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An Investigation on Two Types of Crystalline Micro-diamond Film Coated Tools Lapping with Sapphire Wafer

F. Wei [a,b], L. Wenzhuang*[a], Z. Dunwen[a]

[a] *College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, Nanjing 210016, China*
[b] *College of Mechanical Engineering, Yancheng Institute of Technology, Yancheng 224051,China*

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ABSTRACT

Two types of micron-diamond films were prepared on YG6 substrate by hot filament chemical vapor deposition (HFCVD) method. Morphology and orientation of crystalline growth were evaluated by Scanning Electron Microscopy (SEM) and X-ray Diffration (XRD). The surface of the diamond film-coated tools and sapphire wafer was compared before and after lapping . The Raman spectra of two micron-diamond film indicated that a significant change occurred in surface properties after lapping. This change was explained as graphitization of the diamond surface. The friction coefficient of the ZMDF was larger than that of the FMDF tool. The surface roughness of the sapphire wafer lapped by FMDF (<110> texture) was lower than that of ZMDF (<111> texture). Comparing the two kinds of micron-diamond film, the lapping effect of the FMCD tool was found to be better.

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1. INTRODUCTION

Sapphire is an important ceramic material used widely in a range of applications such as optics, electronics and temperature sensing [1]. Another important use of sapphire is for visible and mid-wave infrared airborne windows and radomes [2]. However, further use of sapphire is limited due to its low efficiency of machining and high cost [3]. The hardness of sapphire measures nine on the Moh scale, comparable to that of diamond. Therefore, it is necessary to find new methods of lapping sapphire that are efficient and low cost.

Currently sapphire is processed using mechanical polishing or chemical-mechanical polishing. These are abrasive methods of polishing form and form scratchs on the surface of sapphire. A new method of magnetorheological polishing can produce satisfactory results. To be able to achieve large area coating met thin film form was expected to be most effective and inexpensive way [4]. Diamond films fabricated by chemical vapor deposition (CVD) have been investigated extensively as a protecting coating material for many mechanical components due to their extremely high hardness and wear resistance [5].

This article is focused on preparing <111> and <110> textured micron-diamond film coated tools. Two kinds of diamond film coated tools were investigated for the first time in lapping with sapphire wafer. Resulting changes in the diamond film and sapphire wafer were discussed to provide a mechanistic theory for subsequent enhancement of the lapping removal rate and surface quality. These results prove a basis for a new method for the precision machining of sapphire.

2. EXPERIMENT

2. 1. Preparation of Two Types of Crystalline Diamond Films

The substrate sample was cemented carbide, of which 6% is Co and 94% is WC. The size was 3×6×1.5 mm. The samples were pre-

* Corresponding Author's Email: meewzlu@nuaa.edu.cn (L. Wenzhuang)

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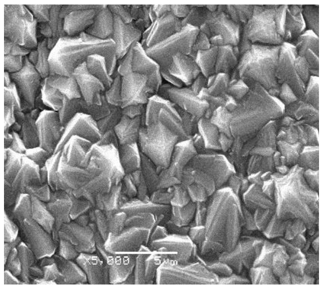
treated before deposition in Murakami's reagent for 20 min to obtain high nucleation density [6]. The surface Co was then removed by etching in 10 ml H_2SO_4+ 30 ml HCl for 1 min. Then the pretreated samples were put into a laboratory-made CVD reactor. The gas phase in CVD reactor was a mixture of CH_4 and H_2, activated by five tungsten filaments (0.4mm diameter). The deposition time was 5h. Two CVD conditions were investigated here. For parameter one, the pressure of the gas mixture in the actor was 3.3 kPa, the carbon-intensity was 1.5% and the substrate temperature was 780℃. For parameter two, the pressure of the gas mixture in the actor was 2.5 kPa , the carbon-intensity was1% and the substrate temperature was 810℃. The diamond films prepared under condition 1 and 2 will hereinafter be referred to as ZMDF (<111> texture) and FMDF (<110> texture), respectively.

