Diamond and lonsdaleite ¹³C and ¹²C formation in a diamond anvil high-pressure cell

Kulnitskiy B.A., Blank V.D., Perezhogin I.A., Tyukalova E.V., Denisov V.N., Kirichenko A.N.

Technological Institute for Superhard and Novel Carbon Materials (TISNCM), 7a Centralnaya str.,
Troitsk, Moscow, 142190, Russia
boris@tisnum.ru

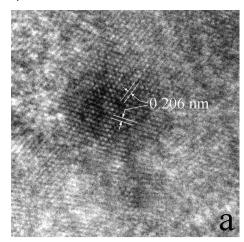
The behaviour of solids under extreme conditions is of great importance and interest for modern physics. A diamond anvil cell (DAC) is a device used in scientific experiments. It allows compressing a small (sub-millimeter sized) piece of material to extreme pressures.

This work was aimed at obtaining diamond from graphite in a diamond anvil cell (DAC) using high pressure and shear strain - a task that the science has so far mostly failed to implement,- and studying the features of this transformation by high-resolution TEM, EELS-spectroscopy, and Raman-spectroscopy, as well as comparing the different mechanisms of graphite-to-diamond transformation. Examinations were carried out using a JEM-2010 high-resolution transmission electron microscope and a JSM-7600F scanning electron microscope.

The particles of diamond and its allotrope lonsdaleite were prepared by graphite treatment in a high-pressure chamber made of diamond anvils (DAC), at room temperature and without catalyst. For greater reliability of the results, the experiments were made using ¹³C graphite, whereas the diamond anvils were made of ¹²C carbon isotope. ¹³C graphite was selected due to the fact that during processing, the anvil may slightly crumble and its dust particles can penetrate the sample. The synthesis was carried out under high pressures and shear deformation. Under some processing conditions, for the first time the carbon onions were obtained during graphite treatment in a diamond anvil high-pressure chamber under conditions of shear deformation at room temperature. It has been determined that the number of layers in the onions increases with the increase of pressure and shear values.

After treatment in the diamond chamber with a shear, a sequence of structures was observed depending on treatment conditions: hexagonal graphite - rhombohedral graphite - diamond or hexagonal graphite - onions - diamond. Along with diamond, some fragments and individual particles of lonsdaleite were found (a diamond allotrope different in packing). In the laboratory, lonsdaleite is prepared together with diamond in conditions of static and dynamic pressures and temperatures.

Diamond formation is confirmed by high-resolution TEM (Figure 1a), and by the EELS spectra (Figure 1b). The Raman spectra of the ¹³C showed that there was a weak broad band at 1265 cm⁻¹. Given that the position of the ¹³C diamond line is 1280 cm⁻¹, and the crystallite size reduction leads to its broadening and shift towards the lower wave numbers, we attribute the 1265 cm⁻¹ line to the appearance of diamond nanocrystals with a particle size of about 3-4 nm in accordance with the phonon-confinement model.



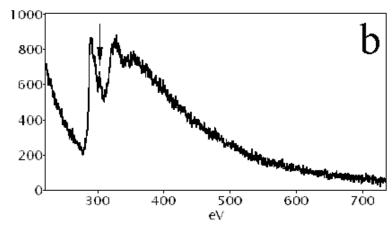


Fig.1a) A high-resolution image of a ¹³C diamond particle after high-pressure-shear deformation treatment of ¹³C graphite. Two {111} diamond planes are shown. The angle between these planes is about 70°. b) EELS spectra of a), the arrow indicates a peak corresponding to the diamond structure.