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## Tribological properties of partly polished diamond coatings

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#### **Abstract**

Extremely low friction coefficient was achieved with "partly polished diamond coatings". Diamond coatings were deposited onto Si substrates by MWCVD with the mixture of  $CH_4$  and  $H_2$ . Deposited films were characterized by X-ray diffraction (XRD), Raman spectroscopy and Electron Spectroscopy for Chemical Analysis (ESCA). Sharp peak derived from polycrystalline diamond was observed by XRD. Whereas Raman profile of partly polished diamond coatings was close to that of ta-C. This result suggests that small diamond grains were surrounded by amorphous carbon structure in the diamond coatings. Deposited diamond coating was polished with each other. Surface roughness  $R_a$  was reduced to 0.3, 0.2 and 0.08  $\mu$ m, respectively. The hardness of the polished diamond coatings investigated by Nanoindentation technique was approximately 40.8 GPa, which was relatively lower value compared with conventional as-deposited CVD diamond coatings. For the tribological properties, we examined the effect of surface roughness using flat-ended pin-on-disk apparatus and ball-on-disk apparatus with bearing ball (SUJ2) and stainless steel (SUS304). Diamond coatings were deposited onto flat-ended pin and disk, and they were polished to  $R_a$ =0.3, 0.2 and 0.08  $\mu$ m. After the 6000 cycle process extremely low friction coefficient,  $\mu$ =0.05, was achieved with the pair of  $R_a$  (flat-ended pin, disk)= $R_a$  (0.08, 0.3) in flat-ended pin-on-disk apparatus. In order to clarify the effect of surface roughness, ball-on-disk was carried out with different surface roughness,  $R_a$ =1.7, 0.3, 0.2 and 0.08  $\mu$ m. Here as-deposited diamond coating,  $R_a$ =1.7  $\mu$ m, was used as a reference point. Friction coefficient of  $\mu$ =0.09 was obtained for both balls. After the tribological tests balls were analyzed by scanning electron microscope (SEM) and energy dispersed X-ray spectrometer (FDX)

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Keywords: Friction; Polishing; Tribology; Coatings

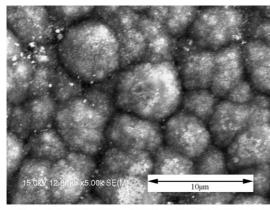
#### 1. Introduction

Since the first breakthrough in chemical vapor deposition diamond coatings, many research works have been done on CVD diamond coatings about their excellent properties such as high hardness, low friction good thermal conductivity and wide band gap structure. For the viewpoint of industrial applications, artificial diamond is used widely for cutting tools. However, a major impediment for their practical application lies in the limitation of the substrates and their rather high hardness. The former problem is being solved by special technique such as the treatment of substrate materials [1] or appearance of machinable substrates, Ti<sub>3</sub>SiC<sub>2</sub> [2]. In order to overcome the latter problem we developed easily polishable diamond coatings. The investigated diamond coat-

ings are categorized as ballas diamond coatings which was first discovered by Fischer [3]. For ballas type diamond films, much research work also has been done in elsewhere [4–7]. Ballas is nearly pure diamond with nano-crystalline structure [8]. But these nano-crystalline diamond grains are connected with each other through a graphitic or amorphous carbon structure, which make it possible to achieve relatively smooth surface by polishing.

The purpose of the study is to investigate the basic properties of easily polishable diamond coatings and their tribological properties. Diamond coatings were prepared on Si and machinable substrate as we mentioned above [2]. Si and cylinder shaped substrates were used as disk and flat-ended pin, respectively. Then, coated diamond was polished properly. The tribological properties were evaluated using ball-on-disk type tribometer under ambient air at room temperature conditions. We carried out tree types of experiment in order to know how following effects contribute the tribological properties; speed, surface roughness and long distance in tribo-

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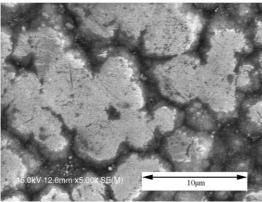


Fig. 1. SEM images of as-deposited (top) and partly polished diamond coatings (bottom). These coatings were deposited onto Si substrate.

testing. The diamond coated disk and pin, and ball were chemically analyzed after the tribo-testing.

