#### Carbon 116 (2017) 103-112

Contents lists available at ScienceDirect

## Carbon

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





Carbon

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#### ARTICLE INFO

Article history: Received 19 August 2016 Received in revised form 26 January 2017 Accepted 27 January 2017 Available online 31 January 2017

Keywords: Polycrystalline diamond compact Tribological property Annealing temperature Ambient air Si<sub>3</sub>N<sub>4</sub> ball

#### ABSTRACT

Tribological properties are the significant engineering factors to evaluate the quality of sintered polycrystalline diamond compact (PDC). An effective approach to control the tribological properties of PDC is extremely desired for engineers. The current paper has systematically investigated the tribological properties of PDC in relation to annealing temperatures. The friction coefficients and the wear rates of PDC and Si<sub>3</sub>N<sub>4</sub> balls were evaluated by the morphologies and chemical conversions on tribological surfaces. Carbonaceous transfer films, induced by tiny diamond grains (TD grains, sizes of  $0-5 \mu$ m), can effectively reduce the friction coefficients of the annealed PDC. The wear of the annealed PDC is caused by the extraction of medium size diamond grains (MD grains, sizes of  $5-15 \mu$ m). The wear of Si<sub>3</sub>N<sub>4</sub> balls is attributed to the abrasion effect of TD grains and the cutting action of the MD/BD (big size diamond with sizes of ~25  $\mu$ m) asperities. It is specially noted that a markedly enhanced wear resistance of PDC can be achieved by the annealing treatment at 750 °C. These results would give significant instructions for regulation and control of the tribological properties of PDC by simple and low cost annealing treatment. © 2017 Elsevier Ltd. All rights reserved.

### 1. Introduction

Polycrystalline diamond compact (PDC), an ultrahard composite diamond related material, has been widely applied in machining tools, thrust bearings and drilling bits due to its perfect properties such as high hardness, excellent wear resistance and good impact toughness [1-3]. Tribological properties are significant engineering factors for evaluating the quality of PDC tools. In the past decades, various techniques for regulating and controlling the tribological properties of PDC have been extensively investigated by many scientific researchers and engineers. Most of the previous efforts have been devoted to improve the tribological properties of PDC and other diamond related materials by optimizing the chemical states and physical structures. Qian et al. [4] sintered polycrystalline diamond (PCD) with the replacement of metallicbinders such as cobalt by inorganic binders such as magnesium carbonate. The PCD with binder of magnesium carbonate behaved a higher wear resistance than that of the PCD with cobalt binder,

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which was ascribed to the higher thermal stability and chemically inert of magnesium carbonate. The refinement method of diamond grains had also been utilized to enhance the wear resistance of diamond tools in the previous researches [5,6]. Konicek et al. [7] explored the origin of ultralow frictional properties of the ultrananocrystalline diamond and they confirmed that the dissociative passivation either by H<sub>2</sub>O or H<sub>2</sub> was responsible for the lower friction coefficients and wear rates. Detonation nanodiamonds (ND) and epoxy-ND composites had hold great potentials to improve tribological characteristics of composites, and it was recently confirmed by Neitzel et al. [8,9]. Special efforts were devoted to diamond like carbon (DLC) and metal-doped DLC coatings. DLC coatings can acquire the greatly low friction coefficients because of its low shearing properties [10,11], which could be acted as a solid lubricant coating covered on other diamond related materials. The doped metal elements had a markedly effect on modifying the microstructures and reducing the friction coefficients of DLC layers [12–14]. Therefore, depositing the DLC and metal-doped DLC coatings were the practical approach to control the frictional property of diamond related materials. Recently, Dwivedi et al. [15] have discovered that the film-substrate interface engineering could effectively control the friction and wear



behaviors of ultrathin carbon film, which were determined by the integrated framework of surface passivation, rehybridization, material transfer and tribolayer formation. Another new effective measure, sintering the PCD with a superhard cutting insert of chemical vapor deposition (CVD) diamond, was designed for manufacturing cutting and drilling tools, which greatly strengthened their abrasion resistance [16,17]. However, these modified technologies were confronted with a common issue that was high cost and high manufacturing difficulty.

Annealing treatment, a conventional method with the advantage of convenience and high efficiency, has been widely used to improve the mechanical properties of engineering materials, especially for metallic materials. For diamond related materials, some previous efforts were devoted to the DLC and CVD diamond. Wang and Tokuta et al. [18,19] found that the pre-heat treatment above 500°C could cause the structural changes and the formation of graphite layer on DLC surface. The lower shearing resistance was obtained from the graphite layer between the sliding surfaces. Unfortunately, the super low friction coefficient for these diamond materials was always at the sacrifice of the wear resistance under dry conditions, which may be ascribed to the graphitization and reduction of hardness [18,20,21]. Khomich and Ralchenko et al. [22,23] have studied the effect of high annealing temperature on structures of CVD diamond films under vacuum condition  $(10^{-5})$ torr). It was showed that the allotrope content was determined by the annealing temperatures. Graphite and microcracks appeared at the annealing temperature above 1300 and 1575 °C, respectively. However, the tribological properties of the annealed CVD diamond were not further explored. Yu et al. [24] recently indicated that the surface graphite induced by vacuum heat treatment could reduce the friction coefficient of CVD diamond, whereas the microcracks caused by vacuum heat treatment resulted in a serious wear.

