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# Dependence of GaN Removal Rate of Plasma Chemical Vaporization Machining on Mechanically Introduced Damage

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A high-efficiency planarization method combining atmospheric-pressure plasma etching [plasma chemical vaporization machining (PCVM)] and mechanical polishing is proposed. The convex part of a substrate surface, considered to be affected by mechanical action, is removed preferentially by PCVM. However, it is not evident whether the PCVM removal rate of the damaged layer of a gallium nitride (GaN) substrate increases. In this study, the dependence of removal rate on removal depth is investigated using a GaN substrate with a damaged layer. As a result, the removal rate of the damaged layer is observed to be three or four times greater than that of deep undamaged layers.

## 1. Introduction

Gallium nitride (GaN) wafers are widely used as substrates for blue or white light-emitting diodes (LEDs) and ultraviolet (UV) sensors. Although a heteroepitaxial GaN

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substrate grown on silicon, sapphire, or silicon carbide wafers is commonly used, there are several defects due to a large lattice mismatch between GaN and these materials. To reduce defect density and improve crystal quality for use in high-efficiency LEDs or power devices, a homoepitaxial GaN substrate grown on a bulk GaN substrate is highly required. The bulk GaN substrate is generally produced by mechanical processes such as slicing, lapping, and polishing after crystal growth. However, because of the extreme hardness and brittle properties of the GaN material, mechanical processing takes a long time, increasing the wafer cost. Thus, in addition to a high-quality and high-speed crystal growth method, a high-efficiency processing method is required.

Plasma chemical vaporization machining (PCVM),<sup>(1-3)</sup> a plasma etching process using atmospheric-pressure plasma, is a candidate method that enables high-efficiency processing of GaN. In this process, a high radical density due to a high pressure enables a high removal rate and a small ion energy owing to a short mean free path, thus producing a damage-free processed surface. Nakahama *et al.* reported the machining properties of GaN by PCVM, obtaining a removal rate of more than 5  $\mu\text{m}/\text{min}$ .<sup>(4)</sup> However, the etching phenomenon of PCVM is isotropic and there is no means to planarize the surface of the workpiece, that is, both the concave and convex parts of the workpiece are removed by PCVM. Thus, we propose a novel processing method that combines mechanical polishing and PCVM.

The concept of the method is shown in Fig. 1. Initially, a damaged layer is introduced to the convex part of the sample by the mechanical action of the polishing plate. Next, the damaged layer is selectively etched and removed by chemical reaction in the plasma. By repeating these two processes, the surface of the sample can be planarized if the etching rate of the damaged layer is larger than that of the undamaged area. It has been reported that the removal rate of the damaged layer is extremely low in the photoelectrochemical etching of a GaN substrate<sup>(5,6)</sup> because of the recombination of the electron and hole generated by UV light irradiation through the defect level.<sup>(7)</sup> Since the surface of the sample during the plasma etching process is irradiated by UV light emitted from the plasma itself, it is possible that the etching rate of the damaged layer does not increase if the UV-generated electron and hole play an important role in the etching process. Thus, the purpose of this study is to investigate whether the PCVM etching rate of the damaged layer of a GaN substrate increases, using a preliminarily damage-introduced GaN substrate as the sample.

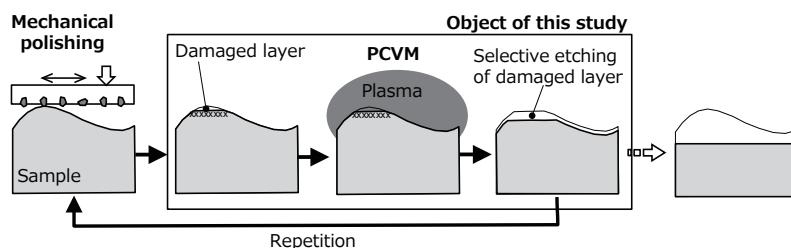


Fig. 1. Concept of a novel processing method, which is a combination of mechanical polishing and PCVM, and object of this study.

## 2. Experimental Apparatus

In PCVM, the vapor pressure of the reaction product should be large. The melting and boiling points of  $\text{GaF}_3$  are approximately 800 and 1000 °C, respectively, whereas, those of  $\text{GaCl}_3$  are 78 and 201 °C, respectively. Thus, we decided to use  $\text{Cl}_2$  balanced with helium as the gas for plasma generation. Since  $\text{Cl}_2$  is known as a corrosion gas, a simple apparatus was developed specifically for a basic PCVM experiment. To reduce the effects of corrosion and chamber volume, the surface area that comes in contact with the  $\text{Cl}_2$  gas should be small.

