail has been described elsewhere (Sano et al., 2015). As the results, we found that the thickness of the damaged layer was independent of the polishing time (5sec–90 sec) and rotation speed of the polisher (5 rpm–30 rpm), although the removal thickness was proportional to them as indicated by Preston's equation (Preston, 1927). We opted for a polishing time of 5 sec and a rotation speed of 5 rpm because a shorter time is better and a slower rotation speed is more effective in preventing edge chipping of the sample. The thickness of the damaged layer was found to increase with increasing processing pressure, as shown in Fig. 5, indicating that a larger

processing pressure was better for our method; however, as shown in Fig. 5(c), some defects (indicated by red arrows) occurred in the deeper area away from the boundary of the damaged layer. This needs to be avoided because it means that the thickness which should be removed by subsequent final CMP process increases and process time of CMP increases. Thus, we opted for a processing pressure of 30 kPa.

4. Basic machining characteristics

4.1 Sample preparation

Because P-CVM etches both the convex and the concave part of the sample surface, information on the decreasing ability of the peak-to-valley (PV) value of the surface concavo-convex shape is necessary. We therefore evaluated a sample that had a mesa structure on the surface, as shown in Fig. 6. A thin aluminum film was first deposited on a 4H-SiC (0001) substrate (10 mm × 10 mm) by vacuum vapor deposition with a shadow mask. The substrate was then placed in atmospheric-pressure plasma with a He and SF₆ mixture. Only the part not covered with the aluminum mask was etched without any crystallographic damage, and a sample with the mesa structure was fabricated after removing the aluminum masks by acid cleaning. Figure 7(a) shows one of the samples, and Fig. 7(b) and (c) shows an example of a surface shape measured by interferometric microscopy (Zygo, NewView 200) and its A–A' cross-sectional profile, respectively. The micrometer-order mesa structure is shown to have been successfully fabricated.

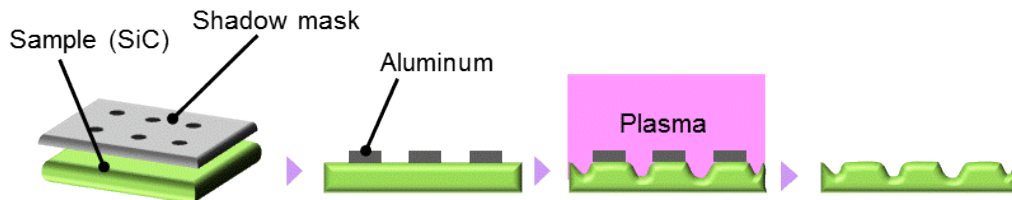


Fig. 6 Procedure for making a sample with a mesa structure. Fabrication is through deposition of a thin aluminum film by vacuum vapor deposition with a shadow mask, atmospheric-pressure plasma etching to etch undeeposited parts of the sample, and acid cleaning to remove the aluminum mask.

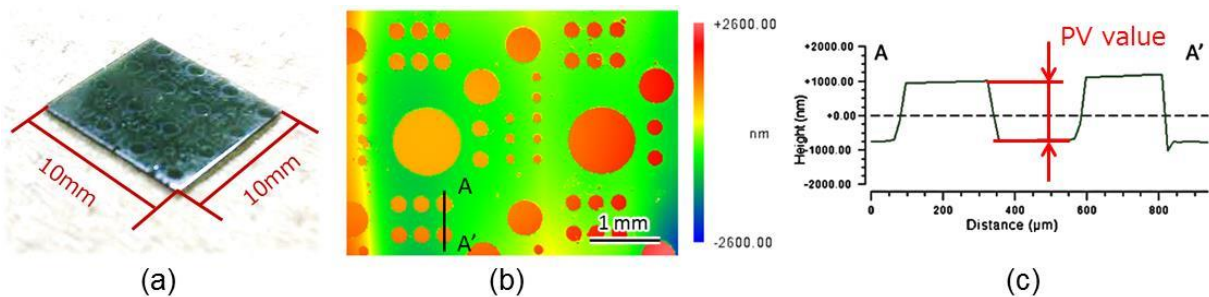


Fig. 7 (a) Photograph of the sample with a mesa structure; (b) surface shape of the sample measured by interferometric microscopy; (c) cross-sectional profile of A–A' of (b).

