Plasma-Etching Enhanced Mechanical Polishing for CVD Diamond Films

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Abstract Chemically vapor deposited diamond films were etched at different parameters using oxygen plasma produced by a DC (direct current) glow discharge and then polished by a modified mechanical polishing device. Scanning electron microscope, atomic force microscope and Raman spectrometer were used to evaluate the surface states of diamond films before and after polishing. It was found that a moderate plasma etching would produce a lot of etch pits and amorphous carbon on the top surface of diamond film. As a result, the quality and the efficiency of mechanical polishing have been enhanced remarkably.

Keywords: diamond film, plasma etching, polishing, oxygen plasma

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1 Introduction

CVD (chemical vapor deposition) diamond film has many excellent properties and numerous potential applications in machinery, microelectronics, optics and defense industry [1]. The desired surface roughness of CVD diamond film varies depending on the different applications in industry. For the coats of tools and drills, in order to lower the roughness of processed machines, the average surface roughness of diamond films must be less than several hundreds of nanometers. When used as a thermal sink for integrate circuits (ICs) and high power lasers, it must be less than one hundred of nanometers to reduce the thermal resistance in thermal management. For optical window applications such as X-ray, and ultraviolet ray (UV) to infrared ray (IR) windows, an average surface roughness less than tens of nanometers can improve the optical transmission of diamond by reducing the scattering of incident light.

However, industrial synthetic CVD diamond films are usually polycrystalline films which have randomly oriented crystals and non-uniform grain sizes. These result in very rough surfaces of CVD diamond films. Usually the surface roughness increases rapidly with the film thickness and the mean surface roughness ranges from less than one to several tens of microns [2,3]. Conventional mechanical polishing is widely used in the industrial application of CVD diamond films. However the polishing rate is too low (10 nm/h) [4] and it needs different sizes of abrasive powders for fine polishing. Thus it is not suitable to polish thick films with R_a (the average surface roughness) beyond several microns [4,5]. Chemically-assisted mechanical polishing (CAMP) technique has already been developed, which can yield improved lapping rates against the conventional mechanical polishing [4]. The reactive ion etching of diamond film has been investigated to further enhance the polishing efficiency of CAMP [6].

In this paper a plasma-etching enhanced mechanical polishing technique is developed. It is a potential commercialized technique for polishing CVD diamond films because of high polishing efficiency and quality.

2 Experimental details

Polycrystalline diamond film was deposited on the tungsten substrate using a 5 kW microwave plasma CVD system. The deposition parameters are listed in Table 1. Several square samples were taken from the diamond film. To investigate the influence of plasma etching on the mechanical polishing, one of the samples was not etched by oxygen plasma and the rest were etched at different parameters in oxygen plasma produced by a DC (direct current) glow discharge. Then all the

Table 1. Deposition parameters and surface roughness of deposited CVD diamond film

Microwave Power (kW)	3.2
Gas resources	$H_2:CH_4=100:2$
Gas pressure (kPa)	13.6
Deposition temperature (K)	1000
Deposition time (h)	72
Thickness (μm)	200
Surface roughness (μm)	$R_a=3.09$, $R_t=16.69$

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Table 2. Parameters of plasma etching and mechanical polishing

Sample	Etching parameters			Polishing parameters		
	Pressure (Pa)	DC Power (V×mA)	Time (min)	Pressure (kg/cm^2)	Rotation speed (r/min)	Time (min)
A	—	—	—	6	500	120
B	100	915×110	15	6	500	60
C	100	780×60	45	6	500	60

Table 3. Repeated plasma etching enhanced mechanical polishing for sample C

Step	Etching parameters			Polishing parameters		
	Pressure (Pa)	DC Power (V×mA)	Time (min)	Pressure (kg/cm^2)	Rotation speed (r/min)	Time (min)
1	100	780×60	45	6	500	60
2	100	780×60	10	6	500	60
3	100	780×60	2	6	500	60

diamond films were polished by a modified mechanical polishing device with diamond powders as abrasive. The parameters of both plasma etching and mechanical polishing are given in Table 2. The mechanical polishing assisted by a repeated plasma etching technology was adopted to substantially improve the polishing quality of sample 3 and the related parameters are given in Table 3. The schematic diagram of DC glow oxygen plasma apparatus is shown in Fig. 1. The discharge chamber consists of a glass tube, a ring anode and a disc cathode.

