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High-pressure, high-temperature plastic deformation of sintered diamonds

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ABSTRACT

The strength of polycrystalline diamond (PCD) was investigated through a high pressure (P) and temperature (T) deformation experiment. Prior to the deformation experiment, two bulk samples were sintered back to back under identical conditions with two different precursors, which shared identical initial grain size distribution. Precursor of one sample (2E) had lower concentration of crystalline defects than that of the other sample (1S). In crushing strength tests, precursor of 2E exhibited an enhanced crushing strength compared to that of 1S. During the high P–T deformation experiment, the two samples were stacked back to back and deformed together. Their mechanical properties were investigated in situ using synchrotron X-ray diffraction and imaging in the deformation DIA apparatus. The strain data based on imaging showed that the two samples were deformed at identical strain rates (ca. 1.5×10^{-5} s^{-1}) and analysis based on lattice plane distortions in the diffraction patterns showed that sample 2E exhibits marginally higher strength than that of sample 1S. The X-ray data indicate that, upon deformation, larger elastic lattice strain builds up within the grains in sample 2E, indicating greater strength at grain-to-grain level in this sample. In addition, lower micro-stress levels are evidenced upon hydrostatic loading of sample 2E, strongly indicating a better sintering state than that in sample 1S. We interpret these differences as due to a lower defect concentration, particularly on the grain surfaces of the precursor powder, that results in stronger diamond–diamond bonding. Altogether, these data suggest that the use of diamond grains with reduced defect concentration and cleaner surfaces as precursors will likely improve the wear resistance of the resulting PCD via stronger individual grains and better sintering.

1. Introduction

Diamond is the stiffest and hardest material known. Years of technical development for synthesizing large sintered polycrystalline diamond (PCD) parts has made diamond a material of choice for various types of machining tools and engineering processes. One example of such applications is oil and gas drilling, for which it is known that PCD bits encounter the greatest wear. Comparative studies available on PCD's mechanical properties [1,2] show that PCD surpasses by far all other materials typically used for similar applications, such as WC.

The performance of PCD bits is essentially determined by their ability to resist fracture under loading. During a down-hole impact event, the PCD bit absorbs impact energy, by deforming under the applied load. PCD bits with higher strain energy capacity will be able to absorb more impact energy during drilling without exceeding the diamond yield strength or fracture toughness limit [3–7]. Therefore, the strength and the fracture toughness of the PCD are the main properties that control its wear resistance and durability. The failure mode of the PCD bits (intra-granular vs. inter-granular fracture) is not well understood, although it is known that it depends on the concentration of crystal defects in diamond powder precursors as well as on the quality of the sintering, i.e., the strength of the diamond–diamond bonds.

The elastic properties of single crystal diamonds and PCD's have been studied extensively in the literature [8–11]. The strength of PCD's, however, remains poorly documented, essentially due to the difficulty to deform diamond plastically at ambient conditions [12,13]. A way to access plastic deformation of diamond is to subject it to high temperature, where ductility is enhanced [12]. However, this requires high pressures in order for the diamond to remain thermodynamically stable and not convert to graphite. In this context, we have performed a high pressure (P) and temperature (T) deformation experiment in which the mechanical properties of PCD samples were investigated in situ using synchrotron diffraction and imaging techniques. During this experiment, two samples were deformed together, under identical P–T conditions. The samples, extracted from PCD cutter bits for oil and gas drilling, were sintered from two different monocrystalline diamond powder precursors sharing identical particle shape and size distribution.

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However, for one sample, 1S, a standard diamond powder (unprocessed for the reduction of crystallographic defects) was used, whereas for the other one, 2E, the precursor powder was processed through mechanical, thermal and chemical steps to reduce the concentration of crystallographic defects in the grains (as described in the US patent #8,734,753 [14]). This results in a greater mechanical strength of the individual grains as evidenced by the crushing strength test results (see Section 2.1 for details), which in turn may be responsible for enhanced wear resistance of the PCD. In the present study, we report characterization of the two precursors and investigate how the difference in the precursors impacts the quality of the sintering and bulk strength of the PCD.

2. Experimental protocol

2.1. Starting materials

Two different diamond powder precursors were used to sinter the PCD cutters 1S and 2E. Both diamond powders share similar particle shape and size distribution. A relatively new technique, dynamic image analysis [15], was used to characterize the two diamond powders with respect to particle shape and size distribution (Table 1).

The majority of the particles exhibit 3-D blocky shapes, ranging in size from 22 to 36 μm, as shown by Scanning Electron Microscopy (SEM) characterization (Fig. 1).

Fig. 2 shows Fourier-Transform Infrared (FTIR) [16,17] spectra of the precursors for 1S and 2E. The spectra are consistent with type Ib diamonds (typical of synthetic diamond), where N defects are primarily present in the form of single substitutional atoms [18], rather than aggregated clusters [19]. These defects result in the sharp absorption band at 1130 cm^{-1} and the broader one at 1340 cm^{-1} [20,21]. The absorptions at 1488 cm^{-1} for 2E and at 1458 cm^{-1} for 1S might be indicative of H related defects [22]. For this latter sample, the absorption band at 1372 cm^{-1} indicates additional H defects although, for both samples, the absence of the characteristic H absorption band at 3017 cm^{-1} precludes the presence of a significant amount of H defects [23].

