

## **Deformation-induced defects in diamonds: contribution of Small-Angle X-ray scattering and Atomic Force Microscopy**

A. Shiryaev (1), R. Gainutdinov (2), and Y. Fedortchouk (3)

(1) Institute of physical chemistry and electrochemistry, Moscow, Russian Federation (shiryaev@phyche.ac.ru, +74959554664), (2) Institute of crystallography, Moscow, Russian Federation, (3) Department of Earth Sciences, Dalhousie University, Halifax B3H 4R2, Canada

Diamond is a textbook example of brittle material. However, the absolute majority of natural diamonds bear traces of plastic deformation. Experimentally, plastic deformation of diamond at high temperatures was demonstrated long time ago (Evans and Wild, 1965). At geologically relevant pressures 4-6 GPa the brittle-to-ductile transition occurs at temperatures 1000-1300 °C (DeVries, 1975). Studies of deformation-induced defects are needed for reconstruction of PT and strain conditions of deformation of natural diamonds. Here we present results of investigation of diamond crystals deformed both in nature and in High-Pressure High-Temperature experiments using several advanced techniques: Small-Angle X-ray and neutron Scattering (SAXS and SANS) and Atomic force microscopy (AFM).

The SAS measurements were performed on high quality synthetic diamonds, intentionally deformed at 6 GPa and 1600 °C (Shiryaev et al., 2007, 2012; Shiryaev 2007) and on natural diamonds with various types of point and extended defects and different degrees of annealing in nature. Both synchrotron radiation (ESRF) and laboratory sources were used for SAXS; SANS was studied at ILL. Thermal annealing and/or mechanical deformation cause formation of nanosized planar and 3D defects giving rise to Small-Angle Scattering. The defects are often faceted by crystallographic planes common for diamond. The scattering defects consist of clusters of intrinsic and impurity-related defects; boundaries of mechanical twins also contribute. In some cases ordering of the defects in crystallographic directions is observed.

Atomic Force microscopy was employed for detailed investigation of surface relief produced both by experimental etching in various media (Fedortchouk et al., 2011) and in natural environment. The AFM studies of small diamond crystals are complicated by roughness of the faces, thus careful selection of the analysis region was needed. Contact and tapping modes of microscope operation were employed. In order to consistently investigate fine features various AFM tip geometries were used and the tips were frequently changed to compensate for blunting.

One of the most remarkable surface features detected by AFM on several diamonds were ridges up to several 10-20  $\mu\text{m}$  long and only few tens nm high. These features are not observable by high resolution optical microscopy and are very difficult to spot using SEM. Close examination of the ridges shape indicate that they represent elastic twins of different types (free and arrested ones). The observation of abundant microtwinning in natural diamonds on (sub)micron scale is rather unexpected and deserves further investigation. The most obvious implication of this phenomenon is the indication of widespread brittle deformation in nature with stresses insufficient to cleave the diamonds.