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Microstructural Study on Toughness of Synthetic and Natural Polycrystalline Diamond using Microscope

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We observe microstructures of synthetic and natural polycrystalline diamond. Natural sample contains pores and cracks, and has not underwent strong shear stress that deforms diamond crystal plastically. Synthetic sample is compacted to be a poreless structure by plastic deformation.

Introduction

Diamond occurs naturally not only in single crystals but also in various sorts of polycrystalline mass. The former cleave rather easily along the ⟨111⟩ plane by suffering abrupt mechanical stress. On the other hand, the latter have greater toughness than single crystal, because random orientation of constituent single crystals breaks paralleism of cleavage planes between crystals. Therefore, polycrystalline diamond are very important in industrial applications. Many workers have studied to synthesize polycrystalline diamond of good quality[1–4]. We also successed to synthesize polycrystalline diamond by sintering diamond particles with cobalt under high pressure andi high temperature. Results of our experiments were reported in the previous paper[5].

The purpose of this article is to evaluate toughness of synthesized and natural polycrystalline diamond by microscopic observation of its microstructure. The microstructure of material is well known to reflect their physical property of toughness. Ordinary method for testing mechanical strength of material cannot be applied to diamond, since specimen shaping is very difficult.

Samples

Sintered polycrystalline diamonds are used as synthetic samples. The starting material of the aggregate were mixture of diamond crystals with the size of $\sim 100\ \mu m$ and cobalt powder of 3% by volume.

Natural samples are commercially available polycrystalline diamond brought from Zaire. Two samples of them are shown in Photo. 1. They are 1.5 to 2 mm in size, yellowish or greysh in colour and subtranspalent.

We observe fractured and polished surface of samples using an optical microscope and a scanning electron microscope. Polished surface is prepared by polishing the

Photo. 1. External appearance of natural polycrystalline diamond. Aggregate of yellowish diamond crystals (left) and greysh diamond crystals with inner spotted inclusions (right). (back ground: 1 sqares per mm)

aggregates using rotating diamond wheel at first and next lapping them with diamond paste less than 1 μm in diameter by hand. Fractured surface is made by crushing the aggregate with hammer. It is noted that synthetic aggregate are more difficult to break than natural one.

Microscopic observation

The types of microstructure focused in this study are pores, cracks and layered structure caused by plastic deformation. Sixteen samples are treated under microscope, and four pictures which show clearly characteristics of the microstructures are presented here.

Photo. 2 shows a fractured surface of natural polycrystalline diamond. In the figure cracks are visible. The surface observed is very smooth.

Photo. 3 shows polished surface of a natural sample. In Photo. 3, A and B denote pores and cracks respectively. Polished smooth surface is bounded by an outline like a tripetlous flower. This suggests that the sample was formed jointing three diamond crystallites, though grain boundaries separating them are invisible.

Photo. 4 shows a fractured surface of a constituent diamond of aggregation sintered at pressure of 5.5 Gpa and temperature of 1550°C for two minutes. In the photograph, we can easily recognize a layered structure of thin plates slipped along certain crystal planes. This is called deformation lamwllae.

Photo. 5 shows polished surface of a synthetic sample which was sintered with cobalt at 10 Gpa and 1800°C for two minutes. The crack runs through the grain

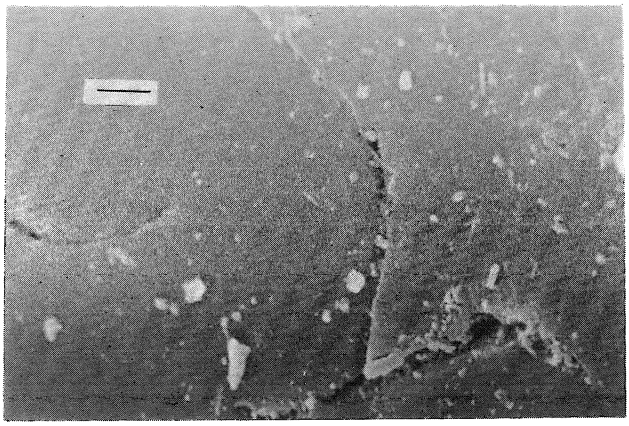

Photo. 2. Scanning electron micrograph of fractured surface of natural aggregate. Cracks can be seen, but surface is smooth. Fragmented very small diamond crystals are also visible. (bar=10 μm)

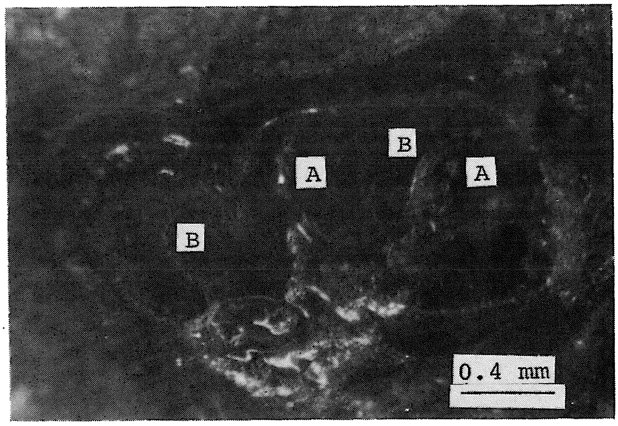

Photo. 3. Photomicrograph of polished surface of natural sample. Inner cracks and pores can be seen. A and B denote pore and crack respectively. Polished surface is bounded by an outline like a tripetalous flower.

boundary between the big crystal and the neighbouring aggregate. This crack was produced by applying mechanical stress after polishing. The parallel lines in the crystal can be identified as the deformation lamellae according to De Vries[6]. Formless dark material around the crystal represents cobalt used to enhance sintering efficiency, and bright material is aggregate of interlocking diamond crystals without pores. Such dencer structure is considered to be formed by plastic deformation in the process of sintering.

