## IN SITU NANO-INDENTATION OF AU CRYSTALS IMAGED BY BRAGG COHERENT X-RAY DIFFRACTION

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Key Words: in situ nano-indentation, Bragg coherent X-ray diffraction imaging, Au crystal, twin

The mechanical properties of micro- and nanostructures were demonstrated to vary significantly from their bulk counterparts. Despite numerous studies, plasticity at the nanoscale is, however, not fully understood yet. *In situ* experiments are perfectly suited for the fundamental understanding of the onset of dislocation nucleation. Recently, we developed a scanning force microscope (SFINX) which is compatible with 3<sup>rd</sup> generation synchrotron beamlines allowing for *in situ* nano-mechanical tests in combination with nano-focused X-ray diffraction [1] such as coherent X-ray diffraction imaging (CDI). This novel lensless imaging method retrieves the sample scattering function from a coherent X-ray diffraction data set using computational inversion algorithms, thus determining the phase of the scattered amplitude, which is not directly measured by a detector. In Bragg condition, the retrieved phase is directly related to the displacement field and, hence to the strain within a crystal.

Our previous BCDI studies on indented Au crystals demonstrated the capability to imaging a single prismatic loop induced by nano-indentation and trapped inside the crystal [2]. Since any movement 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*. Scanning the energy of the incident X-ray beam instead allows for probing the intensity distribution in reciprocal space without any detrimental vibrations [2]. Here, we report about the *in situ* nano-indentation of Au crystals with and without containing a twin boundary parallel to the crystal-substrate interface where the evolution of both strain and defects was imaged by multi-wavelength (mw) BCDI.

Figure 1(a) shows the electron densities for two parts of a twinned Au nanocrystal reconstructed from mw-BCDPs measured at the Au 200 Bragg peaks. The phase, which is directly related to the displacement field inside the structure, is presented in Fig. 1(b) for a gold crystal during nano-indentation allowing for following the evolution of the morphology, the strain field, and dislocations. With increasing applied mechanical load, defects, probably prismatic dislocation loops, appear at about half-height of the indented crystal, which disappear after unloading [4].

To the best of our knowledge, this is the first time that mw-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.

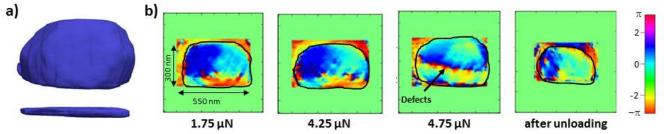


Fig. 1: a) Reconstructions of the upper and the lower part of a twinned Au crystal. b) Z-y cuts of the retrieved phase for an indented Au crystal at various loads as well as after unloading.

[1] Z. Ren, F. Mastropietro, S. Langlais, A. Davydok, M.-I. Richard, O. Thomas, M. Dupraz, M. Verdier, G. Beutier, P. Boesecke, T.W. Cornelius, J. Synchrotron Radiat. 21 (2014) 1128-1133
[2] M. Dupraz, G. Beutier, TW. Cornelius, G. Parry, Z. Ren, S. Labat, M.-I. Richard, G.A. Chahine, O. Kovalenko, M. De Boissieu, E. Rabkin, M. Verdier, O. Thomas, Nano Lett. 17 (2017) 6696-6701
[3] F. Lauraux, T.W. Cornelius, S. Labat, M.-I. Richard, S. Leake, T. Zhou, O. Kovalenko, E. Rabkin, T.U. Schülli, O. Thomas, to be submitted
[4] F. Lauraux, T.W. Cornelius, S. Labat, M.-I. Richard, S. Leake, T. Zhou, O. Kovalenko, E. Rabkin, T.U. Schülli, O. Thomas, in preparation