2. 2. Lapping Experiment The CVD prepared diamond coated tools were used for the first time in lapping sapphire at room temperature. The active member in the lapping process was the sample coated by micron-diamond films. The passive member in the lapping process was an unfinished single crystal sapphire wafer (2 inches in diameter, 5 mm of thickness). Eccentricity was 10 mm and the experiment time was 60 min. The rotation speed was 150 r/min.

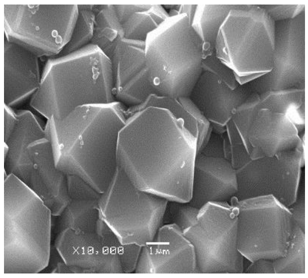
3. RESULTS AND DISCUSSIONS

3. 1. Preparation and Characterization of Diamond Coated Lapping Tools Figure 1 shows the SEM image of the surface of the two diamond coated tools, which were prepared under the two previously mentioned CVD conditions. The grains of parameter one (gas pressure 3.3 kPa, carbon-intensity 1.5%, temperature 780℃) has an uniform size of about 4μm as shown in Figure1(a) and presents a pyramid-shaped surface morphology. It is referred as ZMDF hereafter. The XRD results of ZMDF are shown in Figure 2. The main <111> peak has a higher intensity than the <110> crystallographic peak, implying that it grows along the <111> crystal orientation. Diamond film prepared by parameter 2 (gas pressure 2.5 kPa, carbon-intensity 1%, temperature 810℃) has the uniform size of 3μm as shown in Figure.1(b) and presents plane faces outwards. It is referred as FMDF hereafter. FMDF's XRD results are shown in Figure 2. The intensity of <111> peak decreases significantly compared with ZMDF. The <110> peak increased a significant amount in its intensity and therefore is the main peak. <311> peak is not visible. So it can be inferred that FMDF grows along the <110> plane orientation.

As mentioned previously, we found that the surface morphology was different if the preparation parameters were altered. Thus, different preparation parameters have an important influence on the surface morphology of the diamond film's surface. The parameters of temperature, pressure and carbon-intensity were considered in this experiment. Diamond peak was definitely less noticeable in the Raman spectrum when the deposition temperature dropped below 700℃. The non-diamond phase was more prominent, indicating lower diamond purity of the film [7].



(a) ZMDF



(b) FMDF

Figure 1. The SEM of two diamond coated tools' surface

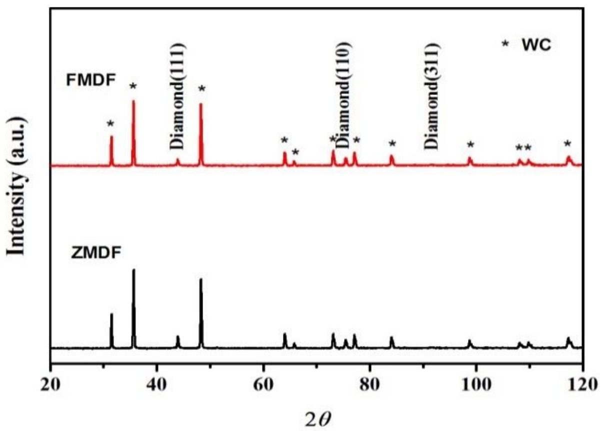


Figure 2. XRD of two samples

The hydrogen atom adsorption weakened and the diffusion rate of the active particles became slower when the deposition temperature was low. Therefore, low temperature is not conducive for the growth of diamond films [8]. According to thermodynamic conditions of diamond film growth, the films will not form when the deposition temperature is raised beyond the scope of the diamond growth curve. So temperature is one of the factors that influences the surface morphology of the diamond film. Three dynamic processes are understood to occur in the diamond kinetic models of hydrogen atoms in etching diamond. The three processes are described as follows: deposition of disordered carbon on the substrate surface, etching of disordered carbon and diamond by hydrogen atoms, and transformation of disordered carbon to diamond. Assuming the growing point of diamond component is D in total surface and components of disordered carbon with different thickness are C_1, C_2, $C_3,\ldots,C_n$, the dynamical evolution equations of the model are:

$$\frac{dD}{dt}=\varphi_{11}KC_1-\varphi_c AD-\varphi_{11}E_d D+\varphi_{11}E_c C_1 \quad (1)$$

