CVD Micro-diamond Coated Tool Lapping with Sapphire Wafer

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(Received 18 January 2016; revised 23 March 2016; accepted 5 January 2017)

Abstract: Micro-diamond films were prepared on YG6 substrate by hot filament chemical vapor deposition method. An innovative micro-diamond coated tool was used to the lap sapphire wafer. The effect of load, rotating speed, and lapping time on material removal rate(MRR) and surface roughness was investigated. The results showed that the best process parameters were 3 N, 100 r/min and 15 min. The surface quality of sapphire improved significantly after lapping . The coating after lapping adhered well and did not show any peeling. The innovative micro-diamond coated tool was feasible and suitable for the lapping of the single crystal sapphire wafer.

Key words: micro-diamond film; sapphire; surface roughness; lapping

CLC number: TG71; TG66 **Document code**: A **Article ID**: 1005-1120(2017)01-0081-08

0 Introduction

Sapphire wafers are the most widely used substrate for GaN light-emitting diodes (LEDS) due to their comparatively low cost, high quality, large diameter, optical transparency, chemical compatibility, and high-temperature stability[1]. It also has important applications in semiconductor applications, light-emitting diodes, lasers, and precision optics. Such applications require superior surface quality and stringent dimensional tolerances and hence require effective, reliable, and economical polishing techniques[2]. Uneda used slurry mixed free abrasive grains to supply to the contact surface between the workpiece and plate, thus improving the lapping and polishing characteristics[3]. Lee et al. proposed a sequential process of electrolytic in-process dressing (ELID) grinding and chemical mechanical polishing (CMP) for hard-brittle materials used in LEDs, with applications to silicon carbide (SiC), sapphire and gallium nitride (GaN)[4]. Liang et al. investigated the material removal characteristics in elliptical ultrasonic assisted grinding (EUAG) of the mono crystal sapphire using single diamond abrasive grain[5]. Kumar researched low temperature wet etching using various etchants reduced the sub-surface damage in sapphire wafer during lapping[6]. Furthermore, most of the published papers have studied the method of sapphire lapped by fixed abrasive pad[7-9]. However, by now, there have been few reports about the sapphire wafer lapped by diamond film coated tools. Diamond films are used in tool applications because of their hardness, excellent wear resistance and chemical inertness[10]. Preparation of diamond film using hot filament chemical vapor deposition (HFCVD) is the most mature one in CVD methods[11], owing to its low cost, simplicity of required apparatus, and the ability to deposite large-scale diamond films[12]. Therefore, CVD micro-diamond coated tool was prepared by hot filament chemical vapor deposition (HFCVD) method. We investigated the effect of mechanical process parameters, such as force, rotation speed of work table, and lapping time on the material removal rate (MRR) and roughness. Best process

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How to cite this article: Feng Wei, Lu Wenzhuang, Yu Yaping, et al. CVD micro-diamond coated tool lapping with sapphire wafer[J]. Trans. Nanjing Univ. Aero. Astro., 2017,34(1):81-88.

http://dx.doi.org/10.16356/j.1005-1120.2017.01.081

parameter of the surface after lapping has good MMR and high surface quality. In this study, we provide basic research for application of HFCVD diamond in the lapping of sapphire.

