Japan Geoscience Union Meeting 2010
(May 23-28 2010 at Makuhari, Chiba, Japan)

SMP056-P04 Room: Convention Hall Time: May 23 17:15-18:45

High pressure and high temperature synthesis of nano-polycrystalline diamond from various types of polycrystalline graph

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Nano-polycrystalline diamond (NPD) is a binder-less, pure polycrystalline aggregate of nano-diamonds, synthesized by direct conversion of graphite (e.g. Irifune et al., 2003). It has extremely high hardness equivalent to or even higher than that of single-crystal diamond. NPD consists of a mixed texture of a homogeneous fine structure and a lamellar structure, which are thought to be originated from different formation pathways (Sumiya et al., 2004). A recent study by our group (Ohfuji and Kuroki, 2009) showed that the graphite-diamond transition pathways and the resultant microstructures of NPD are strongly influenced by the initial structure characteristics of graphite. Here, we studied the influence of micro-textures and structures of polycrystalline graphite rods on the resulting textures of NPD through comprehensive analysis using Raman spectroscopy, X-ray diffractometry and electron microscopy. High pressure and high temperature synthesis of NPD was conducted at 16 GPa and at 2100-2200 K using a 3000 ton multianvil press. Four types of starting materials (polycrystalline graphite rods) from different companies: G1, G2, G3 and G4, were used.

Raman spectroscopic measurements of the four types of starting materials showed that they have various ratios of D band (1350 $_{cm}$-1) and G band (1580 $_{cm}$-1) intensities ranging from (D/G =) 0 to 0.32. Since the D/G intensity ratio reflects the crystalline state of graphite such as lattice defect density and relative ratio of sp2- and sp3-bonded carbons within each analyzed area (ca. 1 um), the observed Raman data suggests that graphite crystals with a variety of crystallinity coexist in a rod sample. This is probably related to the manufacturing method of graphite rods, in which relatively large graphite flakes (coke) are mixed with binder pitch, which will transform to fine graphite particles after baking treatment, to make rod shape. The Raman analysis revealed that the averaged crystallinity of graphite increases and lattice defect density decreases in the order G 3 < G2 < G1 < G4. It is, however, also worthy to mention that G2 and G3 have relatively higher frequency of large well crystalline crystals with significantly low D/G ratio. Crystallite size analysis by XRD and the Scherrer method also gave similar results.

TEM observations of recovered samples showed that grain sizes of nanodiamonds and the relative abundance are apparently different depending on the starting samples. NPDs obtained from G1 and G4 samples have grain size ranges of 50-120 nm and 30-70 nm, respectively, and contain only a small amount of lamella. On the other hand, NPDs obtained from G2 and G3 samples have grain size ranges of 40-90 nm and 30-70 nm, respectively, and contain a much larger amount of lamella. It appears that smaller the crystallite size of the starting graphite sample, smaller the individual grain size of the resultant NPD. Further details will be discussed in the presentation.