



Experiment title:

Bragg Coherent X-ray Diffraction Imaging (BCDI) of nanoscale strain evolution in a single crystal under high pressure.

Experiment number:

HC-4879

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Report:

The aim of this experiment was to perform in-situ experiments combining high-pressure and Bragg Coherent Diffraction Imaging (BCDI), which has become the most effective technique for imaging the 3D structure of individual crystals at the nanometer scale with an unmatched sensitivity to displacement fields. BCDI has shown its capability to image single defects in nano-crystals together with a picometer resolution of the displacement field. BCDI is a quite new technique which offers nowadays a revolutionary tool to image the shape, defects and strain field in 3 dimensions.

We propose to track the strain evolution and physical deformation from a single nanoscale crystal under high-pressure.

Pt nanocrystals are elaborated on Ytria Stabilized Zirconia (YSZ) substrate by solid state dewetting by Eugen Rabkin's group at the Israel Institute of Technology in Haifa. During the dewetting process the thin films agglomerate forming a large number of well-faceted crystals which are all oriented with the (111) plane parallel to the substrate surface (Figure 1).

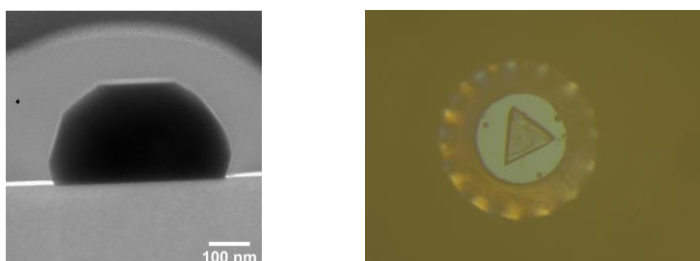


Figure 1: (a) TEM image of Pt nanoparticles. (b) sample and ruby balls introduced in the Diamond Anvil Cell.

The YSZ substrates were thinned down to 50 μm and cut in triangular shape of 100 μm . Two Diamond Anvil Cells (DAC) were prepared, a first one filled with He and another one filled with water. The beam energy was fixed at 33.169 keV, a sufficiently high energy which reduce attenuation when beam goes through diamonds and medium transmitting pressure. The beam was focused down to $2 \times 2 \mu\text{m}^2$ using KB mirrors.

To perform BCDI, a detailed intensity measurement of Bragg peak has to be accomplished. Thus, the detector was put as far as possible at 2.2 m to realise oversampling measurements of the Pt 111 diffraction patterns.

We were able to follow the Bragg peak of several individual Pt nanocrystals when increasing pressure in DAC filled with He or water up to 7 GPa (figure 2).

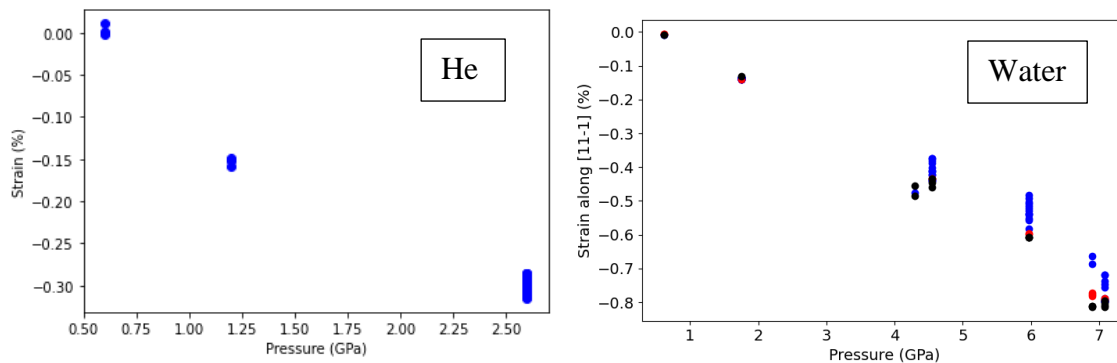


Figure 2: average strain in individual Pt nanocrystal in DAC filled with He or Water.

The intensities of the 111 Bragg peak measured in 3D for all the pressures are inverted using phase retrieval algorithms. **It gives the first images ever done in a DAC at the ESRF from BCDI measurements** (figure 3).

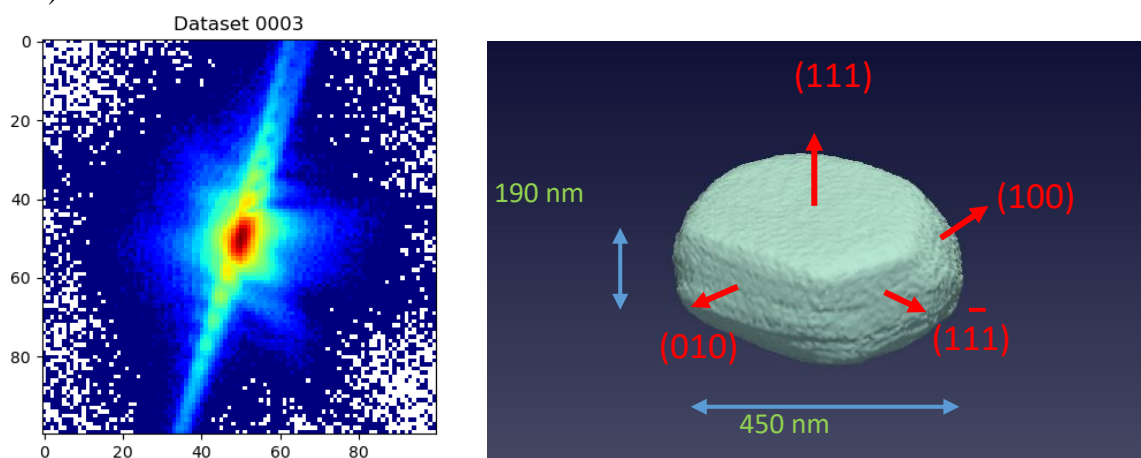


Figure 3: (a) 111 Bragg peak diffraction pattern of individual Pt nanocrystal. (b) 3D reconstruction of Pt nanocrystal.

In addition to the shape of the crystal, the BCDI measurements give the 3D displacement field inside the crystal and its evolution when increasing pressure (Figure 4).

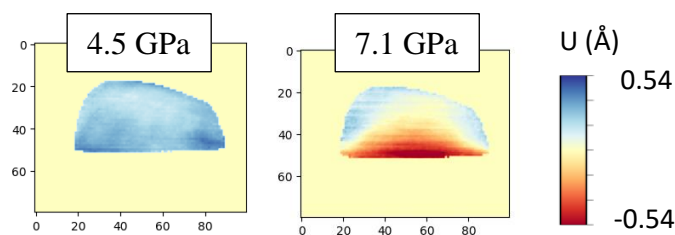


Figure 4: Evolution of the displacement field (component along the [111] direction) inside the nanocrystal when the pressure change.

We clearly evidence changes in the displacement field inside the nanocrystal indicating a modification of the internal strain of the crystal. The data are currently being analyzed with the aim of reconstructing real space images and internal strain of the crystal at different pressure level. These experiments are very promising. The ability to visualize stress-introduced deformation of nanocrystals with high spatial resolution and prominent strain sensitivity provides an important route for interpreting and engineering novel properties of nanomaterials.