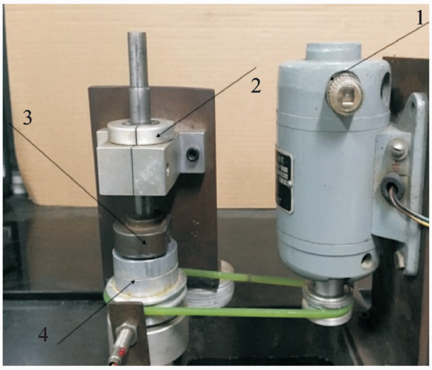
1 Experiment

CVD diamond coatings were deposited by the HFCVD method. The reaction gases are H_2 and CH_4. The substrate sample is YG6, of which 6% is Co and 94% is WC. Its measured diameter is ∅10 mm. The samples were grinded before deposition by W10 metallographic sandpaper. Dirt and impurities were removed by ultrasonic cleaning. And then for the pre-treatment, Firstly, 20 min ultrasonic etching was carried out to make the WC′s surface layer broken. The samples were immersed in 10 g KOH, and 10 g H_2K_3O 100 ml reagent. Secondly, the samples were immersed in 10 ml HNO_3 and 30 ml HCl regent for two minutes in order to remove the Co before deposition. Finally, the samples were put into certain proportion of diamond-acetone and suspended for 20 min to further increase the nucleation rate. Laboratory-made CVD equipment was used in the process of diamond film deposition. The process parameters, such as gas pressure, substrate temperature, ratio of methane and total gas flow are summarized in Table 1. The surfaces of the diamond film were examined by a HITACHI S-3400 scanning electron microscope (SEM). The formations of different phases on the film were identified using D8Advance XRD(X-ray diffractometer).

Table 1 Process parametes of diamond film

Parmeter	Value
Gas pressure/kPa	3.3
Substrate/℃	780
Ratio of method/%	1.5
Total gas flow/($cm^3 \cdot min^{-1}$)	300

Here the sapphire wafers were lapped with CVD micro-diamond coated tools at room temperature without any lapping fluid. The photo of equipment is shown in Fig. 1. The sapplire fixture (part 4) turns to rotate sapphire to lapping with the lapping tool (part 3). The force was 1 N. The rotation speed was 100 r/min. The original sapphire wafer was 1 inches in diameter and its roughness was Ra 0.64 μm.



1.Motor; 2.Presure devive;
3.Lapping tool; 4.Sapphire fixture

Fig. 1 Photo of experimental apparatus

The weight of sapphire before and after polishing was measured by electron balance. The material removal rate (MRR) can be calculated by

$$\mathrm{MRR} = 1\,000 \times \frac{\Delta m \times H}{M \times t} \tag{1}$$

where MRR (μm/min) is the corresponding removal rate, Δm (g) the mass variation of sapphire before and after lapping, t (min) the lapping time, M the sapphire wafer′s original weight, and H the sapphire wafer′s original thickness. Wear depth and surface roughness were measured by a nanomap-500LS 3D profilometer. The data obtained here was from the average of 5—6 measured points for each wafer.

2 Results and Discussion

2.1 Microstructure of CVD micro-diamond film

XRD and SEM were used to test and analyze the CVD diamond film surface topography. Fig. 2 showed the microstructures of CVD diamond film after deposition.

Using the process parameters from Table 1, diamond film surface morphology was produced, which were clear and distinguishable with general structures. Diamond film presents a pyramid-

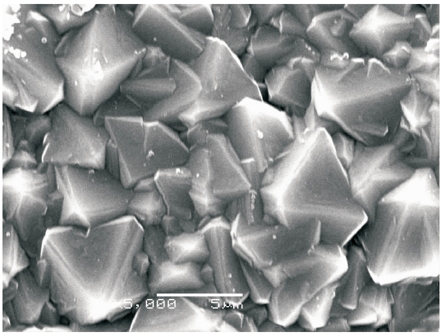


Fig. 2 SEM images of CVD micro-diamond film

shaped surface morphology. The grains of diamond film with uniform size of 4 μm have a micron diamond shape. CVD micro-diamond film's XRD result was shown in Fig. 3. The main peak 〈111〉 has the stronger energy level than 〈220〉 (〈110〉) crystallographic peak. Therefore it can be inferred that it grew along the 〈111〉 crystal orientation, thus leading to an outwards pyramid shape.

