QUANTITATIVE INVESTIGATION OF GRAPHITE AND DISLOCATIONS IN PCD SINTERED AT ELEVATED PRESSURE

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Polycrystalline diamond (PCD) is produced during a high-pressure high-temperature process (5.5 - 7.7 GPa and 1350-1700°C) from micron sized diamond powders bonded by cobalt acting as a sintering aid. This material is used extensively in abrasive applications[1]. During the sintering process diamond grains in contact with each other will undergo plastic deformation at temperatures above 770C. The degree of plastic deformation will depend on the temperature and the localised stress acting on the diamond grains. Diamond grains not in contact are subjected to low pressure during the sintering process leading to formation of graphite along the surface resulting in a tool less resistant to abrasive wear[2]. At temperatures above 1300C the cobalt metal melts and infiltrates into the plastically deformed diamond to redistribute carbon via liquid phase sintering (LPS). The aim of this study is to investigate the dislocation structures and formation of graphite in polycrystalline diamond materials sintered with different processing conditions.

Diamond powders were sintered at pressures of 5.5-15 GPa and temperatures of 1500-2300C: (1) without cobalt added (binderless), (2) with 2 vol. % controlled cobalt addition (admix) and (3) via infiltration from a cobalt layer (infiltration). Raman spectroscopy was used to investigate the graphite content and full width half maximum (FWHM) of the diamond peak (1333 cm^{-1}) - an indication of the degree of plastic deformation. FIB SEM was used to prepare thin sections of the material to be studied in the TEM using annular dark-field (ADF)-scanning (S)TEM, bright-field BF, DF and electron diffraction.

The Raman spectroscopy results of the FWHM and degree of graphitization are shown in Table 1. The binderless samples had significantly higher degrees of plastic deformation. The large peak width for the 10 GPa and 15 GPa samples was due to the higher sintering temperatures. Figs. 1 and 2 shows an BF image, with associated EELS maps, and ADF-STEM image of the diamond-diamond contact points for a binderless sample. Deformation twins are visible running parallel to the contact direction. The 10 GPa and 15 GPa contained no porosity hence having more direct diamond-diamond bonding area. Admixed samples had lower dislocation densities compared to the infiltration samples. Additions of cobalt is thought to act as a hydrostatic pressure medium during the plastic deformation stage of sintering, thus lowering the inhomogeneous stress distribution at the diamond-diamond contact points. Fig. 3 shows reprecipitated diamond areas surrounding the cobalt pool having lower dislocation densities than the plastically deformed diamond grain. Samples sintered via the infiltration method had a higher dislocation density at higher pressures, indicating that the diamond grains experienced a high stress and deformed to a greater extend, before the cobalt infiltration. Less cobalt was found to infiltrate when sintering at higher pressure consequence of more efficient diamond grain packing. In conclusion, sintering pressure and the method of cobalt introduction had a large effect on the resulting PCD microstructure.

References:

1. Pacella, M., et al. (2014) J. Mater. Proc. Tech 214.

2. Klocke, F. (2011) Manufacturing Processes 1: Cutting. Springer.

Table 1. FWHM of the Diamond Raman peak / Intensity ratio of the Diamond to graphite Raman peak.

Sintering	**Binderless**	**Admix**	**Infiltration**
5.5 GPa	18.6 ± 1.4 / 1.7 ± 0.1	*	14.4 ± 0.3 / No graphite
7.7 GPa	*	13.3 ± 0.9 / 2.2 ± 0.2	15.1 ± 0.2 / No graphite
10 GPa	56.7 ± 0.6 / 1.8 ± 0.1	*	16.6 ± 0.3 / No graphite
15 GPa	61.5 ± 1.9 / 2.7 ± 0.3	15.1 ± 1.6 / No graphite	19.5 ± 0.5 / No graphite

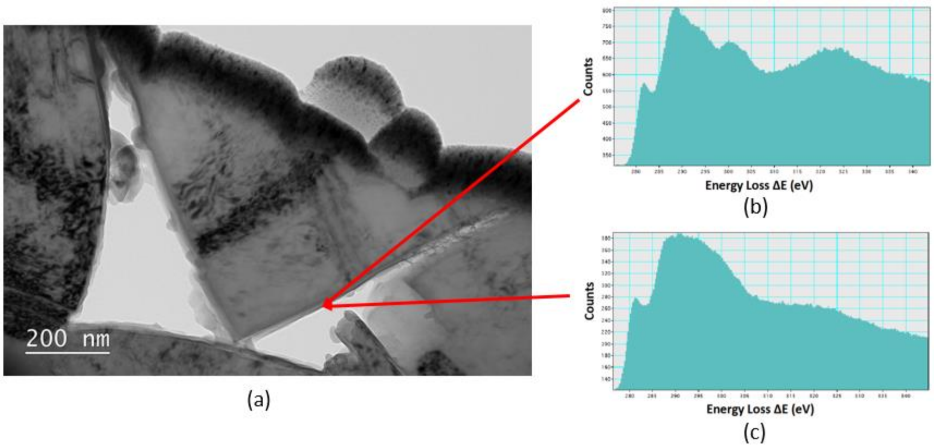

Fig.1. (a) BF image of binderless PCD with (b) graphite and (c) amorphous carbon EELS spectra.

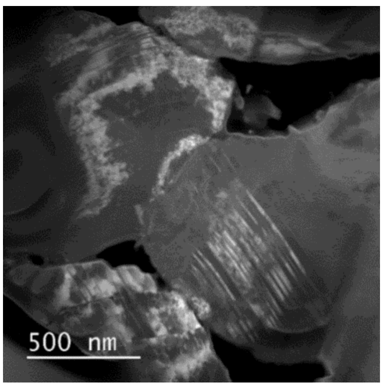

Figure 2. ADF-STEM image of diamond-diamond contact points formed in a binderless sample.

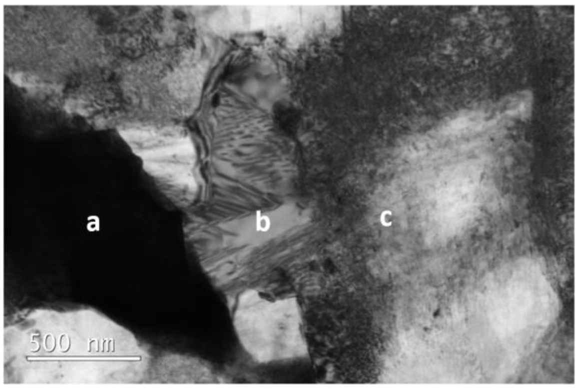

Figure 3. BF-TEM image of the infiltration sample showing: (a) residual cobalt metal pool, (b) reprecipitated diamond and (c) plasticallt deformed diamond.