ESRF	**Experiment title:** In situ X-ray Micro-Diffraction Computed Tomography(XRD-CT) under extreme conditions using a new rotating tomography Paris-Edinburgh cell (RoToPEC)	**Experiment number**: MI - 1086
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Report:

The aim of this experiment was to extend the use of X-ray diffraction and absorption computed tomographies for *in situ* studies under high pressure and high temperature. To achieve this we have used a new rotating tomography Paris-Edinburgh cell (RoToPEC), that allows to rotate (360°) the anvils simultaneously and independently under extreme conditions.

This was the first time this new device was used in combination with synchrotron radiation for *in situ* experiments. The main technical difficulties and issues were understood and solved.

For diffraction-tomography experiments we have focused the beam (2 x 2 μm) using KB-mirrors. An ionization chamber before the sample was used to collect the incoming flux for image normalization. A radial multichannel collimator [1] was used to reduce the x-ray background from the sample environment in the Paris-Edinburgh press (PE). The scattered/diffracted 2D patterns were collected with a fast-readout low-noise (FReLoN) CCD camera (2048 x 2048 pixel resolution) in transmission geometry [2].
For absorption-tomography we used a parallalel beam. The absorption contrast is converted into visible light using a single-crystal YAG phosphor set in the transmitted beam on the downstream sample. A mirror reflects the visible contrast into a high-speed CCD camera, and the image is focused by an objective lens. The spatial resolution of this setup has been estimated as 4 μm [3]. Both, X-ray radiography and diffraction setups are compatible and easily intercheangable. During the allocated beam time 3 *in situ* measurements were done under high pressure.

(1) Resolution of a sphere by absorption tomography
(2) Deformation of a metallic foil followed by absorption tomography
(3) Diffraction tomography on a C60 sample under high pressure
(4) Partial measurement: Diffraction tomography on a C60 sample under high pressure and high temperature

As first measurement, we have used absorption contrast to perform a fast-tomography in order to evaluate the resolution of the new device under pressure.
The sample was a 180 µm tungsten carbide (WC) sphere loaded inside a 7 mm boron epoxy gasket (boron nitride capsule, graphite furnace) and pressurized to 8 GPa. The anvils were rotated simultaneously in the same direction. Radiographies were taken every 0.5° over half turn. This measurement was carried out at two different pressure points (Low P= 2GPa) and (High P = 8GPa) (figure 1).

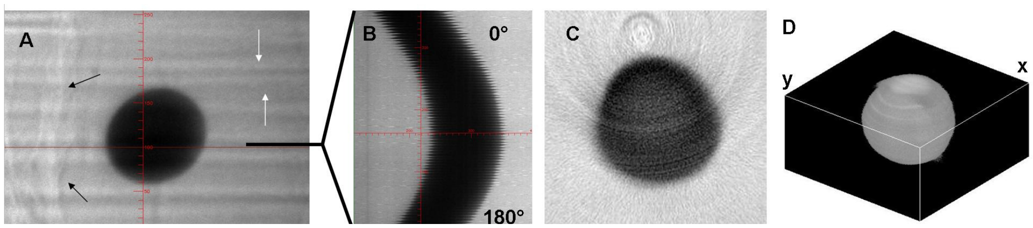

Figure 1. (A) single radiograph of WC sphere inside RoToPEC loading at 8 GPa. The black arrows indicate the contrast due to cell-extrusion. The white arrows show strikes due to beam-coherence. (B) Line integrals of intensity (Sinogram) for a single height of the sample (black horizontal line in A) (C) Reconstructed slice after Radon transform and back projection. (D) Rendered volume (stack of reconstructed slices).

The shape of the object has been perfectly reconstructed. The resolution of the new device will be determined from area/volume measurements for each pressure point. This experiment was also useful and necesary for fast detection of possible problems during rotation (centering, wobbling, shearing) and to follow *in situ* the effects of gasket extrusion.

Using the same X-ray radiography configuration, we have performed the first in situ high pressure deformation, followed by a fast-tomography of the deformed object.
A thin foil of Iron (thickness 50 microns) was loaded inside a 7 mm boron epoxy gasket (boron nitride capsule, graphite furnace and sodium chloride as pressure medium) and pressurized to 2 GPa. The anvils were rotated simultaneously in opposite dirrections by 55° and -55° at constant speed . The deformation process was followed in situ under high pressure (figure 2), then radiographies of the deformed foil were taken every 0.5° over half turn.