Figure 2 shows a schematic of the apparatus; the apparatus mainly consists of three parts: the electrode, the body, and the quartz spacer, which is located between the electrode and the body, acting as a barrier wall. Furthermore, the quartz spacer functions as a window for observing the plasma. A micrometer, which functions via elastic deformation of the diaphragm, is used to adjust the gap between the electrode and the sample surface.

Figure 3 shows a schematic of the experimental system. A pump, whose part in contact with the gas is made of polytetrafluoroethylene, was used for gas replacement. The exhaust port of the pump was connected to the exhaust gas treatment equipment by which the redundant  $\text{Cl}_2$  and other active reaction products were trapped and removed. A buffer tank allowed for flexible gas conditions with regard to concentration and flow rate. The electrode was covered by a mesh shield and connected to the 13.56 MHz power supply through an impedance matching circuit.

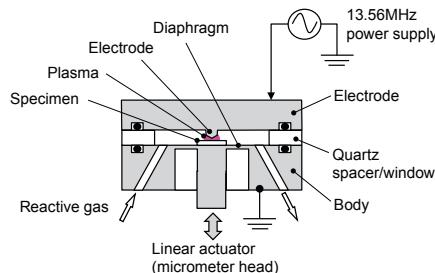


Fig. 2. (Color online) Schematic of a simple apparatus for a basic PCVM experiment.

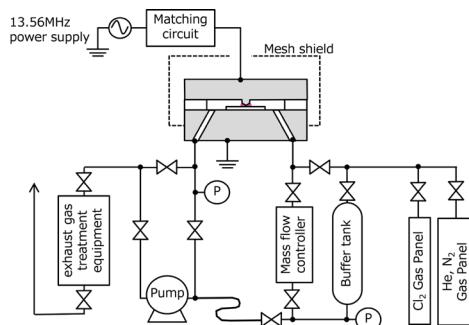


Fig. 3. (Color online) Schematic of PCVM experimental system.

### 3. Experimental Results

A commercially available free-standing GaN (0001) substrate was used as the specimen. The size was approximately  $10 \times 10 \text{ mm}^2$ , and the surface was finished by chemical mechanical polishing. To observe the thickness of the damaged layer of the specimen, a focused ion beam and a cross-sectional transmission electron microscope (TEM) were used. Figure 4 shows the obtained TEM image of the surface; the thickness of the damaged layer was observed to be approximately 50–100 nm.

After setting the specimen, the electrode and the quartz spacer were fixed to the body by four screws. The gap between the electrode and the specimen was set to approximately 300 nm. After evacuation, the reactive gas (0.3%  $\text{Cl}_2$  balanced with He) was allowed to flow at a rate of 300 sccm. The chamber was maintained at atmospheric pressure, and 80 W of 13.56 MHz power was supplied for 1 min to the electrode to generate the plasma. The specimen was removed after another evacuation and  $\text{N}_2$  purge, and a surface profile was subsequently measured using an interferometer microscope (Zygo, NewView200). One-minute processing and measuring were repeated until a total removal thickness of 200 nm, which includes both the damaged layer (50–100 nm) and the undamaged area, was obtained.

Figure 5 shows measured surface profiles for every 1 min of processing. A groove was generated by PCVM, which increased in depth with processing time. It is worth noting that a scratchlike shape was observed in the processed area, indicating selective etching of the damaged area. As shown in Fig. 5, it was difficult to precisely specify the removal depth because of surface roughness. Thus, the removal depth was calculated by subtracting the mean height of the deepest line (line A in Fig. 6) from that of the initial surface (line B in Fig. 6). By this method, the removal depth at each processing time was calculated and is plotted in Fig. 7.

The relationship between removal depth and processing time was almost linear, at times greater than 3 min; however, this relationship was not observed at 1 and 2 min. The removal rate for each processing time was calculated and is plotted in Fig. 8 as a function of etching depth. Although the removal rate was almost constant for etching depths greater than 100 nm, it was several fold larger at depths less than 100 nm. Since the thickness of the damaged layer was observed to be approximately 50–100 nm by TEM, it can be considered that the constant removal rate observed at depths greater than 100 nm is that of undamaged GaN, and the increased removal rate observed at

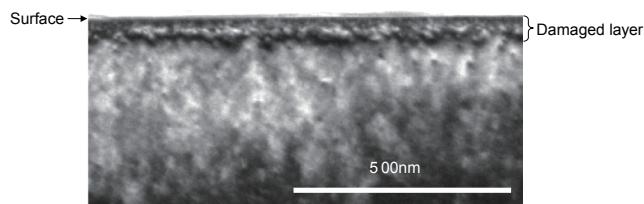


Fig. 4. Cross-sectional TEM image of the surface of a GaN specimen before PCVM processing.