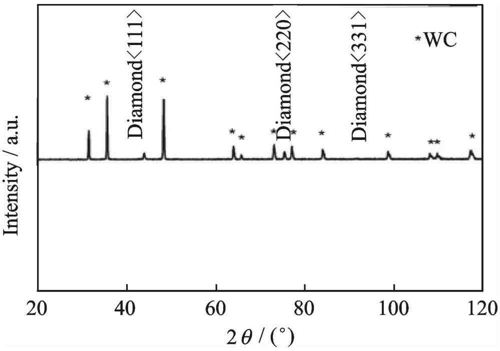


Fig. 3 XRD pattern of CVD coating

2.2 Material removal rate and surface roughness

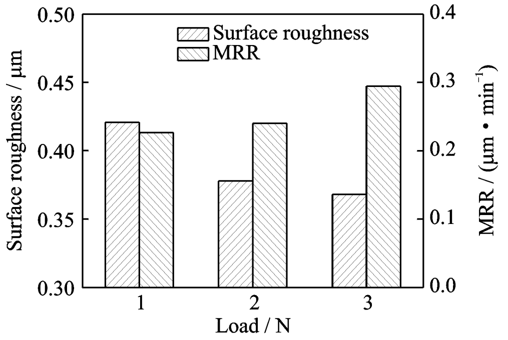
The effect of load, rotating speed of the workbench and the lapping time on the MMR and surface quality of sapphire wafer was studied in this experiment. Three factors were considered: load, rotating speed of workbench and lapping time. The orthogonal experimental method was used.

Each factor has three levels. Load levels were 1 N, 2 N, and 3 N. Rotating speed levels were 50 r/min, 100 r/min, and 150 r/min. And lapping times were 3 min, 15 min, and 30 min. The L9 (3^4) orthogonal experimental table was chosen. Material removal rate and surface roughness results are shown in Table 2.

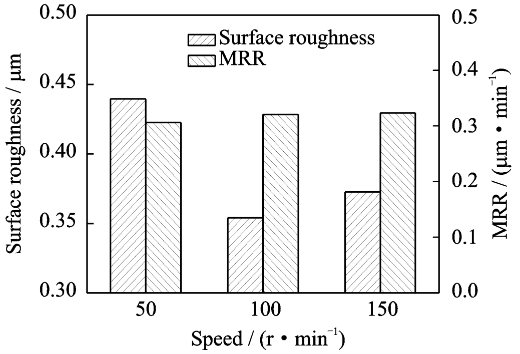
Table 2 Factors and results of orthogonal experiment

No.	Factor			Result	
	Load/N	Speed/($r \cdot min^{-1}$)	Time/min	Surface roughness Ra/μm	MRR/($\mu m \cdot min^{-1}$)
1	1	50	3	0.462	0.214 8
2	1	100	60	0.393	0.212 6
3	1	150	15	0.407	0.252 3
4	2	100	15	0.315	0.179 2
5	2	150	3	0.428	0.310 8
6	2	50	60	0.390	0.220 4
7	3	150	60	0.283	0.2142
8	3	50	15	0.467	0.289 8
9	3	100	3	0.354	0.378 7

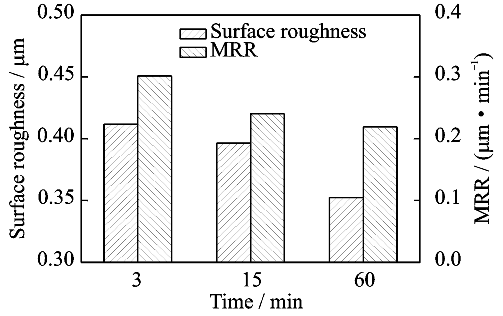
To further investigate the effect of each factor, Fig. 4 was drawn based on Table 1. The effects of the lapping parameters on the MMR and surface roughness were shown in Fig. 3. Each level of the experiment was repeated three times and the mean value was used. Fig. 4(a), as a trend graph, showed how the load influenced the material removal rate and surface roughness. Obviously, with the increase of load, the material removal rate increases. This is because, the depth of the micro-diamond grains pressed into the sapphire surface is much higher when the load is increasing. Meanwhile, the number of effective grains increase and accordingly the indentation depth. Finally, the increase of the cutting output results in an increase in the removal rate. In addition, with an increase in the load, the surface roughness of the substrate gradually decreases. After the load increases, the depth of the equivalent abrasive grains embedded in the matrix layer of the substrate was relatively uniform because of the undulating surface of diamond coating, thus bringing forth the decrease in roughness. At the same time, for the sapphire substrates, incomplete brittle fracture occurs under an appropriate load[13] and under the embedding, extrusion and cutting action. Meanwhile, phenomena similar to lateral plastic flow and accumulation of metal materials during plastic metal cutting occur. In the plastic cutting deformation process, the surface damage can be weakened significantly and uneven brittle delamination can be improved, obtaining better surface roughness[14].



