



Original Article

Thermal damage mechanisms of Si-coated diamond powder based polycrystalline diamond

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ABSTRACT

The polycrystalline diamond (PCD) composites were synthesized from Si-coated diamond or diamond powder mixtures with Si. The characterizations of phase structures, morphologies and thermal stability of PCD were investigated by x-ray diffraction (XRD), scanning electron microscopy (SEM) and thermal gravimetric-differential scanning calorimetry (TG-DSC). The thermal damage mechanisms of PCD were studied under air condition up to 750 °C by thermal weight loss, XRD, SEM and wear tester. The results showed that the PCD was successfully synthesized by the Si-coated diamond with a homogeneous structure. The thermal stability and wear resistance of PCD with Si-coated diamond were better than those of PCD with uncoated diamond. It was determined that the chemical thermal damage mechanism with oxidation reaction dominated the damage of the PCD after high temperature annealing and the physical thermal damage mechanism also took effect on the exfoliation of binder and fine diamond grains from PCD.

1. Introduction

Diamond with exceptional physical, chemical, mechanical and electrical properties is arousing great interests in modern industries. However, the poor modifiability due to the high hardness and high cost limit the application of large bulk diamond as tools [1]. With the advent of diamond composites, tremendous changes have taken place to solve these problems. Diamond composites are produced from the micron and nanoscale diamond powders with binders to form a compacted body. They are extensively used as drilling bits, cutting tools and wire drawing dies [2–4]. The diamond composites made by high pressure high temperature (HPHT) method consist of polycrystalline diamond compact (PDC) sintered through direct diamond bonding and polycrystalline diamond (PCD) bonded by adding binder materials. The former PDC shows excellent physical properties such as friction and wear behaviors in vacuum and air [5–7]. However the metallic agent in the PCD led to deterioration such as graphitization and micro-cracks at high temperature [8,9]. Conversely, the latter PCD composites usually present an excellent thermal stability with refractory materials as binders. The PCD produced by the mixing of silicon and diamond powder showed a thermally stable bonding up to 1200 °C due to the existence of SiC [10]. Diamond and MgCO₃ sintered system showed that neither

graphitization nor micro-cracking was observed on the PCD after vacuum annealing above 1400 °C for 30 min [11]. Furthermore, diamond-CaCO₃ and diamond-Li₂CO₃ were synthesized by the penetrating of carbonates into diamond layers and the diamond-carbonates had better wear resistance than the conventional PDC after annealing at 1200 °C in vacuum for 10 min [12]. In addition, Ti-Si-C compounds such as Ti₃SiC₂ were also proposed to be the binders in the preparation of PCD [13–15]. Among these binder materials, SiC usually develops from the reaction of silicon and diamond or graphite. The melting point of Si decreases as the pressure increase, which provides a diamond stable temperature region. The high melting point of reaction production SiC ensures the adaptability on high temperature environment. Furthermore, due to the structure similarity of diamond and Si, a strong bond can be achieved. Thus, Si becomes the most common binder in the industry. Basically, there are two approaches to the synthesis of diamond-SiC PCD, the infiltration of liquid Si on diamond powder and the sintering of diamond and Si powder mixtures. Ekimov et al. studied the nanocrystalline diamond ceramics synthesized by infiltration liquid silicon into nano-diamond powder and detected the self-stop silicon infiltration process by the closure of pores near the diamond boundary due to the formation of SiC [16]. The diamond-SiC nanocomposites sintered from the mixtures of diamond and silicon nanopowder or amorphous silicon or

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silicon powder by ball milling were also reported [17,18]. During the HPHT process, the Si nuclei dissolves the carbon under high pressure and then grows into SiC crystals. The wetting between Si and diamond is caused by the rapid formation of SiC which nucleates and quickly grows along the diamond and into the molten Si [19]. The reaction usually takes place on the surface defects concentrated area of diamond [20].

However, it is reported that the surface of diamond chemically bonded a large amount of oxygen containing functional groups, which critically affected the interfacial bonding between the adjacent grains [21]. The coating deposition technology is an effective method to enhance the interfacial bonding condition. Liu et al. reported that Ti, Mo and Cu were plated on the diamond surface through the forming of chemical bonds and the coating can protect the diamond from being eroded [22]. Chang et al. found that Ti-coated diamond grits were prepared by the spark plasma sintering method and the compressive fracture strength and toughness index of the Ti-coated diamond increase due to the chemical bonding of Ti and diamond [23]. Zhu et al. pointed out that Si-coated diamond particles can improve the heat conduction and bonding strength of diamond/copper composite by spark plasma sintering process [24]. Furthermore, it is supported that coating the diamond surface with barrier layer is a practical way to protecting the grits from degradation at elevated temperatures in oxidizing and corrosive environments [25–27]. Wang et al. studied that the Ti coating on the diamond can protect the diamond from oxidation [25]. Lu et al. proposed silicon film deposited on the diamond by atomic layer deposition method showing starting oxidation temperature higher than uncoated diamond [26]. Therefore, coating film on the diamond not only can prevent the erosion of diamond, but also realizes the strong binding between diamond and the bond. Moreover, Wang et al. successfully synthesized PCD by Ti-coated diamond under HPHT [28].

Based on the above studies, the PCD obtained by Si-coated diamond was arisen. Si as the coating can prevent the existence of oxygen containing functional groups. Besides, Si as the binder can provide the nuclei for the further formation of SiC binder. It is essential to investigate the properties of PCD with Si-coated diamond.