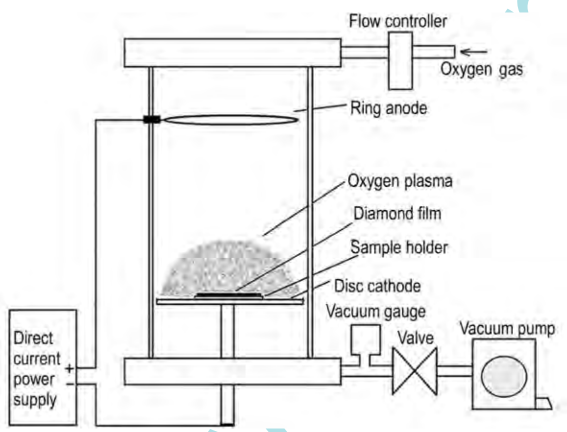


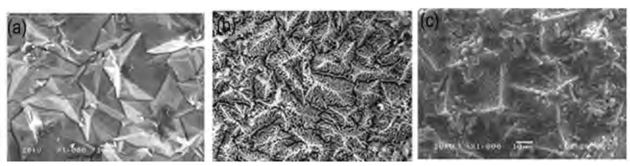
Fig.1 Schematic diagram of DC glow oxygen plasma device

JSM-5510LV scanning electron microscope (SEM) and Renishaw System RM-1000 micro-Raman spectrometer (Ar^+ laser, 514.5 nm) were used to observe the changes of surface morphology and structure of the samples, respectively. Benyuan CSPM-4000 atomic force microscope (AFM) was used to detect the surface roughness of polished films. Eight points were measured on each sample for getting R_a and the maximum peak-valley height roughness (R_t) after polishing.

3 Results and discussion

3.1 Plasma-etching enhanced mechanical polishing

Fig. 2 shows the SEM images of the surface of non-etched samples and those etched at different parameters. Fig. 2(a) shows the surface morphology of sample A not etched by oxygen plasma. The surface is very rough and consists of a large number of pyramidal grains with well-defined crystalline shape and sharp edges. Fig. 2(b) shows the etched surface morphology of sample B etched at a higher DC power of 915 V×110 mA. It is found that numerous very deep pits appear within the surfaces and the boundaries of the crystals. The appearance of the deep pits can be attributed to the higher etching rate of dislocations with positive oxygen ions accelerated by the sheath voltage drop between the plasma and the diamond film [7]. By lowering the DC power to 780 V×60 mA, the intensity of plasma etching decreased and the surface was weakly etched. As seen in Fig. 2(c), the deepness of the etch pits of the sample C becomes smaller than that of sample B shown in Fig. 2(b) and the pits mainly appear on the crystal surfaces. This result can be ascribed to the weakening of oxygen ion etching with the decrease of plasma power. Lowering the DC power would reduce

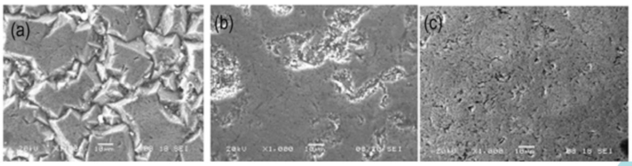


(a) Sample A without plasma etching; (b) Sample B etched at 100 Pa and 915 V×110 mA for 15 min; (c) Sample C etched at 100 Pa and 780 V×60 mA for 45 min

Fig.2 Surface morphology of samples

both the electron temperature and the sheath voltage drop between the plasma and the diamond film. As a result, the etching of oxygen ion was correspondingly weakened.

Fig. 3 shows the SEM images of the mechanically polished samples without and after plasma etching. The polished surface morphology of sample A without plasma etching is shown in Fig. 3(a). It is found that only the tops of diamond grains were smoothed after mechanical polishing for 120 min. Fig. 3(b) shows the surface morphology of sample B which was etched by plasma with higher DC power and then mechanically polished for 60 min. Compared to that of sample A, the protuberant crystal grains are entirely removed after mechanical polishing for 60 min, which results in a relative smooth surface. At the same time, deeper etch pits around the crystal boundary result in some large area pits on the polished surface. Fig. 3(c) shows the mechanical polishing result of sample C, which is etched by lower power DC plasma. It is found that confining the etch pits mainly to the crystal surface can not only result in a fast removal of the protuberant crystal grains, but also lead to a better polished surface of sample C than that of sample B.



(a) Sample A with mechanical polishing for 120 min; (b) Sample B with mechanical polishing for 60 min; (c) Sample C with mechanical polishing for 60 min

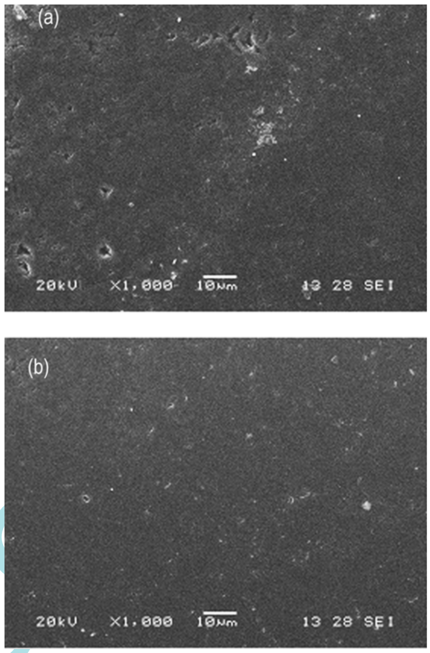
Fig.3 Polished surface morphology of samples

Table 4 lists the surface roughness results of the polished samples. It is revealed that polished sample C has the best surface polishing among the samples A ~ C. This result is consistent with the SEM observation and shows that a moderate plasma etching can enhance the efficiency of mechanical polishing remarkably.