(a) Load



(b) Speed



(c) Time

Fig. 4 MRR and surface roughness under different levels of various factors for load, speed and time

The relationship between the rotating speed of the workbench and MMR is shown in Fig. 4 (b). Withan increase in the rotational speed of the sapphire substrate, the material removal rate tends to increase. When the rotational speed increases, the number of diamond grains involved in the material removal per unit time increases. Moreover, for a single grain, its trajectory length on the surface of sapphire substrate extends. Hence, the removal rate increases. In addition, in Fig. 4, with an increase in the rotational speed, the surface roughness first decreases, and then increases. As the surface roughness value of the initial sapphire substrate is relatively higher, the flatness is poor after slicing.

With an increase in the rotational speed, the larger protrusions remain on the grain removal surface, which help improve the microscopic flatness of the surface. Moreover, the increase in the rotational speed keeps the operation stable and reduces surface roughness. When the rotational speed reaches 150 r/min, the roughness increases lightly. As the depths of the different grains on the same coating layer embedded in the sapphire substrate vary, the removal amount is different. With a continuous increase in the rotational speed, the removal amount of different grains varies greatly, and the surface roughness increases accordingly.

The relationship between MMR and the lapping time is shown in Fig. 4(c). With the increase in the grinding time, the material removal rate tends to decrease. The initial cutting trace of the sapphire substrate was relatively deep and the surface was relatively rough. After the micro-convex peak on the surface was first removed during grinding with diamond grains, the abrasion dust adhered to the surface of the coating with time and the material removal rate thus decreases with time. Besides, with an increase in the grinding time, the surface roughness tended to decline. The surface roughness of the part decreased from 0. 64 μm to about 0. 43 μm in a short time (3 min). As the surface of the sapphire substrate after the initial cutting was rough, the larger convex peaks on the surface of the sapphire substrate were removed rapidly by the diamond grains during the initial stage of grinding. Therefore, the surface roughness decreased rapidly in a short time.

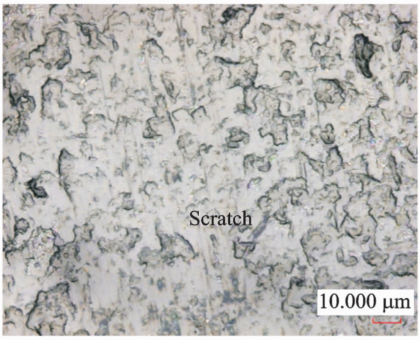
The orthogonal experiment range was analyzed for the material removal rate and the surface roughness. Various factors such as grinding time, load and rotational speed of workbench have different effects on the material removal rate. To obtain the maximum sapphire material removal rate, the optimum experimental combination scheme is $A_3B_3C_1$. Similarly, various factors, such as rotational speed of the work bench, grinding time and load have different effects on the surface roughness. For the minimum surface roughness, the optimum experimental combination scheme is $B_2C_3A_3$. It can be seen from Fig. 4(a) that, with an increase in load, the sapphire material removal rate increased and the surface roughness decreased. Hence, the optimum load should be 3 N. In Fig. 4(b), the rotational speed of the work bench has less impact on the material removal rate. A lower surface roughness can be obtained at the rotational speed of 100 r/min. So the optimum rotational speed of the workbench should be 100 r/min. Fig. 4(c) demonstrates that, with an increase in the lapping time, the material removal rate decreased. With a decrease in Lapping time, the surface roughness increaseed. Hence, 15 min was suitable for lapping. Considering the impact of various process parameters on the removal rate, the surface roughness, and their trends, the lapping process parameters were determined as follows, based on the lapping efficiency and the improvement in the surface quality: load of 3 N, rotational speed of 100 r/min and grinding time of 15 min. When the validation experiment was conducted using the afore-mentioned set of process parameters with the diamond-coated tool used to lap, the sapphire material removal rate 0.33 μm/min and the surface roughness is 0.331 μm. These were considered as the optimal process parameters for lapping.