4.2 Planarization characteristics

CMP/P-CVM combined processing was performed using the above sample under the conditions shown in Table 1. P-CVM followed by mechanical polishing was repeated 320 times, and surface profiles were measured after 40, 100, 200, and 320 repetitions (Fig. 8). We found the convex parts of the mesa structure to be preferentially removed and that the surface of the sample was being planarized with increasing number of repetitions. Line profiles of the approximate center parts of each surface profile are shown in Fig. 9, and Fig. 10 shows the relationship between the PV value of the mesa structure in the central area and repetition times. The PV value, which was initially approximately 6 μm, decreased with increasing repetition and reached less than 0.5 μm after 320 repetitions. This improved rate of the PV value decreased from 100 repetitions to 200 repetitions because of the decrease in contact pressure due to the increase in contact area associated with planarization. Thus, we increased the work-holder pressure to 60 kPa, which resulted in a large improvement to the decreasing rate of the PV value between 200 and 320 repetitions.

Table 1 Experimental conditions of CMP/P-CVM combined processing

| Target sample | 4H-SiC (0001) 2° off substrate with a mesa structure | |
|-------------------------------------|--|--|
| Mechanical polishing | Abrasive | Diamond film (#8000) |
| | Processing pressure | 30 kPa (repetitions of 1–200) 60 kPa (repetitions of 201–320) |
| | Rotation speed (polishing plate and sample holder) | 5 rpm |
| | Process time | 5 sec |
| Atmospheric-pressure plasma etching | Reactive gas | He:SF ₆ = 99.4:0.6 |
| | Gas flow rate | 1.5 l/min |
| | Gap (electrode–specimen) | 300 μm |
| | RF (13.56 MHz) power | 50 W |
| | Process time | 15 sec |

