Diamond & Related Materials 84 (2018) 119–126

Contents lists available at ScienceDirect

Diamond & Related Materials

journal homepage: www.elsevier.com/locate/diamond

An efficient titanium-containing corundum wheel for grinding CVD diamond films

Hanqing Xu[a], Jianbing Zang[a], Guoping Yang[a], Pengfei Tian[a], Yanhui Wang[a,*], Yiqing Yu[b], Jing Lu[b], Xipeng Xu[b,*], Pingwei Zhang[c]

[a] State Key Laboratory of Metastable Materials Science and Technology, School of Materials Science and Engineering, Yanshan University, Qinhuangdao 066004, PR China
[b] MOE Engineering Research Center for Brittle Materials Machining, Huaqiao University, Xiamen 361021, PR China
[c] Hebei Plasma Diamond Technology Co., Ltd.(Hebei Institute Of Laser), Shijiazhuang 050000, PR China

ARTICLE INFO

Keywords:
CVD diamond film
Grinding
Spark plasma sintering
Dynamic friction polishing
Material removed rate

ABSTRACT

The titanium-containing corundum wheel was designed for a high-efficiency grinding chemical vapor deposition diamond film. It had been shown that the grinding wheel exhibited the highest material removal rate compared with metal grinding wheel (e.g. Ti alloy, SUS 304 and cast iron) and by using this grinding wheel the material removal rate could reach 5.57–56.35 μm h^{-1} at a grinding speed ranging from 400 rpm to 700 rpm. The surface character of the diamond films under different grinding speeds had also been studied by means of the scanning electron microscope. Graphite and carbide were detected by means of X-ray diffraction, the transmission electron microscope and Raman spectroscopy. The results indicated that the chemical reaction between diamond and titanium and the graphitization combined with mechanical cracking account for the high material removal rate in the grinding process.

1. Introduction

Chemical vapor deposition (CVD) diamond films have identical or similar physical and chemical properties compared to natural diamonds [1–5]. However, CVD diamond films are generally uneven in thickness, different in grain size, rough at the surface, with the roughness increasing in line with the thickness of the film increasing [6]. Surface roughness and non-uniform thickness severely restricted the application of the diamond film in cutting tools, heat sinks, semiconductors, optical windows [7,8]. Therefore, the CVD diamond film polishing technology has become one of the key technologies in precision finishing technology. In the process of polishing CVD diamond film, grinding the CVD diamond film to achieve a relatively flat surface is the first step. Then surface planarization is the second step. On account of the superior hardness and chemical inertness of the diamond, it is difficult to polish diamond. Various polishing techniques have been put forward, including mechanical polishing [9,10], thermochemical polishing [11–15], laser polishing [16] chemo-mechanical polishing [17–19]. Among them, some polishing methods either require a complicated apparatus or a special environment. Most of them fail to meet the requirements of high efficiency and low cost. A high efficiency and cost-effective polishing method is needed urgently.

Evan L. H. Thomas et al. [20] polished CVD diamond films using Chemical Mechanical Polishing (CMP). The roughness values have been reduced from 18.3 nm to 1.7 nm in 4 h. Therefore CMP is a precision polishing method for thin film diamond. With the successful synthesis of colorless transparent large single crystals diamond in the high temperature and pressure and a large amount of industrial production, processing large single crystal diamond requires a high removal rate. Dynamic friction polishing (DFP) is an efficient and cost-effective polishing method [21–23]. The DFP utilizes the thermo-chemical reaction between the CVD diamond films and the rotating polishing wheel by frictional heating at the sliding interface. The common polishing wheel consists of one or more metals (e.g. transition metal), which can accelerate the formation of the metal carbides or metal carbon oxides. The graphitization of diamonds will be more likely to occur due to the reduced energy barrier between diamond and graphite. Nevertheless, the traditional polishing wheel deformed easily at high temperature conditions in the DFP process, which decreased the material removal rate. Consequently, a new polishing wheel is prepared by adding active metal to a corundum wheel. Active metal can catalyze the diamond graphitization at high temperature caused by friction heating, or some carbides are formed, then the corundum abrasive can remove these soft phases (non-diamond carbon and carbides) quickly. E. Paul [24] indicated that the unpaired d electron of the transition elements may be the driving force of diamond graphitization. Besides, O. Johnson et al.

* Corresponding authors.
E-mail addresses: wangyanhui@ysu.edu.cn (Y. Wang), xpxu@hqu.edu.cn (X. Xu).

https://doi.org/10.1016/j.diamond.2018.03.018
Received 5 November 2017; Received in revised form 10 March 2018; Accepted 18 March 2018
Available online 19 March 2018

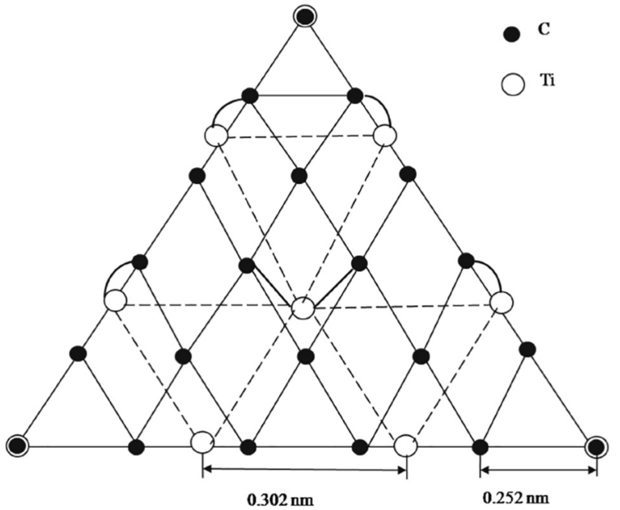

Fig. 1. The relationship of structure between α - Ti and the diamond (111) plane.

