Formation, distraction and drying of the "secondary structure" in detonation nanodiamonds

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Disintegrated detonation nanodiamonds (DDND), though originally divided into individual diamond crystals (d = 5.2 nm) in the water dispersion, forms stable porous network ("secondary structure") as aqueous gel and dried powder [1].

In present study we attempted to follow formation and distraction of such a network by means of differential scanning calorimetry (DSC). The fingerprint of a network is a DSC peak corresponding to the melting of nano-sized water confined into the pores.

The network was not found in the original crude DND material. It appeared after an extensive acidic treatment of the samples at elevated temperature. Increasing the time of the treatment led to the diminishing of the size of voids filled with water. The minimum size (~6 nm) was close to the size of primary DND particles (d~5 nm). The smaller was the diameter of the voids in the "secondary structure", the smaller was the size of DND clusters in water dispersion made from the same material by ultrasonication.

The "secondary structure" disappeared in the DNDD samples subjected to high temperature – high pressure treatment (10 kbar, 870 K). Further increase of the temperature led to graphitisation of DDND. X-ray and SEM confirmed formation of a graphite phase.

Isothermal drying of aqueous gels DDND and DDN at temperatures 303–353 K was studied by means of thermogravimetry (TG) and DSC. The liquid was quantitatively removed from the samples. Two evaporation rates were observed for DDND at each temperature. The first corresponded to the evaporation of bulk water while the second – to nano-sized water. The amount of nano-sized water was 0.5 g per gram of ND. Parameters of the "secondary structure" account for the distinctive features of drying isotherms.

[1] Korobov M.V., Batuk M.M., Avramenko N.V., Ivanova N.I., Rozhkova N.N., Ōsawa E., *Diamond & Related Materials* **19**, 665 (2010).