Table 4. Surface roughness of polished samples

Sample	R_a(nm)	R_t(nm)
A	1532	7860
B	350	1547
C	30	266

To further improve the polishing quality, a polishing technique, in which the mechanical polishing was enhanced by repeated plasma etching, was developed. Fig. 4 is the SEM result of sample C after repetitious plasma etching enhanced mechanical polishing. Fig. 4(a) and (b) are the SEM images of sample C after the second and the third treatment, respectively. As the protuberant areas were faster removed by plasma etching enhanced mechanical polishing, the pits of grain boundaries decreased and thus a smoother surface topography was obtained. Fig. 5 is the 2D view of AFM result of polished sample C after the third treatment. According to the analysis of image management software, R_a of the polished sample is 4.25 nm and R_t is 50.7 nm.



(a) Etched for 10 min and polished for 60 min; (b) Etched for 2 min and polished for 60 min

Fig.4 SEM images of polished diamond sample C during mechanical polishing enhanced by repeating plasma etching

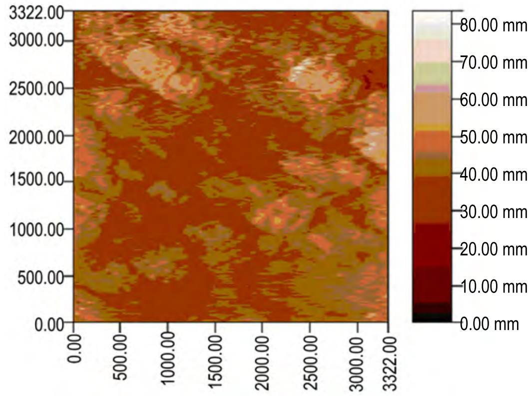
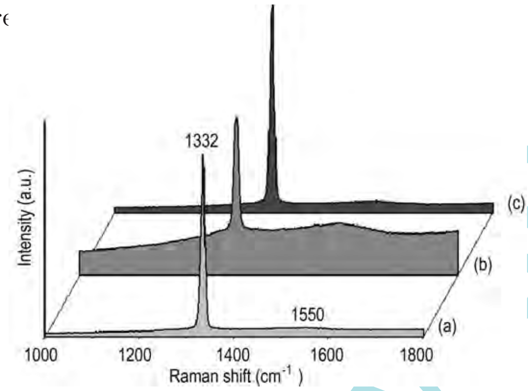


Fig.5 AFM result of sample C after the third round of mechanical polishing enhanced by repeating plasma etching

3.2 Enhancing mechanism of plasma etching

In order to investigate the enhancing mechanism of plasma etching, we analyzed the difference of surface morphology and Raman spectra between the original and etched diamond grains. It can be found in Fig. 2(a) that the original diamond grains have compact grain surfaces and sharp edges which are the encumbrances of mechanical polishing. Plasma etching can lead to the emergence of numerous etch pits on the diamond grain surface which can be removed more easily as shown in Fig. 2(b) and (c). In the process of mechanical polishing, the protruding portions that come into contact with the diamond powder will cause micro-cracking and then are removed by the process of brittle fracture on a microscopic scale called micro-chipping [8,9]. On the beam plasma etching can bring about numerous etch pits which may accelerate the growth of micro-cracks and thus results in faster micro-chipping between the diamond powder and the diamond film.

Fig. 6 is the Raman spectra of as-deposited diamond film, etched diamond film and diamond film after etching enhanced polishing, respectively. From Figs. 6(a) and (b) it is found that the intensity of the band centered at 1550 cm^{-1}, corresponding to the sp^2-bonded amorphous carbon, increases with the oxygen plasma etching. A combination of this result and the SEM observation shows that plasma etching can not only produce numerous etch pits but also result in amorphous carbon on the diamond film surface. The weak sp^2-bond of amorphous carbon can accelerate the growth of micro cracks and thus result in faster micro-chipping. After polishing for one hour, the obvious broad band centered at 1550 cm^{-1} becomes very weak as shown in Fig. 6(c). This result indicates that the amorphous carbon produced in the plasma etching can be easily removed during the mechanical polishing without the decre



(a) As-deposited diamond film; (b) Etched at 100 Pa and 780 V×60 mA for 45 min; (c) Etched and mechanically polished for 60 min

Fig.6 Raman spectra of diamond films

4 Conclusion

Moderate plasma etching of CVD diamond film can enhance the polishing efficiency of mechanical polishing. Through mechanical polishing enhanced by repeated oxygen plasma etching, the diamond film can be polished from that of an average surface roughness of 3000 nm to that of 5 nm within 5 hours. This preliminary result reveals a great potential for the commercialization of the technique.

The mechanism of plasma etching enhanced mechanical polishing is expected as follows.

a. Plasma etching can eliminate the polishing encumbrances by transforming the compact grains with sharp edges into the grains with thin surfaces and obtuse edges.

b. Plasma etching can produce a lot of etch pits and amorphous carbon which can accelerate the growth of micro-cracks and thus result in faster micro-chipping of the top surface of diamond film.

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