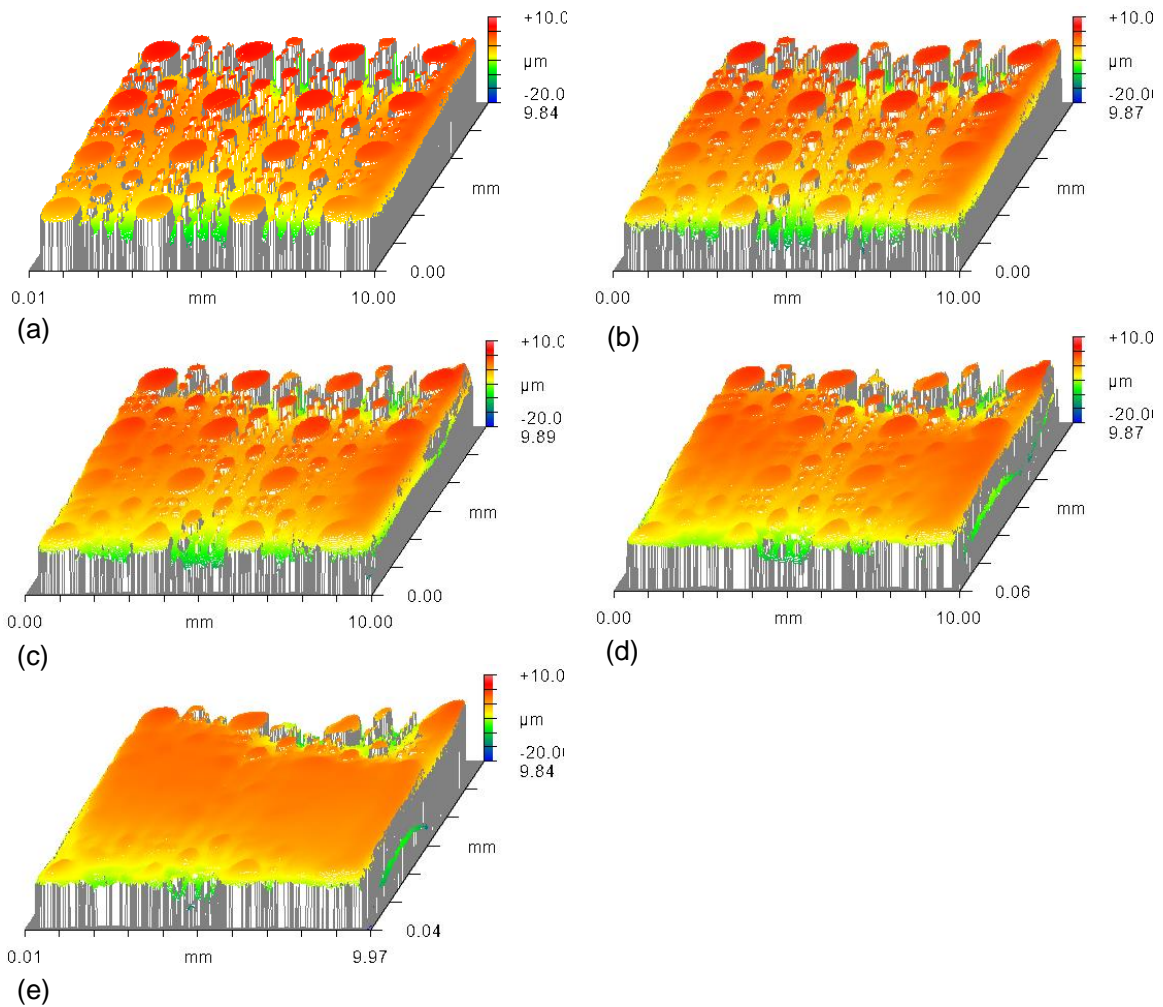


Fig. 8 Surface profiles of the sample (a) before processing and after (b) 40 repetitions, (c) 100 repetitions, (d) 200 repetitions, and (e) 320 repetitions of a set of mechanical polishing and P-CVM.

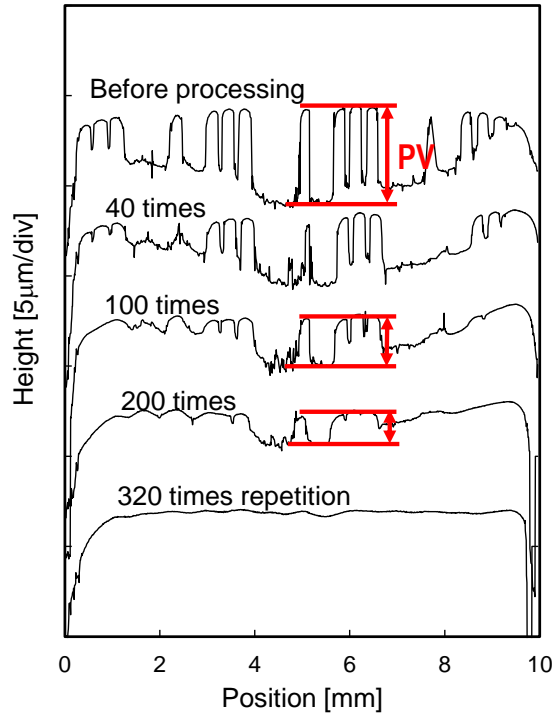


Fig. 9 Transition of the line profile from before processing to after 40, 100, 200, and 320 repetitions. Each profile has been shifted vertically for a clearer view.