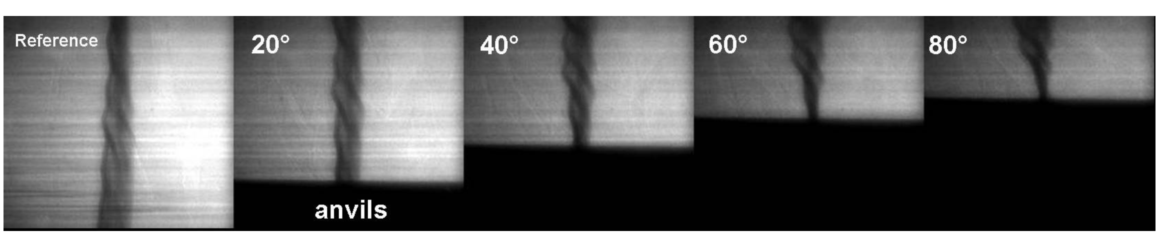

Figure 2. The reference image of the non-deformed foil is taken at 2 GPa. Indeed the object is already slightly deformed due to the pressure itself. The labels of the figures indicate the absolute displacement between upper and lower anvils. The dark shadow in the images corresponds to the absorption of the anvils. As the deformation rate increases the extrusion becomes more important, and the gap between the anvils decreases.

These results demonstrate the potential use of this new device for *in-situ* deformation experiments. Our new set-up will certainly become an important tool in geosciences.

For the third measurement we have changed to the diffraction-setup. We have performed the first in situ diffraction-tomography experiment under high pressure (ambient temperature).

High purity (99.9%) C60 powder was charged on a 1mm diameter boron nitride capsule, surrounded by a graphite furnance and loaded on a 10 mm boron epoxy gasket. Then the sample was compressed to 5 GPa and a set of micro-diffraction patterns were collected every 4°. The total scanned length was 1370 µm with a step size of 27 µm. Fast-online reconstruction was carried out simultaneously during data collection using XRDUA software (figure 4) [4].

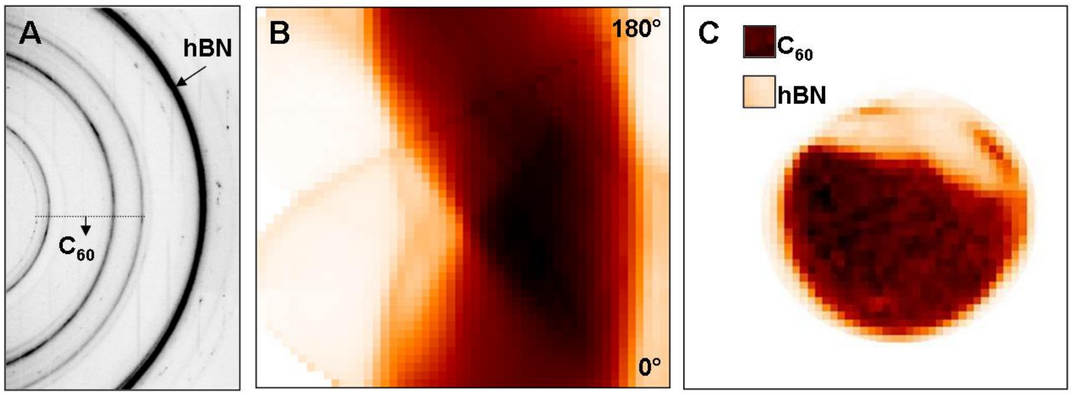

Figure 4. (A) Single 2D diffraction pattern showing reflections from the sample (C60) and the container (hBN). (B) Line integrals of intensity (Sinogram) for each rotation. (C) Reconstruction of the full signal of the corresponding slice, showing deformation of the cylindrical hBN container under high pressure.

We have started a similar experiment under high pressure and high temperature, were C60 undergoes polymerization. Due to time reasons only partial information has been collected, and to continue this study extra-beam time will be asked in the future.

We have determined a number of improvements for future experiments:(a) automation of specific routines to speed data collection (b) improve intrinsic resolution of the imaging system (c) eliminate effects from beam-coherence on the reconstruction. The technical details of the press as well as this first *in situ* demonstration will be the object of two publications (in preparation).

The ID27 team has performed a considerable amount of work to host this press in the experimental hutch EH1. The experimentalists would like to acknowledge Stany Bauchau, for the design and setup of mechanical elements used on this new type of experiment.

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