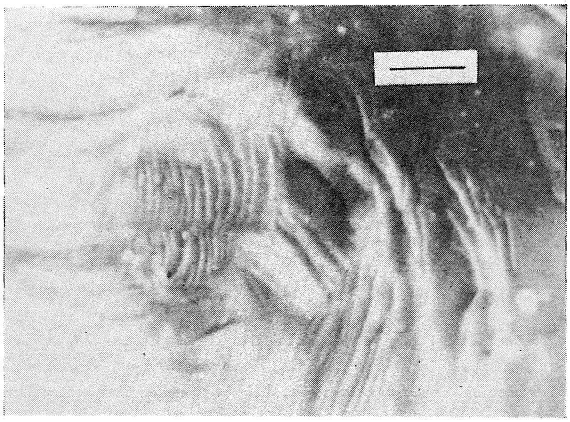

Photo. 4. Scanning electron micrograph of fractured surface of a constituent crystal in synthetic aggregate. Deformation lamellae can be seen. (bar=10 μm)

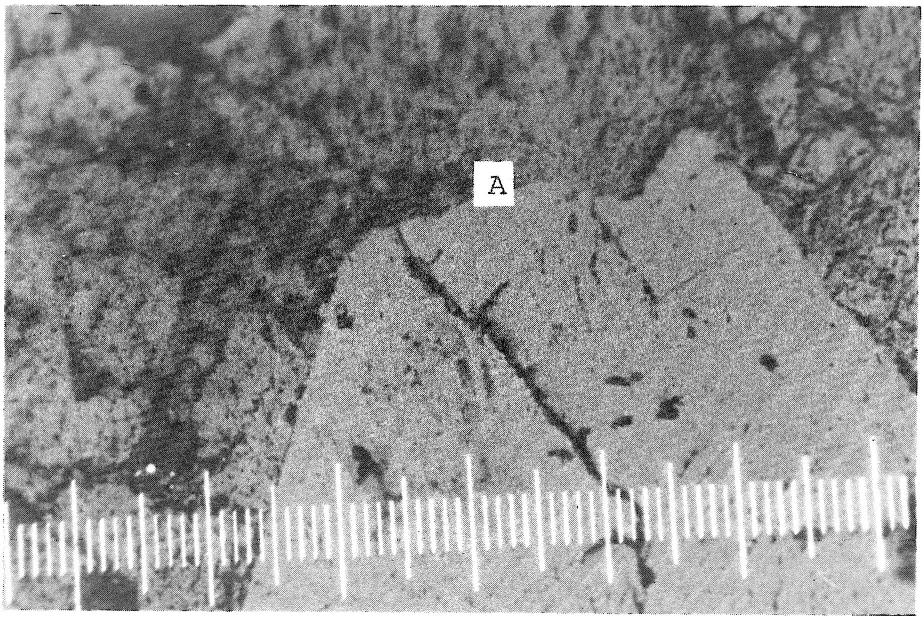

Photo. 5. Photomicrograph of polished surface of synthetic sample. At the place near A the place near A the texture is very uniform. Slip lines can be seen at the lower part of a big crystal. (1 div.=2.5 μm)

Applied pressure breaks a part of starting diamond crystals into small fragments, and they form porous aggregates between bigger crystals. Plastic deformation caused by pressure and increasing temperature squeezes the pores and forms denser structure.

It is noted that the texture near A in the photograph is uniform.

Discussion

Absence of chemical bond at the surface of pores cause reduction of toughness of material. The pores often induce cracks due to stress concentration to them. The cracks have larger ratio of surface area to volume than the pores. Then, existence of cracks spoils the toughness of material more significantly than the pores. Plastic deformation reduces total volume of cavity, increases total number of chemical bond at the grain boundary and so strengthen the toughness of the aggregate. Other important factor for sintering of powder is diffusion process, but in case of diamond the process hardly occurs because of its strongest caovalent bond.

The crack running transgranularly in Photo. 5 means that strength of chemica bonding at the grain boundary is comparable with that of C–C bonding in the diamond lattice. The texture near A is very uniform (Photo. 5). It seems to me that recrystallization of diamond crystal has occured at a place near A. Similar texture in CaF_2 has been illustrated as an example of grain growth[7].

Trueb and Butterman closely examined the microstructure of natural polycrystalline diamond, called carbonado[8]. They reported that carbonado is porous and contains impurities up to 20% by volume, and the constituents are fixed tightly with each other by diamond to diamond bondings.

Our synthetic aggregates contain cobalt of 3% by volume and poreless. These are also shown to have diamond to diamond bondings in the aggregate. Then, our synthetic aggregate excell carbonado in toughness.

We point out the importance of plastic deformation to obtain good quality of diamond aggregate.

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