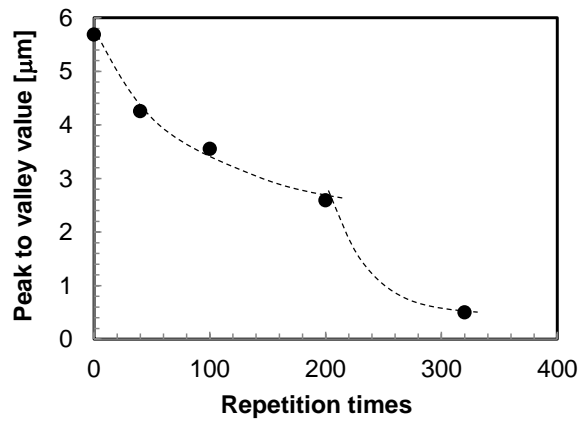


Fig. 10 Relationship between the peak-to-valley value of the surface profile and repetition times.

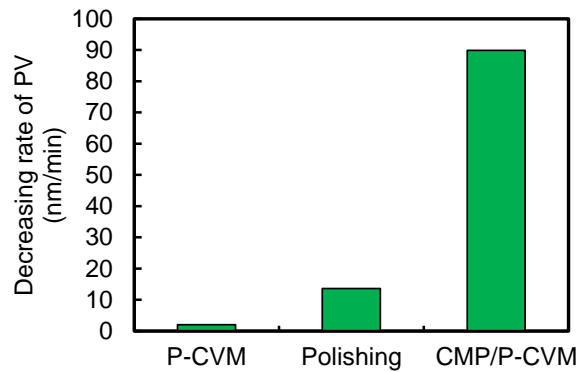


Fig. 11 Comparison of the decreasing rate of the PV value of the mesa structure using P-CVM, CMP (mechanical polishing), and the CMP/P-CVM combined process.

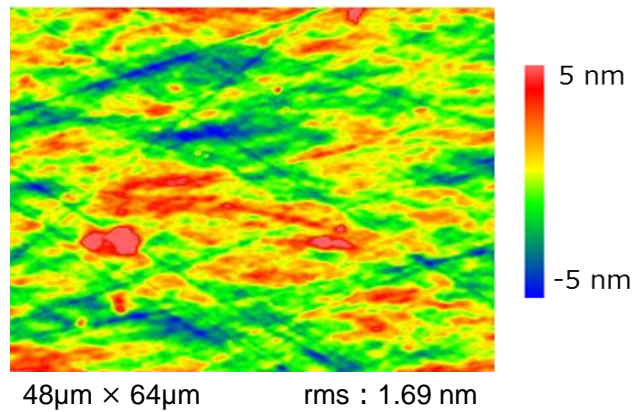


Fig. 12 Typical surface roughness after the CMP/P-CVM combined process measured by a microscopic interferometer. Many scratch-like grooves formed by selective etching of the damaged area around the scratch generated in mechanical polishing can be observed.

4.3 Comparison of the decreasing rate of the PV value

The decreasing rates of the PV value of the mesa structure under the three processes of P-CVM alone, CMP (mechanical polishing) alone, and CMP/P-CVM are shown in Fig. 11. The experimental conditions of CMP under the process of CMP alone and that of P-CVM under the process of P-CVM alone are the same as those under the process of CMP/P-CVM combined process (shown in Table I). The rate of decrease of the PV value obtained by CMP/P-CVM combined processing was approximately seven times higher than that during mechanical polishing.

4.4 Surface roughness

Figure 12 shows a typical surface roughness, measured by microscopic interferometer, after the CMP/P-CVM combined process. Many scratch-like grooves can be seen on the surface. Scratches are generated by mechanical action in the mechanical polishing process before P-CVM; these scratches accompany other wider and deeper crystallographic damaged areas but can be removed preferentially by P-CVM. For this reason, such scratch-like grooves appear after the subsequent P-CVM process. Importantly, these shapes are not accompanied by crystallographic damage, which should be removed by a finishing process, such as CMP. Thus, we assume they have little influence on the load of the finishing process.

5. Conclusion

By combining atmospheric-pressure plasma etching and mechanical polishing, we demonstrated that plasma etching can be used as a planarization technique. Using this technique, we also demonstrated that the decreasing rate in the step height was seven times greater than the value obtained from mechanical polishing alone. This technique will be useful for undamaged and high-efficiency planarization of hard-to-machine semiconductor substrates.

Acknowledgments

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