This work attempts to sinter the PCD from Si-coated diamond and uncoated diamond powders. Furthermore, the characterizations and thermal damage mechanisms of PCDs under air condition were explored so as to give insight into the thermal stability and oxidation properties for the further application as tools.

2. Experimental details

2.1. Materials synthesis

Commercial grade uncoated diamond powder, Si-coated diamond powder with a grain size of 12–30 μm (Sumitomo Electric Industries, Ltd) and Si powder with an average grain size of 3 μm (Zhongnan Diamond Co., Ltd) were used as starting materials. The Si-coated diamond was prepared by the chemical vapor deposition (CVD) method with 1 wt% Si. During the deposition process, the silicon was deposited by pyrolytic decomposition of silane at temperature ranged from 600 to 700 $^{\circ}\text{C}$ with the helium carrier gas flowing by 10 L/min. The mixtures of the Si-coated diamond and Si powder were prepared by weight ratio 100: 8.79. Thus, in the mixtures the weight ratio of diamond to silicon is 91:9. As a reference material, the 91 wt% uncoated diamond powder and 9 wt% Si powder were mixed with the same method. The mixture powders by wet mixing in methanol were continued for 24 h in a rotary blender with JS14S type (Xianyang Jinhong General Machinery Co., Ltd.). The methanol was removed by evaporation at 120 $^{\circ}\text{C}$ after mixing. In order to remove the impurities on the surface of the diamond and Si, the mixtures were treated in a vacuum furnace with 500 $^{\circ}\text{C}$ for 24 h.

High-pressure sintering was carried out by using a China-type cubic

high-pressure apparatus with an anvil top face of $34 \times 34 \text{ mm}^2$. The temperature was estimated from the predetermined relation between input electrical power and temperature, which had been obtained in advance by measuring the temperatures up to 1700 $^{\circ}\text{C}$ with Pt6%Rh-Pt30%Rh thermocouple. Pressure was calibrated using the known phase transitions of Bi, Tl, and Ba at room temperature. Based on the data obtained in a piston-cylinder apparatus, pressure correction at high temperature was made by using the melting curve of Ag. Samples were first subjected to a pressure of 5.0–6.0 GPa, then heated to a temperature about 1300–1400 $^{\circ}\text{C}$ for 3 min. The pressure of the samples was released after they cooled to room temperature. The samples were chemically purified to remove graphite residues. Sintered samples with uncoated diamond and Si-coated diamond powders were defined as P-PCD and Si-PCD, respectively.

2.2. Annealing tests

To examine the high temperature oxidation behaviors of the PCDs, the polished PCD samples were treated at temperatures of 600 $^{\circ}\text{C}$, 650 $^{\circ}\text{C}$, 700 $^{\circ}\text{C}$ and 750 $^{\circ}\text{C}$ for 30 min under air condition with a muffle furnace (SX-8-10) following by air cooling.

2.3. Characterization methods

Thermal gravimetric-differential scanning calorimetry (TG-DSC) analysis was conducted with air flowing at a heating rate of 20 $^{\circ}\text{C}/\text{min}$ from room temperature to 1200 $^{\circ}\text{C}$ (STA449F3, German). X-ray diffraction (XRD, D/max2500, Cu $K\alpha$, and $\lambda = 1.5406 \text{ \AA}$) was utilized to investigate the phase structure of raw materials, sintered PCD and oxidized production through a continuous scan mode with speed of $4^{\circ}/\text{min}$. Scanning electron microscopy (SEM, JEOL JSM-7001 F) equipped with an energy dispersive x-ray spectroscopy (EDS) was carried out to check microstructure and chemical composition of the annealed samples. The weight loss was measured before and after the annealing treatment.

The wear tests were conducted in cutting granite based on a CS6140 A screw-cutting lathe as shown in Fig. 1(a) [29,30]. In order to fix the rock core during the test, a special split cylinder and a sleeve were made on the lathe. Meanwhile, a drill chuck shape like special workholder was also fixed in the lathe for sample. The sample showed right circle cylindrical geometry with 4 mm in height and 3 mm in diameter. The bottom surface of the samples were polished by diamond abrasive sandpan with the surface roughness of 0.30–1.0 μm . The granite with a cylinder size of $\Phi 75 \times 400 \text{ mm}$ came from Miyun District, Beijing, which was characterized by stable properties, high hardness and abrasivity. The granite was fixed in the chuck of the special workholder, approached to the rock core surface and was longitudinally cut at a set penetration depth. After the sample was fixed on the special workholder, the polished bottom side of the sample was used to cut the granite by vertical direction. The relative position of the sample and granite can be seen in Fig. 1(b). The water lubrication condition was given during the wear test. To measure wear loss of samples during the tests a Sartorius type balance was used. The cutting parameters are rotation frequency $\nu = 355 \text{ r}/\text{min}$, penetration depth $a_p = 0.25 \text{ mm}$, tool feed $f = 0.026 \text{ mm}/\text{r}$. The wear rate I was defined as the rate between the mass wear of the rock destruction element Δm and the drifting for a tool path length element L with a value of 200 mm,

$$I = \Delta m / L \quad (1)$$

$$\Delta m = m_{in} - m_f \quad (2)$$

$$L = \pi D n t / 60 \quad (3)$$

Where m_{in} and m_f were the sample masses before and after testing, respectively, D was the core diameter, n was the rotation frequency of the work spindle, t was the cutting time. Each test in the experimental

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