ESRF	Experiment title: X-ray topographic investigation of diamond anvils for high pressure generation – Correlation between defects and early failure	Experiment number: ME-684
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Report:

This work is the continuation of **HS-1036** and **ME-383** experiments. It is devoted to a better understanding of the failure of the diamonds anvils used in Diamond Anvil Cells (DAC) under high pressure. The diamond anvils are generally selected on the basis of their chemical purity, detected by infrared spectroscopy. However, this chemical purity does not guarantee the resistance of the diamond anvils under high pressure operation. X-ray topography could help to establish an alternative diagnostic of the anvils resistance, because this method evidences crystallographic defects. However, the liability of x-ray topography diagnostic has to be proven. For this purpose, diamond anvils used by our laboratory are characterized by x-ray topography before/after high pressure operation, and we correlate their resistance with the observed defects.

During the experiment **ME-684**, white beam x-ray topography has been used to characterize the crystallographic defects of :

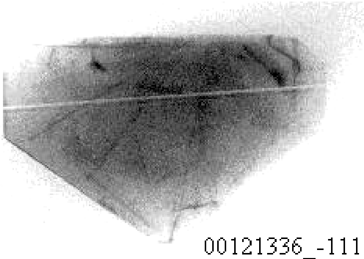
(1) 13 new diamonds (11 natural, 2 synthetic)
(2) 3 used diamonds (2 natural, 1 synthetic)
(3) 6 broken natural diamonds.

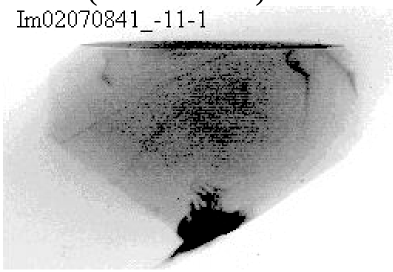
Monochromatic x-ray topography has also been carried out on 2 stones, to test whether the quality of the images was better than in white beam mode or not.
In the white beam mode, 3 different views of each stone have been recorded, which correspond to diffraction vectors perpendicular to 3 different high symmetry axis of the stones (axis (001) for the front view and axis (110) and (-110) for side views). It allowed us to detect as much crystallographic defects as possible.

We present here the general conclusions of the three experimental runs dedicated to this project.

On the basis of their crystallographic defects, we have sorted the diamonds in 3 classes. We observed that these class' diamonds have different resistance under high pressure, especially when compressed in contact with Helium. Helium is the best pressure transmitting medium in DAC experiments, but weakens diamond anvils by diffusion. Among the 48 studied diamonds, 16 have already been stressed under high pressure with helium. All of them were IA type.

Class 1. 11 out of 48 stones (~25 %) contain numerous dislocations, emerging from a central defect; this defect is almost always located at the centre of the stone and may be the seed. 4 of these diamonds have been used in contact with He and 2 of them broke (50 % failure).



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.

Class 2. 9 out of 48 stones (~20 %) contain strains that caused large contrasts on topographs ; these stresses have probably been created during growth (typical growth sectors, (111) planes, or stacking faults) . 4 of these siamonds have been used in contact with He and 3 of them broke (75 % failure).

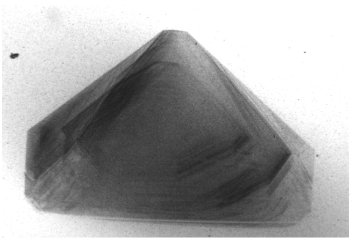
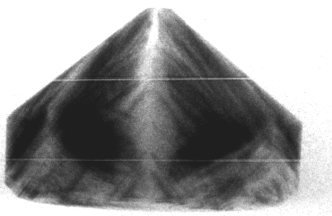
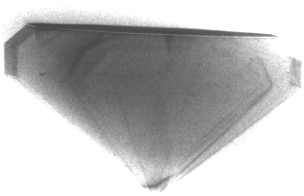
(a)


(b)


Figure 2 : (a) Im00121342_-11-1 and Im00121342_-110; **D64**, failure with He; (b) Im2709D83_1b11b Im2712D83_11b1, **D83**, not yet tested

Class 3. for 28 out of 48 stones (~55 %), no clear feature appeared on topographs (except weak growth sectors) ; contrast is very weak. Probably, these stones don't have large crystallographic defects. The synthetic diamonds belong to this category. 8 of these diamonds have been used in contact with He and 2 of them broke (20 % failure).

(a)

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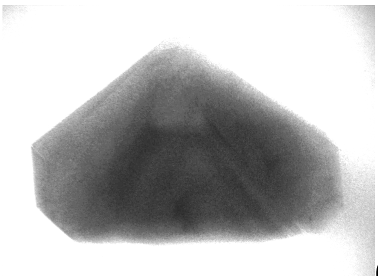
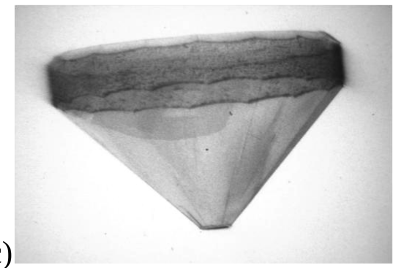

(c)


Figure 3: (a) diamond **D28**, He-resistant (b) diamond D109 ; not yet tested under He (c) synthetic diamond **N1918**, not yet tested under He.

It appears that the diamonds that have more crystallographic defects, evidenced by x-ray topography (class 1 and class 2), break more easily, even if this trend is not systematic.
The observation of the 15 broken stones (not observed before breakdown) qualitatively supports this conclusion : among them, 9 contain numerous dislocations emerging from a central defect (thus, much higher ratio than the natural occurrence of approximately 25 %).
This statistic correlation need to be further confirmed by high pressure operation of all diamonds already viewed by topography, and by x-ray topography of more new diamonds.