As a basic study for innovating a method for lapping sapphire, the experiments are performed under dry conditions. The MMR will be greatly improved upon adding a lapping liquid, because the liquid can react with the sapphire and soften it. Gagliardi[15] studied the two-body free abrasive lapping of sapphire, obtaining an average roughness *Ra* 0.877 μm. Compared with the free abrasive, our method has a lower surface roughness. Li [16] studied the fixed abrasive method for lapping sapphire, obtained a *Ra* of 0.309 μm, similar to our results. This is associated with the actual experimental parameters and environment. The new method has unique advantages, which is associated with the growth mechanism of the diamond film.

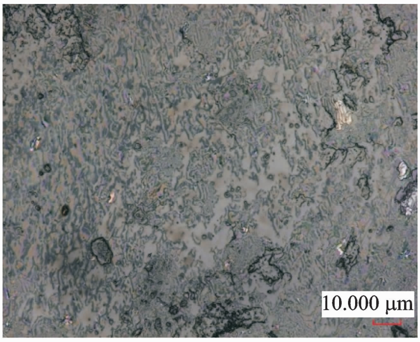
The fist growth step of a diamond film is nucleation. Then, according to a certain orientation, it grows into diamond grain. Furthermore, overlay of staggered grains gradually forms the diamond film on the surface of substrate. The diamond grain shape and size can be controlled by processing parameters. There are gaps between the diamond grains and a part of the hard phase abrasive dust falls into the gap and separates from the meshing surface during lapping. Other parts form grinding grains plough between the diamond and sapphire. The diamond film coating tools display self-dressing characteristics because of the layer growth feature as mentioned above. In addition, the grain size of the diamond film can be in a nanometer range. Hence, compared to other lapping methods, our method can adjust the lapping surface quality more flexibly by controlling the preparation parameters.

2.3 Comparison of sapphire and diamond surface before and after lapping

KEYENCE-VK-X100 measuring laser microscope system was used to observe the grinding crack on the surface of the single crystal sapphire wafer after lapping. Surface characteristics are shown in Fig. 5. The long sapphire crystal rod was formed at one time, and then wire-electrode was cut into wafers. Since the goal of the follow-up study was to produce a controlled trial textured diamond tool for grinding and polishing single crystal sapphire wafer, unfinished sapphire wafer was chosen in our experiment, which has a large number of surface defects and scratches, as



(a) Before lapping



(b) After lapping

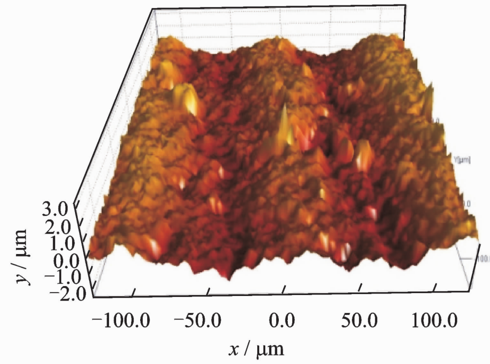
Fig. 5 Surface characteristics of sapphire before and after lapping

shown in Fig. 5 (a).