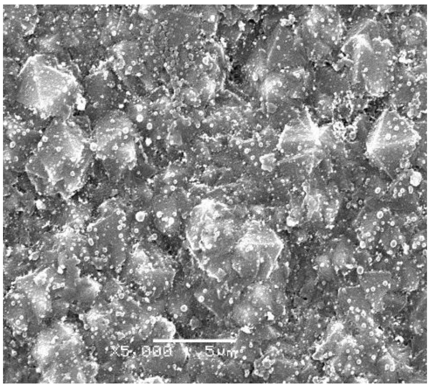
$$\frac{dC_1}{dt}=-\varphi_{11}KC_1+\varphi_c AD+\varphi_{11}E_d D-\varphi_{11}E_c C_1+\varphi_{11}E_c C_2 \quad (2)$$

$$\frac{dC_n}{dt}=\varphi_{11}AC_{n-1}-\varphi_{11}E_c C_n-\varphi_c AC_n+\varphi_{11}E_c C_n+1 \quad (3)$$

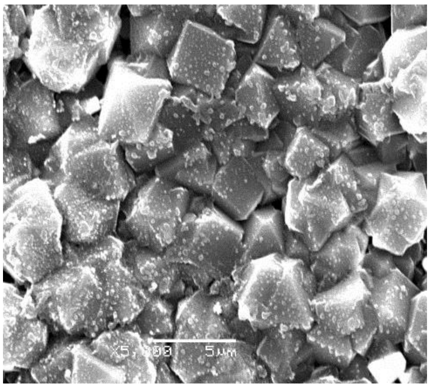
Diamond and disordered carbon competitive growth process can be obtained as follows when Equation (1)-(3) are solved. Deposition of disordered carbon was suppressed completely and the disordered carbon was converted to diamond when the ratio of carbon-hydrogen was in the area between two balance levels. The first balance level was between atomic hydrogen etching of diamond and diamond growth.

The second balance level was between atomic hydrogen etching and disordered carbon deposition. Diamond growth was completely inhibited and the growth of disordered carbon was a continuous process when the ratio of carbon-hydrogen was larger than the balance level between atomic hydrogen etching and disordered carbon deposition [9].

This showed that the ratio of carbon-hydrogen (carbon-intensity) was another factor that influenced the surface morphology of the diamond films. Gas molecules collided with one another more frequently when the deposition pressure was high. Under this condition, the density of hydrogen atoms is higher, which, in turn, suppresses nucleation of diamond phase [10]. So the gas pressure was the third factor that affected the surface morphology of the diamond film. ZMDF and FMDF can be prepared by carefully choosing above mentioned preparation parameters.



(a) ZMDF



(b) FMDF

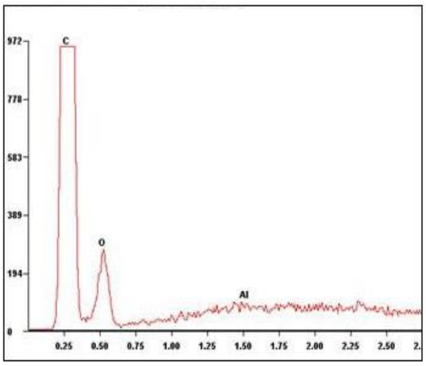
Figure 3. SEM of diamond coated tool's surface after lapping

3. 2. Comparison of Diamond Coated Tool and Sapphire before and after Lapping Figure 3 shows the surface morphology of the diamond films after lapping. Some debris was present outside the ZMDF and FMDF diamond film coated tools surface. Some of these debris would be swept out from the contacts through mechanical motions of the counterparts and others would be trapped on the worn diamond film surface. Severe wear occurred especially under the intensive interactions occurring during the initial period. These wear debris would transfer from the sapphire wafer to the diamond film surface and accumulate on it gradually [11].