Until now, the approach to controllable tribological properties of sintered PDC through annealing treatment has not attracted the attention of researchers. It may attribute to previous studies on the thermal stability of PDC. As the previous references reported [25,26], some tiny spalling pits without chemical conversions appeared on PDC surfaces at the annealing temperature below 500 °C. With further elevating the annealing temperature (600-800 °C), the microcracks, surface graphitization, oxidation and extrusion of Co phase appeared. Wang et al. [27] heated the PCD layer in air and nitrogen atmosphere at 700 °C for 10 min and then studied its machinability. It revealed that microcracks caused by thermal stress greatly reduced the wear resistance. Deng et al. [20] further verified that the microcracks, oxidation and extrusion of Co phase were detrimental to the wear resistance of PDC under the in situ high temperature conditions (600-700 °C). Hence, it seemed that the tribological property of PDC was unlikely to be promoted by a pre-heat treatment, especially at high temperature. However, some contrary results have been achieved in present works. The tribological properties markedly depend on annealing temperature and the pre-heated PDC samples under a higher temperature (700 and 750 °C) will have a perfect comprehensive tribological property, with a relatively low friction coefficient and nearly undetectable wear.

This paper provided a systematic work on the tribological properties of PDC samples depending on the annealing temperatures in ambient air. Special efforts had been devoted to understand the effects of tribological surface characteristics on the tribological properties of annealed PDC samples. The mechanisms of the formation of transfer film, and the wear mechanisms of PDC samples and  $Si_3N_4$  balls were particularly discussed. It would significantly instruct the engineers to regulate and control the tribological properties of PDC by the annealing treatment for industrial applications.

#### 2. Experimental details

#### 2.1. Characteristics of materials

PDC was sintered at Zhongnan Diamond Co., Ltd. It consists of a cemented carbide substrate (WC-16wt%Co) and a sintered polycrystalline diamond (PCD) layer. The PDC, as shown in Fig. 1b, was cut into  $1 \times 1$  cm<sup>2</sup> samples by wire-electrode cutting. Fig. 1a illustrates that the total thickness of PDC sample is 3.7 mm with 3.2 mm of WC-Co substrate and 0.5 mm of PCD layer. The previous results [26] indicated that the PCD layer was mostly composed of diamond, Co and WC phase (Fig. 1c). Fig. 2a specifically shows that the big size diamond grains (BD grains, sizes of ~25 µm), medium size diamond grains (MD grains, sizes of  $5-15 \mu m$ ) and tiny diamond grains (TD grains, sizes of  $0-5 \mu m$ ) are compactly distributed on PDC surface. Moreover, some tiny holes and Co binder are enriched among those diamond grains, especially among the MD and TD grains. According to references [28,29], the diamond grains were bonded by carbon-carbon bonds, which insured the sufficient stability and strength of PCD layer. Surface topography of PDC and the corresponding EDS mapping images can be found in Fig. 2. They further confirm that the content of W is negligible. Moreover, the Si<sub>3</sub>N<sub>4</sub> balls, with a diameter of 6 mm and surface roughness of 15-20 nm, are selected as counterparts in tribotests.

### 2.2. Annealing tests

The PDC samples, ultrasonically rinsed with acetone and alcohol for 30 and 15 min, respectively, and then they were annealed at temperature from 200 to 750 °C in a SX-8-10 muffle furnace for 30 min in ambient air. The temperature was detected by a thermocouple with a deviation of  $\pm 20$  °C. Then the annealed PDC samples were cooled with the process of air-cooling.

#### 2.3. Ball-on-disc tribotests

The tribotests were performed on a CSM ball-on-disc rotation tribometer in ambient air (relative humidity,  $35 \pm 3\%$ ). The Si<sub>3</sub>N<sub>4</sub> ball was the upper sample and the PDC acted as a disc. Prior to the tribotests, both Si<sub>3</sub>N<sub>4</sub> balls and annealed PDC samples were firstly ultrasonically rinsed with acetone for 30 min, and then with alcohol for 15 min. During the tribotest, the Si<sub>3</sub>N<sub>4</sub> ball was fixed, while the PDC sample was rotated with the rotation radius of 2.5 mm and



**Fig. 1.** Optical images and composition of PDC: (a) cross section image of PDC, (b) image of PDC, (c) XRD pattern of PDC surface. (A colour version of this figure can be viewed online.)

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