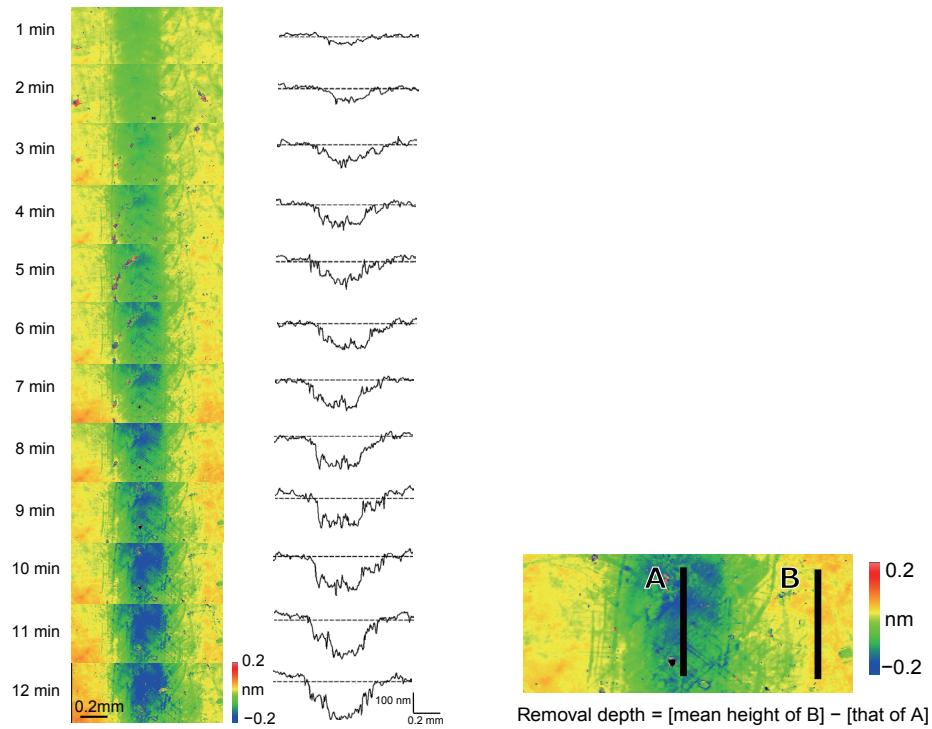


Fig. 5 (left). (Color online) Surface profiles after every 1 min of PCVM processing.

Fig. 6 (right). (Color online) Method for calculating removal depth.

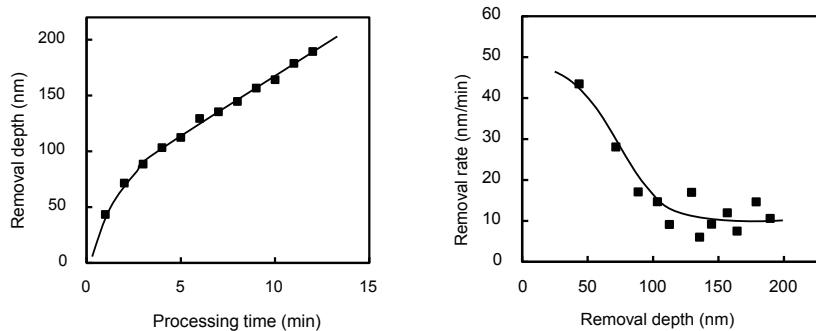


Fig. 7 (left). Relationship between removal depth and PCVM processing time.

Fig. 8 (right). Relationship between removal rate and removal depth.

depths less than 100 nm is caused by the existence of the damage. The removal rate in the damaged layer is considered to increase because of the weakened Ga-N bond or the existence of a dangling bond.

#### 4. Conclusions

It is clearly determined that the PCVM removal rate of the damaged layer of a GaN substrate is higher than that of the undamaged layer. Compared with the low photo-electrochemical etching rate result for damaged layers, it is considered that the electron and hole generated by emitted UV do not play an important role in PCVM. In the near future, high-efficiency planarization of GaN substrates will be realized by combining PCVM with mechanical polishing that introduces the damaged layer only on a convex part of a GaN substrate.

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