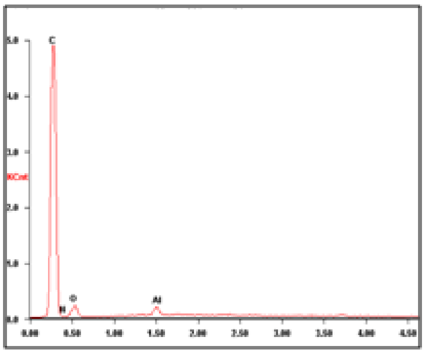
Energy Dispersive Spectrometer (EDS) of the two samples are shown in Figure 4. The main elements at the surface after lapping were C, O and Al, with very little evidence for the presence of other elements. This indicates that the debris composition is sapphire(Al_2O_3).

Figure 3(a) shows that, ZMDF is still presented in a pyramid surface morphology after lapping, while. Figure 3(b) shows, FMDF too retains its surface morphology after lapping. Sapphire debris was granularly spread over the grains. More debris was present on the ZMDF surface than on the FMDF surface.

The lapping friction coefficient curves are shown in Figure 5. As indicated in Figure 5, the friction curve of lapping between the diamond coated tool and sapphire wafer starts at the highest value, decreases gradually over a certain time period, and finally stabilizes at a constant value. In the stationary phase, the friction coefficient of the ZMDF tool is larger than that of the FMDF tool. This may explain why the debris left on the ZMDF's surface is more than that of FMDF.



(a) ZMDF



(b) FMDF

Figure 4. EDS of diamond film coated tool's surface after lapping

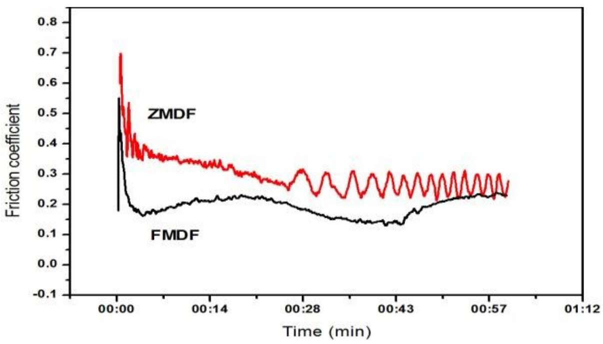
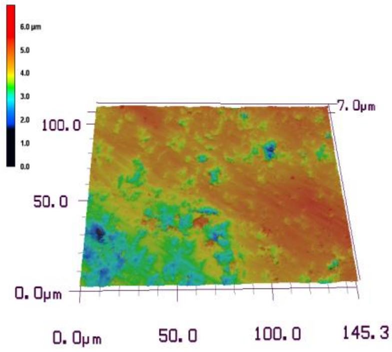
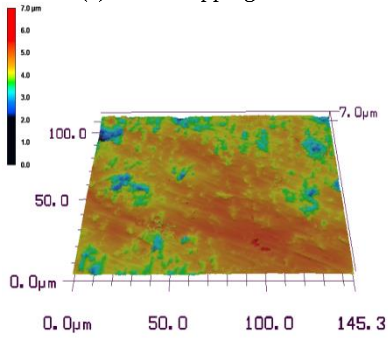


