High Pressure Synthesis of Carbon Nanocrystals using Reduction Reaction between Supercritical Carbon Dioxide Fluid and Metal in Diamond Anvil Cell

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Abstract ()

Supercritical fluids combine the solubility of liquids with the diffusivity of gases and are widely studied as a technology that is both environmentally friendly and cost-effective. Among these, supercritical carbon dioxide can be handled under relatively mild conditions, with a critical point of T = 304 K and P = 7.38 MPa. Furthermore, because it is non-toxic and non-flammable, it is expected to serve as a new solvent to replace conventional organic solvents. However, much of the research on supercritical carbon dioxide have conducted in the MPa range, and virtually no research has been conducted in the ultra-high-pressure range of GPa. Therefore, in this study, we investigated the direct reaction between metals and supercritical carbon dioxide under ultra-high pressure using a laser-heated diamond anvil cell (LH-DAC).

To generate high pressure, a diamond anvil cell (DAC) equipped with a 450 μm diameter culet was used. A sample chamber with a diameter of approximately 150 μm was cut out of a pre-compressed stainless-steel gasket, and a metal piece shaped into a square of approximately 100 μm, a ruby for pressure measurement, and carbon dioxide were loaded into the sample room. The synthesis experiments were conducted in the pressure range more than 1 GPa. After pressurizing to the target pressure, the sample was heated by irradiating the metal piece with an infrared laser. The metal sample area was heated for 10–20 minutes under laser scanning irradiation and then rapidly cooled to room temperature. After decompression, the sample was recovered under atmospheric pressure, and the synthesized phases were identified using powder X-ray diffraction measurement.

The XRD patterns indicated that magnesium oxide was synthesized in the experiment conducted at 1 GPa. In contrast, in the experiment conducted at 10 GPa, magnesium carbonate was synthesized instead of magnesium oxide. Furthermore, SEM observation of the sample recovered from the 1 GPa experiment revealed fibrous crystals. Based on their size and shape, these crystals can be classified as nanotubes or nanofibers. Besides, Raman spectroscopy and SEM-EDX compositional analysis have revealed that these nanotubes and nanofibers are carbon-based crystals. Therefore, in this study carbon nanotubes and carbon nanofibers were successfully synthesized by reducing carbon dioxide with magnesium under high-pressure and high-temperature conditions.