



	<b>Experiment title:</b> 3D-XRD study of the orientation and size distribution of grains in mysterious, cryptocrystalline natural diamond- carbonado	<b>Experiment number:</b> ES-117
<b>Beamline:</b> ID11	<b>Date of experiment:</b> from: 09 April 2014 to: 13 April 2014	<b>Date of report:</b>
<b>Shifts:</b> 12	<b>Local contact(s):</b> Wolfgang Ludwig	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): Shiryayev AA Institute of physical chemistry and electrochemistry RAS, Moscow, Russia Senin RA Kurchatov Institute, Moscow, Russia Zolotov DA Institute of crystallography RAS, Moscow, Russia		

## Report:

In the current project a comparative study of several widely different natural and synthetic polycrystalline diamonds was performed using diffraction contrast tomography (DCT) and phase contrast tomography with the final aim to constrain mechanisms of formation of natural nano-polycrystalline diamond variety - carbonado.

Phase contrast tomography of carbonado samples from Central African Republic (CAR) revealed extensive porosity and inclusions, thus confirming previous observations. In addition to archetypal carbonado from CAR we have studied carbonado-like polycrystalline diamonds from Yakutian kimberlite pipes. Remarkably, phase tomographs of these samples are largely similar, though for kimberlitic material the porosity and amount of inclusions are smaller (fig. 1). One of hypotheses about carbonado formation invokes impact events. In our project several different specimens of impact diamonds from well-known Popigai crater (Siberia) were studied. The collection includes both (presumably) ejecta-related material (large black aggregates called yakutite) and small flakes recovered directly from impacted rocks. These samples markedly differ from carbonado and from kimberlitic specimens, since no porosity was observed even with 0.7 microns resolution. Instead, numerous cracks are present. In the flakes the cracks might reflect direct graphite-

diamond transformation. Note that this work is the first phase contrast tomography investigation of natural polycrystalline diamonds.

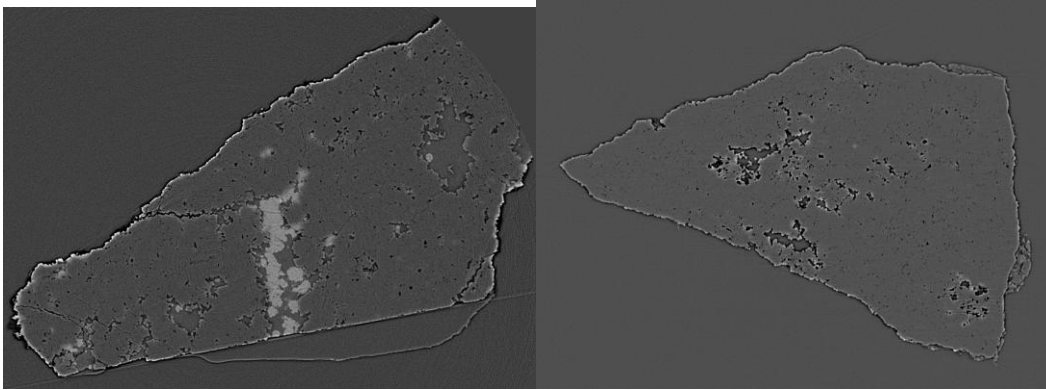


Figure 1. Phase tomographs of a carbonado from CAR (left) and of polycrystalline diamond from kimberlite.

3D-XRD data show that size of diamond crystallites in carbonado may differ considerably between specimens: whereas distinct diffraction spots are present in some cases, other samples show virtually continuous Debye rings. Small lattice parameter of diamond and relatively large specimen size (several mm) required some modification of experimental geometry for DCT measurements, namely, the diffraction patterns were collected at different sample-to-detector positions (far field and near field) and for the near field measurements the beam was shifted to the detector rim to allow measurement of 3 reflections. This procedure, however, limits number of accessible grains and requires some modification of data treatment. Detailed evaluation aimed at combination of near- and far-field data is currently in progress.

A set of synthetic polycrystalline diamonds was investigated as a reference. These samples were sintered at high pressures and temperatures (e.g., 5-6 GPa, 1300-1500 C) from diamond powder with grain sizes 10-20 microns with and without binders. The synthesis conditions are geochemically relevant, but the samples underwent very significant recrystallisation of the grains with dramatic decrease in sizes, making diffraction tomography impossible.