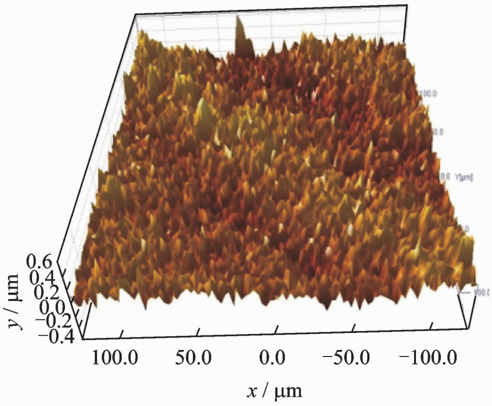
Fig. 6 showed the comparison of the sapphire surface topography before and after lapping tested by nano-map. The surface quality has been significantly improved after lapping. The roughness (Ra) was about 0.8 μm. The roughness was about 0.3 μm after lapping. So Figs. 4, 6 all proved that the innovative micro-diamond coated tools lapping with single crystal sapphire wafer was feasible and can produce certain effects.

Fig. 7 was a typical SEM image showing the surface morphology on the worn diamond film surface. The grains of diamond film presented a pyramid-shaped surface morphology. A lot of sapphire debris remained on the film surface. The coating was tightly combined and had no peeling off phenomenon. From Fig. 8, it can be observed that the sapphire debris produced discontinuous short shavings. It was hardly removed due to its high hardness[17].

Sapphire debris adhered to the grain groove



(a) Before lapping



(b) After lapping

Fig. 6 Surface morphology of sapphire substrate before and after lapping with CVD diamond coating tool

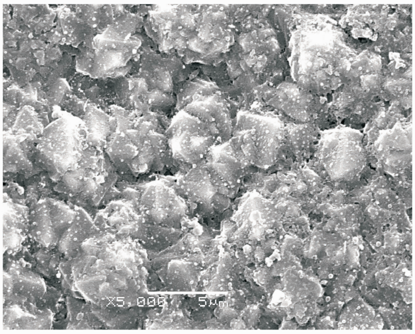


Fig. 7 SEM image of sapphire surface after lapping

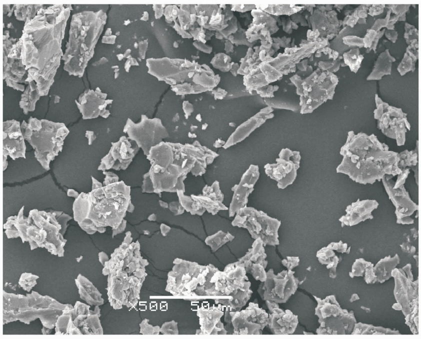


Fig. 8 SEM image of sapphire debris in lapping process

was shown in Fig. 8. There were a few deep trenches besides large amounts of debris adhering to the surface. Since the diamond is harder than alumina, diamond was probably plugged into the wafer's surface owing to the outside force and their sharp edges and corners, acting like knives to destroy the surface[18]. This was also helpful to improve the removed rate of sapphire by micro-diamond film coating tool. So it is a good method to lapping sapphire wafer with micro-diamond film.

3 Conclusions

(1) A micro-diamond film was prepared by controlling the deposition parameters. The film presented a pyramid-shaped surface.

(2) The descending order of factors that impact MMR were the lapping time, load, and finally the rotating speed of the workbench. In the same order, the factors that impact the surface roughness were the rotating speed of the workbench, lapping time, and load.

(3) The optimal best process parameters were 3 N, 100 r/min, and 15 min. The MMR of the sapphire was 0.33 μm/min and the surface roughness was *Ra* 0.331 μm when the experiment was performed under three process parameters. The coating after lapping adhered well and did not show any peeling. Use of the innovative micro-diamond coated tool for lapping single crystal sapphire wafer was thus found to be feasible.

Acknowledgements

This research was supported by the National Nature Science Foundation of China (No. 51275230), NUAA Research Funding (No. kfjj20150507), Six Talent Peak of Jiangsu (No. ZBZZ-002), the Funding of Jiangsu Innovation Program for Graduate Education (No. KYLX_0227), and the Fundamental Research Funds for the Central Universities.

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