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| ESRF | **Experiment title:**<br>Coherent diffraction imaging of dislocations and elastic strain fields during in-situ plastic deformation of metallic nanoparticles. | **Experiment number**:<br>SI-2022 |
| **Beamline**:<br>ID01 | **Date of experiment**:<br>from: 14 July 2010 to: 20 July 2010 | **Date of report**:<br>28 August 2010 |
| **Shifts:**<br>18 | **Local contact(s)**:<br>Dr. Thomas Cornelius | *Received at ESRF:* |

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## Report:

The study of mechanical properties of solids at the nanometre scale is still an open and challenging question [1-3]. As the yield strength increases significantly when the size decreases, the stress/strain fields are generally considerable in these small objects and may noticeably modify their physical properties. However the relationship between the actual defect content of the submicron object and its mechanical properties has been overlooked. It requires experimentally a combined approach of loading conditions for mechanical measurement on small scale and detailed micro-structural characterizations.

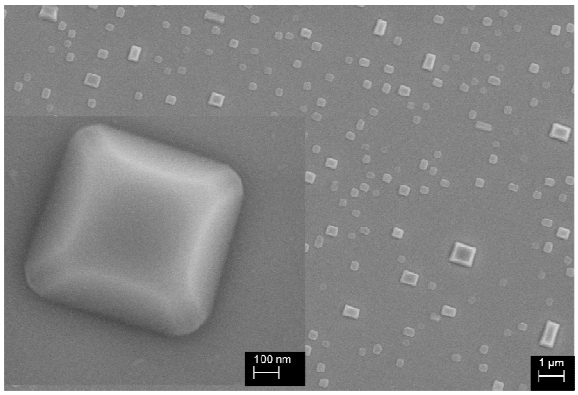


*Figure 1: Image of a typical sample.*

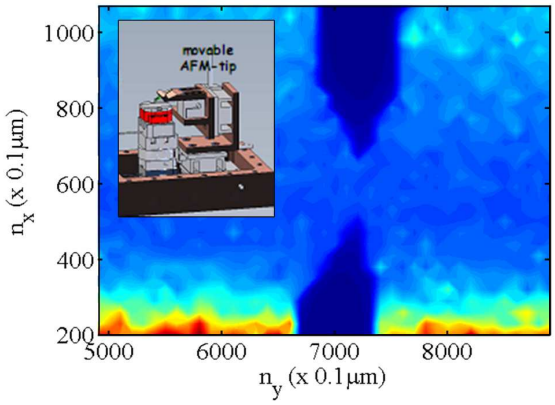


*Figure 2: Mesh of the movable AFM-tip in diffraction condition.*

In this way, microfocused coherent x-ray diffractive imaging (CXDI [4]) has been combined with an *in situ* atomic force microscope at ID01 with the AFM-tip used as a nano-indenter, to measure *in situ* the related strain response during nano-indentation of pure copper crystals of micron size grown epitaxially on a tantalum 001 substrate by dewetting a thin copper layer (see Figure 1).

The 8keV monochromatic beam was focused with a Fresnel zone plate, achieving a spot of approximately 1.2 µm(H)x0.6 µm(V) and allowing to measure a single Cu crystal. The coherence was obtained by closing the slits in front of the Fresnel zone plate and the speckle patterns were recorded with a two-dimensional (2D) Maxipix (pixel array) detector. More than ten Cu crystals were studied. *In situ* nano-indentation experiments were performed to peak depths of several hundreds of nanometres with an accuracy of 100nm. During *in situ* indentation, a 2D detector slice was recorded around the Cu 002 reflection to provide a real-time monitoring of the strain field. Before indentation and after unloading, full rocking curves of the Cu 002 reflection were measured in order to obtain a three-dimensional map of reflection in the reciprocal space to provide

information on local elastic and plastic deformations. The movable AFM tip was easily positioned above the selected crystal. By making a mesh of the tip along the *x* and *y* directions and in diffraction condition (see Figure 2), the position of the tip in the beam, where is the selected crystal, is given by the middle position in-between the shadows of the direct and diffracted beams by the AFM tip.

Figure 3 displays two typical central frames of a rocking curve of the Cu 002 reflection collected (a) before indentation and (b) during loading (~100 nm below contact). Principal streaks are related to the facets of the crystal. Additional fringes are due to dislocations and/or residual strain field.

