IN SITU NANO-INDENTATION OF SINGLE Au NANO CRYSTALS IN COMBINATION WITH BRAGG COHERENT DIFFRACTION IMAGING

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The mechanical properties of micro and nanostructures have been demonstrated to vary significantly from their bulk counterparts. However, despite numerous studies, plasticity at the nanoscale is not fully understood yet. In situ experiments are perfectly suited for the fundamental understanding of the onset of dislocation nucleation. In particular, Bragg Coherent X-ray diffraction imaging (BCDI), which is non-invasive and does not need complicated sample preparation, is perfectly adapted for in-situ nano-mechanical studies thanks to its high sensitivity to strain and defects. Previous BCDI on indented Au crystals demonstrated the capability to image a single prismatic loop induced by nano-indentation and trapped inside the crystal [1].

Here, Au crystals grown on sapphire substrates by dewetting magnetron sputtered Au thin films with a thickness of 45 nm at 900°C for 24h, were indented using the scanning force microscope SFINX [2] and the elastic and plastic deformation was imaged by in-situ BCDI of the Au 111 Bragg peak at the ID01 beamine at ESRF. Since any movements of diffractometer motors may induce vibrations that eventually lead to damaging the nano-crystal under load, ordinary rocking scans are not suitable for recording 3D reciprocal space maps in-situ. Thanks to the achromacity of the KB mirrors, we scanned instead the energy of the incident X-ray beam, thus probing the intensity distribution in reciprocal space. Such E-BCDI was performed at different loading steps, hence imaging the evolution of strain and defects.

3D coherent diffraction patterns obtained during nano-indentation by scanning the energy of the incident X-ray beam as well as the phase are presented in Fig 1. With increasing applied mechanical load, the central part of the diffraction peak splits which translates in the appearance of defects separating the crystal in two domains (Fig. 1(b)). We further evidenced changes in the strain field with the increase of the applied load (Fig. 1(c-f)) which probably shows the deformation around the indent. After unloading the peak splitting and the corresponding defects as well as the variation in the strain field disappear. The high resolution 3D reconstructions of individual Au nano-crystals obtained by E-BCDI during nano-indentation thus give access to their morphology, the strain field, and dislocations therein as a function of the applied mechanical load.

To the best of our knowledge, this is the first time that E-BCDI has been successfully employed during in-situ experiments providing direct insight into the plasticity at the nanoscale and, in particular, the onset of defect nucleation.

[1] M.Dupraz, 3D imaging of a dislocation loop at the onset of plasticity in an indented nanocrystal, Nano Lett. 17 (2017)
[2] Z. Ren; Scanning force microscope for in situ nanofocused X-ray diffraction studies, J. Synchrotron Radiat. 21 (2014)

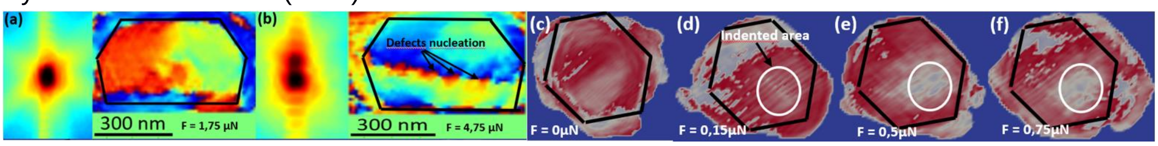

Figure 1 : *a,b) Qz-Qy integrated images of the core of 3D diffraction patterns for two different mechanical loads and Z-y cut of reconstructions of the phase for the corresponding mechanical loads. c,d,e,f) X-y cut of reconstructions of the strain field for different mechanical loads.*