[25] also considered that if the catalyst metal possessed not only unpaired d electrons, but also correspondingly aligned atoms to form vertical bonds, the metal could have a dual effect on the diamond graphitization. In this paper, pure titanium was chosen as an active metal. The titanium has a close-packed hexagonal structure, and the atomic distance on the dense plane is 0.302 nm, but the atomic spacing on the diamond surface is 0.251 nm, as shown in Fig. 1.

A part of the Ti atoms is periodically aligned with the C atom and connected into a vertical covalent bond. Another part of the Ti atoms form a bridge three central π bond with two adjacent C atoms.

This paper focused on grinding CVD diamond films. The influence of the grinding speed on the surface morphology, the relationship between the material removal rate and the grinding speed, and the grinding mechanism were also investigated.

2. Experimental

In this work, the as-prepared diamond films were cut as disk-shaped with diameter of 30 mm and thickness of 2 mm (diamond film, purchased from Hebei Plasma Diamond Technology Co., Ltd. China). Copper powders (45 μm in size), tin powders (45 μm in size), aluminum oxide powders (7 μm in size) and titanium powders (45 μm in size) were used as raw materials to prepare the grinding wheel. Copper, tin, aluminum oxide and titanium at a mass ratio 16:4:30:50 were mixed before spark plasma sintering (SPS) in a planetary ball milling equipment. After being mixed well, adding glycol was used as the temporary binder. The mixture was poured into a graphite mold with a size of Φ30 mm, and the hot-press sintering was conducted. The sintering system was evacuated to a low vacuum of about 10^{-2} Pa prior to sintering with a rotary vacuum pump. The sintering pressure, sintering temperature and holding time were 30 MPa, 800 °C, and 15 min, respectively. After cooling to room temperature, the samples were taken out. The obtained samples were cut into an annular shape, whose external diameter, inside diameter, and thickness of were 30 mm, 18 mm, and 5 mm, respectively. The compound containing different contents of titanium was denoted as T_0 and T_{50}. The component of T_0 and T_{50} was shown in Table 1.

Table 1
Composition of mixture weight ratio (wt%).

	Cu	Sn	Al_2O_3	Ti
T_0	32	8	60	0
T_{50}	16	4	30	50

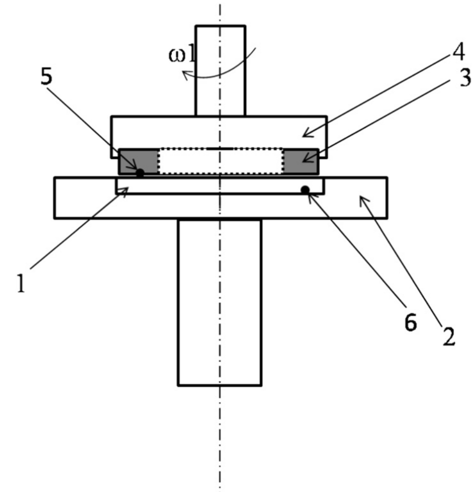

Fig. 2. Schematic illustration of grinding. (1) CVD diamond film. (2) Cast iron mold. (3) Grinding wheel. (4) Grinding wheel specimen holder. (5) Point for measuring temperature. (6) Pressure sensor.

The average interfacial temperature between the grinding wheel and the CVD diamond film was measured by means of a single-point temperature measurement method at different grinding speeds. The nickel-chromium-nickel-silicon thermocouple temperatures ranged from 200 °C to 1300 °C. The samples of hardness were measured by full-automatic microhardness testing system (FM-ARS 9000). The samples of hardness (HV) were calculated by Eq. (1).

$$HV = \eta \frac{F}{d^2} \tag{1}$$

where η is the constant, $\eta = 1.85 \times 10^{-3}$, F is the load (gf, 300 gf for T_{50}, Ti alloy and SUS 304, 200 gf for cast iron), d is the average diagonal length of the indentation (mm).

The diagram for grinding the CVD diamond film was shown in Fig. 2.

The CVD diamond film was fixed in the panel, pressed by the grinding wheel under pressure. The grinding parameters: grinding speed was 400 rpm (0.63 m/min), 500 rpm (0.79 m/min), 600 rpm (0.94 m/min) and 700 rpm (1.1 m/min), respectively. The pressure of grinding was 0.66 MPa, and the time of grinding was 15 min. After the grinding experiment, the fragment was collected.

The stylus profilometer and the S4800 scanning electron microscope (SEM) and were used to measure surface roughness and characterize morphology of the diamond films before and after grinding, respectively. The transmission electron microscope (TEM) (JEM-2010) was used to observe the morphology of the grinding fragments. We used the energy-dispersive spectroscope (EDS) to analyze the chemical composition, and the X-ray diffraction (XRD) (XRD, Cu Ka radiation, λ = 0.154056 nm, 40 kV, 100 mA) to identify the chemical phases of the grinding fragments and the grinding wheels before and after grinding. Roman spectroscopy (the excitation wavelength is 514.5 nm) was used to identify the different structures of carbon.

3. Results and discussion

3.1. Mechanical properties and microstructures of composites

Before grinding experiments, the bending strength of the composites was tested, as shown in Fig. 3. The morphologies of composites fractures are shown in Fig. 4. Obviously, pores and cracks occupy a high proportion of the structure and Al_2O_3 is connected with very few binders in the T_0 composite, which will lead to a weak interface adhesive strength with abrasive, as shown in Fig. 4a. T_{50} composite reveals a more compact structure than T_0 composite and the abrasives were held