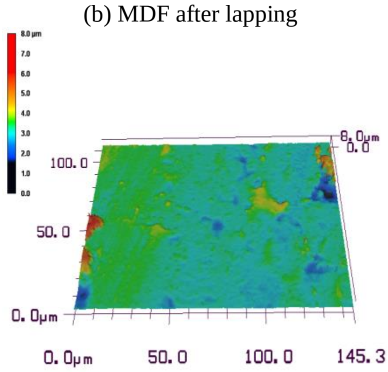
Figure 5. Friction coefficient curves



(a) Before lapping



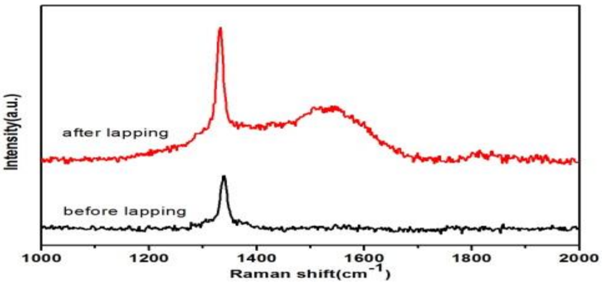
(b) MDF after lapping



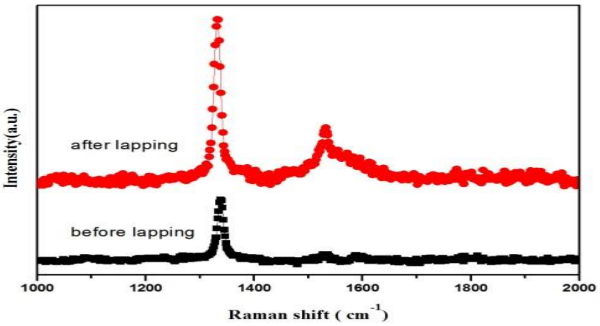
(c) FMDF after lapping

Figure 6. 3D Surface morphology of sapphire wafer

The 3D morphology of the sapphire before and after the lapping test was compared, as shown in Figure 6. The surface roughness of the sapphire wafer was about 0.605μm before lapping by the diamond coated tool, as shown in Figure 6a. The surface roughness decreased to about 0.440 μm(R_a) after lapping by ZMDF as depicted in Figure 6b. As shown in Figure 6C, sapphire wafer's surface roughness was about 0.248μm(R_a) after lapping by FMDF tool. The quality of the sapphire surface was improved with both methods and resulted in semi-finishing effects.



(a) ZMDF



(b) FMDF

Figure 7. Raman of two coated tool's surface before and after lapping

Raman spectra of the two diamond films before lapping are shown in Figure 7. A sharp peak is observed at 1332 cm^{-1}, which indicates that the diamond film grew with the desired quality with negligible amount of the non-diamond phase. The peak which presents at 1332 cm^{-1} is typical of a diamond sp^3 structure. Raman spectrum of the films after lapping are also shown in Figure 7. It shows that besides a peak at 1332 cm^{-1}, there is another peak at about 1540 cm^{-1} [12]. The broad band ranging from 1500 cm^{-1} to 1600 cm^{-1} is associated with the G band of sp^2 graphitic carbon. The transformation after lapping may result from the oxidizing ions reacting with diamond shown by Equation (4):

$$\text{Diamond} + O \rightarrow C + CO \quad (4)$$

The carbon from Equation (4) could be in the form of graphite or amorphous carbon [13]. Due to the dynamic frictional heating, the interface temperature was very high, and the high temperature could stimulate the transformation of diamond to non-diamond carbon [14].

4. CONCLUSION

<111> and <110> textured micron-diamond film coated tools were prepared on YG6 substrates. Friction coefficient of lapping between the diamond coated tool and sapphire wafer are all started at the highest value, decrease gradually in a certain time period, and finally stabilize at a constant value. Two types of micron-diamond film's Raman spectrum were changed after lapping that the peak at the position of 1560 cm^{-1} appeared. Sapphire wafer's surface roughness was about 0.440 μm after lapping by ZMDF and about 0.248 μm after lapping by FMDF. Surface graphitization was obvious. Sapphire wafer surface is much improved after lapping with <111> and <110> textured micron-diamond film coated tools.