A full characterization of the nature of the defects (including dislocations and twins) and their concentration in these powders would require a detailed study using transmission electron microscopy (TEM). Nevertheless, we note that the lower baseline transmittance observed in the starting powder used for 2E suggests a lower concentration of crystallographic defects [16,24]. FTIR transmittance is related to elastic light scattering due to crystallographic defects such as dislocations, stacking faults, twins and grain boundaries — the higher the concentration of these intrinsic defects, the lower the baseline transmittance (see [25,26] and references therein). This is consistent with the thermal treatment consisting in a high temperature (≥700 °C) water vapor etching [14] — applied to powder 2E. In most materials, indeed, annealing is expected to reduce the concentration of intrinsic defects, resulting in an improved infra-red transmittance [27]. Variations in baseline transmittance may also be caused by changes in the surface conditions. However, due to the etching treatment of 2E, the surface roughness of this latter sample is expected to be larger (due to the presence of additional etch pits [14]), which, if significant, would result in a lower transmittance. Since the baseline transmittance difference observed is the opposite (i.e., larger for sample 2E), we infer that it is the consequence of a lower intrinsic crystal defect concentration [28].

Table 1
Results of dynamic image analysis. Particle sizes are in microns.

Particle shape	1S	2E
Total particle count	~5000	~5000
Average aspect ratio	0.717 ± 0.005	0.712 ± 0.0009
Particle size (equivalent spherical diameter)		
5%	23.3 ± 0.5	22.48 ± 0.02
50%	27.6 ± 0.5	26.9 ± 0.1
95%	32.7 ± 0.5	31.9 ± 0.1
99.9%	38.4 ± 0.9	36.9 ± 0.3
Maximum size	46.2 ± 2.9	39.7 ± 1.7

However, FTIR data cannot be used to infer surface defect concentrations, which may be different in the two samples, as suggested in Fig. 1 by additional charge accumulation at the grain surfaces of 2E. Time-of-Flight Secondary Ion Mass Spectroscopy (ToF SIMS) analyses are summarized in Fig. 3. Spectra were collected for 5 min over a 100 μm^2 area with a sampling depth up to 0.5 nm. These results thus reveal impurities mostly on the surface of the diamond grains. As shown in Fig. 3a, all the data collected in negative mode seem to indicate that 1S contains higher concentration of O and H impurities. The ToF SIMS is operated under high vacuum conditions, at pressures <1 kPa, which ensures that little to no background atoms contribute to the measurement. However, since both samples contain very little H defects (as shown by the FTIR data), the corresponding low detector counts obtained here may be influenced by the minute amounts of residual hydrogen gas present in the chamber during the analysis, which may explain the disagreement between data in positive and negative mode regarding H. The present data can therefore not be interpreted in terms of relative H defect concentration. In positive mode (Fig. 3b), aside from the H peak, only data corresponding to an atomic mass number of 28 (i.e., N$_2$) show higher concentration in 2E than in 1S. The fact that all other peaks are higher in 1S than in 2E indicates that sample 1S contained more surface impurities than 2E. The integrated number of counts over all channels in positive mode is much larger for sample 1S, indicating larger impurities (including N defects) concentrations. These observations, together with the FTIR data presented in Fig. 2, thus convincingly demonstrate that precursor for 2E contained lower defect concentrations with cleaner surfaces than precursor for 1S.

The mechanical strength of these powders was determined through crushing tests (see US Patent 7,275,446 for a description of the apparatus and technique used). The Crushing Strength Index (CSI) — the percentage of “on size” particles (particles between 50% and 95% of the number distribution) in the crushed and uncrushed diamond powder — was used to evaluate the mechanical strength. Three trials were run on each of the powders used in 1S and 2E, the average values and corresponding standard deviations, σ, are 37 ± 0.3 and 44 ± 4.1%, respectively (see Table 2). The lower standard deviation in 1S shows a better reproducibility. It is believed that the processing of powder 2E results in the presence of some grains with drastically increased fracture toughness. The proportion of such grains may vary from test to test causing the larger standard deviation observed here in powder 2E. In any case, since all three tests indicate larger CSI value, it is clear that the process utilized to reduce the defect concentration within and at the surface of the grains somehow results in an enhanced average toughness of the diamond grains in 2E.

The PCD cutters, designated as 1S and 2E, were sintered simultaneously, back to back, under the same P–T conditions (above 6 GPa and 1400 °C). In this process, the diamond precursor powder is placed on a sintered Co and WC substrate, from which W and Co diffuse into the diamond layer, forming diamond–diamond bonds [3]. Cylindrical PCD samples, having 0.8 mm in diameter and 0.4 mm in length were then laser cut perpendicular to the diamond table of the PCD cutters.

2.2. Experimental details

We present here the results of one deformation experiment, where these cylindrical samples, from PCD cutters 1S and 2E, were stacked and deformed together (Fig. 4). The experiment was carried out using the deformation-DIA (D-DIA) apparatus installed at beamline 13BM-D of the Advanced Photon Source (APS, located at Argonne National Lab., Illinois, USA). This experimental setup has been extensively used for rheology studies at high P–T [29–31]; experimental details can be found in the literature, e.g., [32,33]. The D-DIA is a cubic type multi-