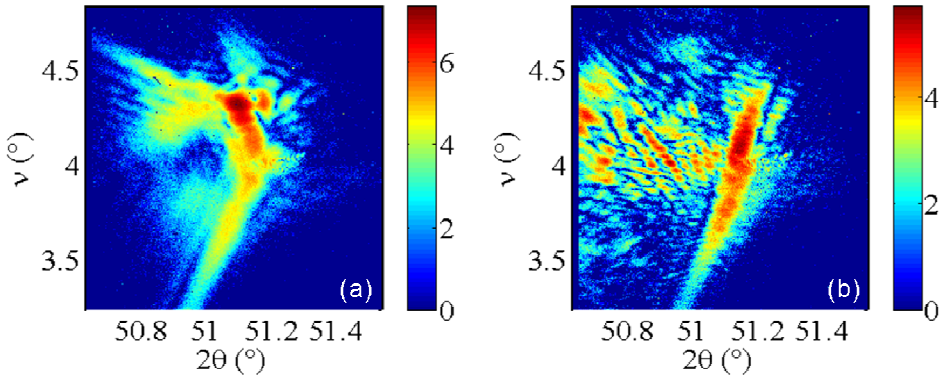


*Figure 3: Central frames of a rocking curve of the Cu 002 reflection collected (a) before indentation and (b) during loading. 2θ and ν are the out-of-plane and in-plane angles of the detector.*

Figure 4 displays 3D diffraction patterns around the Cu 002 reflection collected before indentation (a) and after successive compressive loadings (b and c). Changes are clearly observed and explained by plastic deformation. The measured data will be processed using the phase retrieval [5], from which we intend to reconstruct the displacement field around the inserted nucleations to provide insights for defect nuclation.

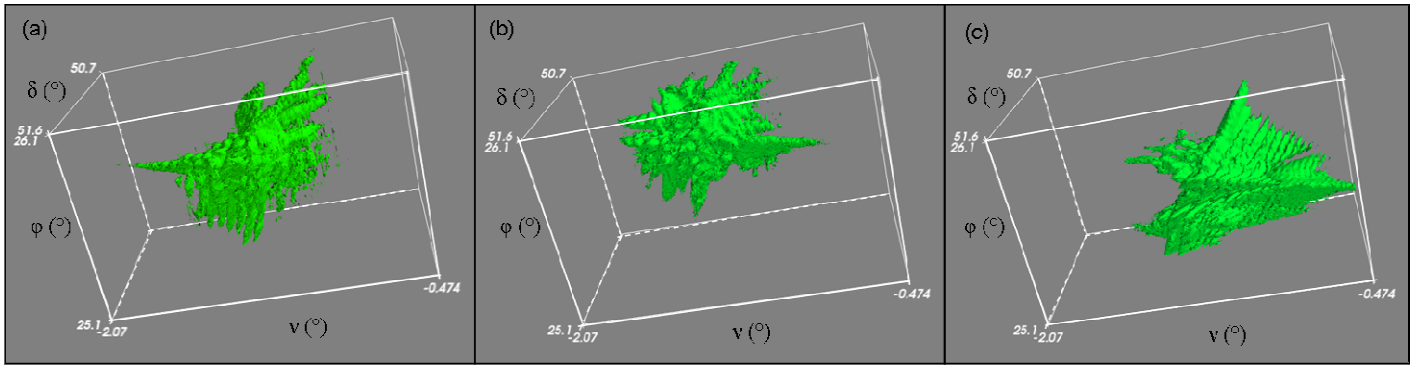


*Figure 4: Isosurface representation of a 3D rocking curve of the Cu 002 reflection (a) before indentation and after successive loadings (b)-(c). φ is the sample rotation angle.*

These results show the great potential of combining micro x-ray diffraction with an *in situ* atomic force microscope to observe *in situ* the initial stages of plastic deformation in Cu crystals under the load of an AFM tip. The coherent x-ray diffraction pattern revealed unambiguous and characteristic signatures of dislocations appearing during the load and at different stages of deformation. Unfortunately, the AFM apparatus available on the beamline could not monitor the force applied on the crystal, but only the displacement, and with insufficient accuracy (~100 nm). The further step will be to control quantitatively and reproducibly plastic loading.

## References:


[1] D. Maugis, "Contact, Adhesion and Rupture of Elastic Solids", Springer (1999).
[2] J.W. Kysara *et al.*, J. Mech. Phys. Solid **55**, 1554 (2007).
[3] W. Yang and B.C. Larson *et al.*, J. Mater. Res. **19**, 66 (2004).
[4] Newton *et al*, Nature Materials **9**, 2607 (2009).
[5] J. R. Fienup, Appl. Optics **21**, 2758 (1982).