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6. REFERENCES

1. Zhu, H., Tessaroto, L.A., Sabia, R., Greenhut, V.A., Smith, M. and Niesz, D.E., "Chemical mechanical polishing (CMP) anisotropy in sapphire", ***Applied Surface Science***, Vol. 236, No. 1, (2004), 120-130.
2. Park, H. and Chan, H.M., "A novel process for the generation of pristine sapphire surfaces", ***Thin Solid Films***, Vol. 422, No. 1, (2002), 135-140.
3. Liang, Z., Wang, X., Wu, Y., Xie, L., Liu, Z. and Zhao, W., "An investigation on wear mechanism of resin-bonded diamond wheel in elliptical ultrasonic assisted grinding (EUAG) of monocrystal sapphire", ***Journal of Materials Processing Technology***, Vol. 212, No. 4, (2012), 868-876.
4. Vaezi, M. and Zameni, M., "Synthesis of zinc oxide nanostructured thin film by sol-gel method and evaluation of gas sensing properties", ***International Journal of Engineering-Transactions B: Applications***, Vol. 27, No. 5, (2013), 757-763.
5. Tomé, M., Fernandes, A., Oliveira, F., Silva, R. and Carrapichano, J., "High performance sealing with cvd diamond self-mated rings", ***Diamond and Related Materials***, Vol. 14, No. 3, (2005), 617-621.
6. Kamiya, S., Takahashi, H., Polini, R. and Traversa, E., "Quantitative determination of the adhesive fracture toughness of cvd diamond to wc–co cemented carbide", ***Diamond and Related Materials***, Vol. 9, No. 2, (2000), 191-194.
7. Sha, L., "Study on the substrate surface pretreatment at cemented carbides and diamond coatings", ***Central South University***, Vol. 5, (2003), 457-462.
8. Long, F., Wei, Q., Yu, Z., Luo, J., Zhang, X., Long, H. and Wu, X., "Effects of temperature and mo 2 c layer on stress and structural properties in cvd diamond film grown on mo foil", ***Journal of Alloys and Compounds***, Vol. 579, No., (2013), 638-645.
9. Guowei, Y. and L. Jing and "A kinetic model for the growth of diamond films by chemical", ***Journal of Synthetic Crystals***, Vol. 82, No. 12, (1997), 6293-6300.

10. Xianming, M., W. Fengying, H. Lifu, , "Effects of deposition parameters on cvd nanocrystalline diamond thin films", ***Materials Science and Technology***, Vol. 3, No., (2009), 393-396.

11. Shen, B. and Sun, F., "Effect of surface morphology on the frictional behaviour of hot filament chemical vapour deposition diamond films", ***Proceedings of the Institution of Mechanical Engineers, Part J: Journal of Engineering Tribology***, Vol. 223, No. 7, (2009), 1049-1058.

12. Wang, T., Jiang, X., Biermanns, A., Bornemann, R. and Bolívar, P.H., "Deposition of diamond/β-sic composite gradient films by hfcvd: A competitive growth process", ***Diamond and related materials***, Vol. 42, No., (2014), 41-48.

13. Field, J.E., "The properties of diamond, Academic Press, (1979), 234-241.

14. Zhou, L., Huang, S., Wang, X. and Xu, L., "High-speed mechanical lapping of cvd diamond films using diamond wheel", ***International Journal of Refractory Metals and Hard Materials***, Vol. 35, No., (2012), 185-190.

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چکیده

دو نوع فیلم میکرون-الماس بر روی سوبسترای YG6 با روش رسوب شیمیایی بخار رشته داغ (HFCVD) ساخته شد. مورفولوژی و جهت رشد کریستالی توسط میکروسکوپ الکترونی روبشی (SEM) و پراش اشعه X (XRD) مورد بررسی قرار گرفت. سطح الماس ابزارهای روکش دار شده با الماس و ویفر یاقوت قبل و بعد از تا خوردگی مقایسه شدند. طیف رامان از دو فیلم میکرون-الماس نشان داد که تغییر قابل توجهی در خواص سطحی پس از تا خوردن رخ داده است. این تغییر به گرافیتی شدن سطح الماس مربوط می شود. ضریب اصطکاک ZMDF بزرگتر از ابزار FMDF بود. زبری سطح ویفر یاقوت تا خورده توسط FMDF (بافت <110>) کمتر از (<111>) ZMDF بود. با مقایسه دو نوع فیلم الماس-میکرون معلوم شد که اثر تا خوردگی ابزار FMCD بهتر بود.

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