ESRF	**Experiment title: X-Ray topographic investigation of the distribution of stress in a diamond anvil under high pressure operation. Correlation with the intrinsic defects of the stone**	**Experiment number**: ME-383
Beamline: ID19	**Date of experiment**: from:07/07/2002 to: 07/09/2002	**Date of report**: 30/07/2002
Shifts: 6	**Local contact(s)**:J. Haertwig	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Dewaele Agnès
CEA/DPTA/SPMC
BP 12; F-91680 Bruyères le Châtel
*André Ramesh, same address
*Weck Gunnar, same address
Loubeyre Paul, same address

Report:

The aim of this work is a better understanding of the failure of diamond anvils under high pressure in the diamond anvil cell. In particular, we believe that characterization by x-ray topography can be a valuable alternative to infrared spectroscopy and optical microscopy as a diagnostic of anvil quality for high-pressure applications. During this experiment, synchrotron white beam x-ray topography has been used to characterize the crystallographic defects of:

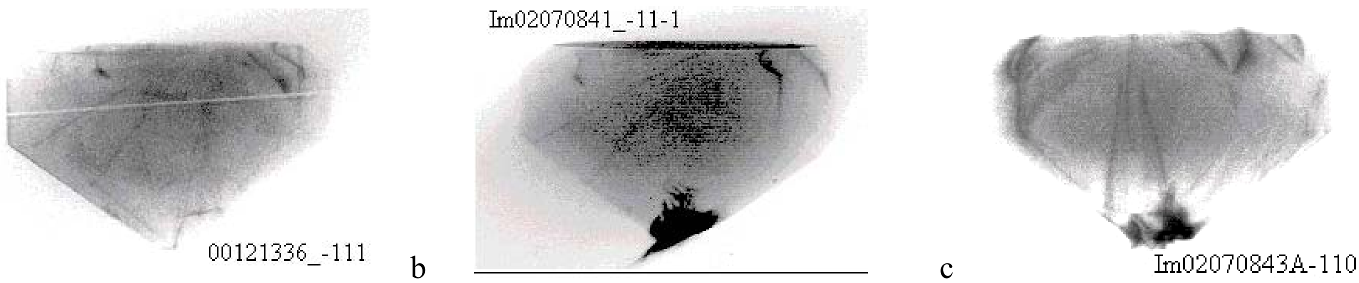
(1) 15 commercial diamond anvils of various types for use in high pressure cells (12 new, 3 previously subjected to high pressures);
(2) two diamond anvils fractured under high pressure (that had been characterized during HS-1036); and
(3) diamond anvils and an enclosed sample at high pressure in an attempt to detect the onset of plastic deformation.

This experiment is a continuation of HS-1036. In the following section, we present the results from both ME-383 and HS-1036.

(1) and (2) : defects of diamonds and influence on high pressure properties:

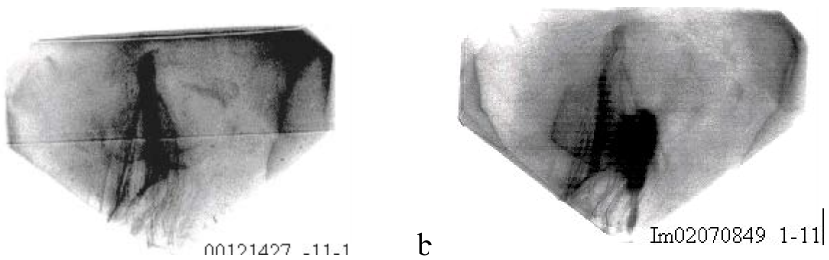
The topographs (side view and top view) of 38 natural diamonds have been recorded (22 type IA diamonds, 8 type IIA diamonds, and 8 commercial white diamonds provided by the Drukker company).
The IIA diamonds' topographs exhibited large plastic strain, associated with strong mosaicity (with rocking curves reaching 2 deg. FWHM for one diamond). In fact, type IIA diamonds strain easier than IA diamonds.
Topographs of IA and commercial white diamonds showed that these stones were of much higher crystallographic quality. In these classes, 8 out of 30 diamonds contained a large number of dislocations, mostly emerging from a central defect (inclusion ?). This is a feature of heterogeneous nucleation. Also, striations and strains associated with growth were obvious in 11 stones; planar defects following the (110) plane (stacking faults) have also been observed in 3 stones.

Among those diamonds, 8 have already been compressed in contact with He or H_2 samples, species that are known to weaken the diamonds by diffusion. The two diamonds that contained numerous dislocation broke, as well as an other diamond containing large growth strains. Five diamonds did not break, and some of them exhibited large growth strains. This number is still too small to establish a clear statistics between defects and failure, but it seems that dislocations, present in one stone out of 4, weaken the diamonds. As far as we know, topography is the only experimental method that allows detection of these defects.
Some other stones containing dislocations have been compressed in contact with other chemical species than He and H_2, and there was no evidence of weakening of the stone.
Thus, He and H_2 may diffuse along dislocations and embrittle the diamond.
The comparison of the topographs of one stone type IA (D68) before and after high pressure strain showed no evidence of an evolution of dislocation array with pressure. Thus, we did not observe any plastic strain under pressure, as proposed previously [1]. This needs to be confirmed for orther type of diamonds.
Topographs of stones studied before and after breakdown are presented on figures 1 and 2. It is not easy to establish a clear geometrical relationship between preexisting dislocations and crack planes. It seems however that the main crack plane in figure 1 flollows a peexisting dislocation, and that the crack in figure 2 extends up to a highly strained zone.



a b c

Figure 1: Diamond D57 : (a) before high pressure operating, numerous dislocations emerging from a central defect. (b) and (c) broken at 70 GPa, main fracture plane follows a preexisting dislocation.



a b

Figure 2: Diamond D57 : (a) before high pressure operating, numerous dislocations close to the tip, planar defect. (b) after break at 25 GPa, no obvious relationship between the fracture plane (a (110) plane) and the dislocations.

(3) silicon single crystal strained in a diamond-anvil cell.

This last experiment has been performed to observe both strain of the diamond and plastic strain of a silicon single crystal loaded in a diamond anvil cell. For this purpose, a (110) single crystal of 50 μm thickness and roughly 200 μm diameter has been loaded in a neon pressure transmitting medium in a diamond cell and strained up to 2.6 GPa. X-Ray were parallel to the compression axis and passed throughout both diamonds and silicon sample. Unfortunately, the silicon single crystal was too small to distinguish features in its volume. Increase of the strain of the diamond upon pressure increase was also clear on the topographs, but we did not see any appearance of any defects with strain. Actually, near the tip of the diamonds, possible defects were hidden by the large elastic strain of the diamonds. This made in-situ observation of defects evolution difficult.

References:

[1] Mao et al., Rev. Sci. Instrum., 50, 1002 (1978)