#### 2. Experimental

Diamond coatings were deposited onto Si and  $Ti_3SiC_2$  substrate by Microwave Plasma Chemical Vapor Deposition (Nihon Koshuha Co., LTD.) using the mixture of methane (2%) and hydrogen gas (98%). Before deposition diamond seeding was done and substrates were cleaned ultrasonically in ethanol. We fixed deposition time as 8 hours and thickness is around 6  $\mu$ m. Surface roughness of as-deposited films was 1.7  $\mu$ m. Diamond coatings were polished each other until  $R_a$ =0.3, 0.2 and 0.08, here  $R_a$  is arithmetic average roughness.

For structural characterization Raman spectroscopy (Jobin Yvon LabRAM HR-800, He-Ne laser, 20 mW, ambient air at room temperature), X-ray diffraction (Pan Analytical X'pert MRD), Electron Spectroscopy for Chemical Analysis (ULVAC PHI, Quantum 2000) and Scanning Electron Microscope (Hitach High-Technologies, S-4700 equipped with Energy dispersive X-ray Spectrometer) were performed.

Friction tests were performed at room temperature under ambient air condition using rotation type pin-on-flat tribometer manufactured by CSM Instrument. We used flat-ended pin and ball for diamond vs. diamond and ball vs. diamond tribotesting, respectively. The Ti<sub>3</sub>SiC<sub>2</sub> was used for the pin. The shape of the pin is circular cylinder and its diameter is 10 mm. We deposited the diamond film in the area of flat part. The

diamond on flat-ended pin was also polished until  $R_a$ =0.3 and 0.08  $\mu m$ .

#### 3. Results and discussions

#### 3.1. Structural characterization

The surface of as-deposited and polished diamond coatings was observed by SEM (see Fig. 1). One can see that deposited coatings are classified into course ballas diamond [9]. Ballas type shows the crystalline diamond areas in micro twinned matrix. For the structural evaluation of fabricated diamond films, Fig. 2 shows the Raman spectra of as-deposited and polished diamond coatings. The results clearly demonstrate the mixture of diamond and diamond-like carbon structure in the deposited films. Raman profile was fitted by four Gaussian functions. We obtained close spectra between both coatings. One peak centered at  $1333 \pm 1.3$  cm<sup>-1</sup> corresponds to the diamond peak, which was relatively weak. The broadening peak indicates the existence of micro- or nano-size of diamond grains [10]. The other peaks can be found at  $1130\pm1.6$ ,  $1360\pm3.0$  and  $1541\pm1.4$  cm<sup>-1</sup>. The peak at the lowest frequency corresponds to nano-crystalline diamond. Other two peaks correspond to diamond-like structure, which is well-known disordered peak (lower frequency) and graphitic peak (higher frequency) [11]. X-ray diffraction was performed for the evaluation of preferred orientation in crystal and crystal size on diamond films. Four clear peaks were obtained, which represents diamond (111), (220), (311) and (400) reflection. The grain size was calculated by the Scherrer's formula and estimated as 45.9 nm. Besides, the hardness and Young's modulus are 40.8 and 540.2 GPa. Furthermore, the results on depth profile of ESCA analysis for C1s peak indicate the no clear difference was observed in our coatings compared to previous report [4].

#### 3.2. Tribological properties between diamond coatings

In order to estimate the proper rotation speed in tribological testing, we examined the effect of rotation speed for friction

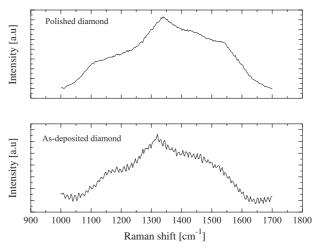


Fig. 2. Raman spectra of diamond coatings. The spectra were superpositioned by several Gaussian functions.

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