ESRF	**Experiment title:** Mechanical properties of single Au nano-crystals with single planar defect studied by in situ nano-indentation in combination with Bragg coherent X-ray diffraction imaging	**Experiment number**: MA-4612
Beamline: ID01	**Date of experiment**: Nov 7th to 11th, 2018	**Date of report**: 22 February 2021
Shifts: 12	**Local contact(s)**: Marie-Ingrid Richard	*Received at ESRF:*

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Report:

The aim of this experiment was the study of the mechanical properties of twinned Au (111) crystals by *in situ* nano-indentation in combination with Bragg coherent X-ray diffraction imaging (BCDI) to elucidate the influence of this single planar defect. We intended to measure two Bragg peaks, the Au 111 Bragg peak that is not sensitive to the twin boundary oriented parallel to the crystal-substrate interface and the Au 200 Bragg peak that is sensitive only to one of the two variants.

Gold crystals were prepared on sapphire substrates by dewetting magnetron sputtered Au thin films with a thickness of 30 nm at ~900 °C for at least 24 hours. During the dewetting process the thin films agglomerate forming a large number of well-facetted crystals which are all oriented with the Au(111). For BCDI the 8 keV X-ray beam was focused down to 500 x 500 nm^2 using KB mirrors. For *in situ* nanoindentation the *in-situ* AFM "SFINX" was installed on the diffractometer. The crystals, the AFM-tip, and the focused X-ray beam were aligned with respect to each other recording AFM-topography and scanning X-ray diffraction mapping.

Bragg coherent X-ray diffraction images of the Au 111, the Au 200, and the Au 002 Bragg peaks of a twinned Au crystal were ecorded by rocking scans. The Bragg electron density being displayed in Figure 1 was reconstructed from the Bragg coherent X-ray diffraction patterns presented in Figure 1 using the PyNX code. While the Au 111 Bragg reflection is insensitive to a twin boundary parallel to the crystal-substrate interface, the Au 200 and Au 002 Bragg peaks are sensitive to the two variants of the twinned crystal. The two variants have a thickness of 330 and 40 nm in agreement with the total thickness of the entire crystal of 370 nm.

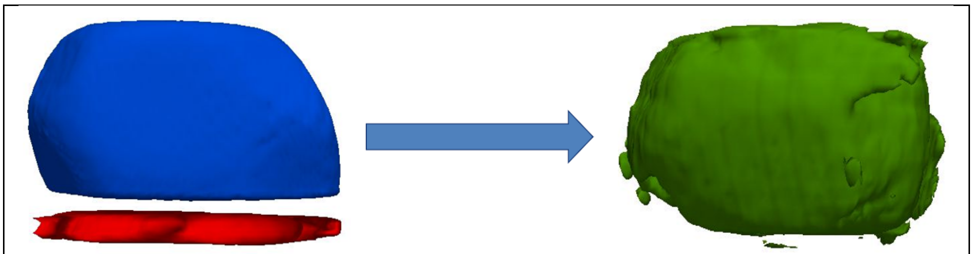

Figure 2. Reconstructed Bragg electron density of the twinned Au crystal.

In order to follow two independent Bragg peaks simultaneously, we developed a multi-Bragg coherent X-ray diffraction imaging technique where we installed two detectors at the same time – a Eiger 2M detector on the diffractometer arm and a Maxipix detector on a separate mounting as shown in Fig. 3. Employing the HKL mode of the spec-software used for operating the high-precision diffractometer at the ID01 beamline, the twinned Au crystals are oriented so that the Au 111 as well as the Au 200 lattice planes fulfill the Bragg condition at the same angle of incidence of 18.32° and at the same energy of 9 keV of the incident X-ray beam.

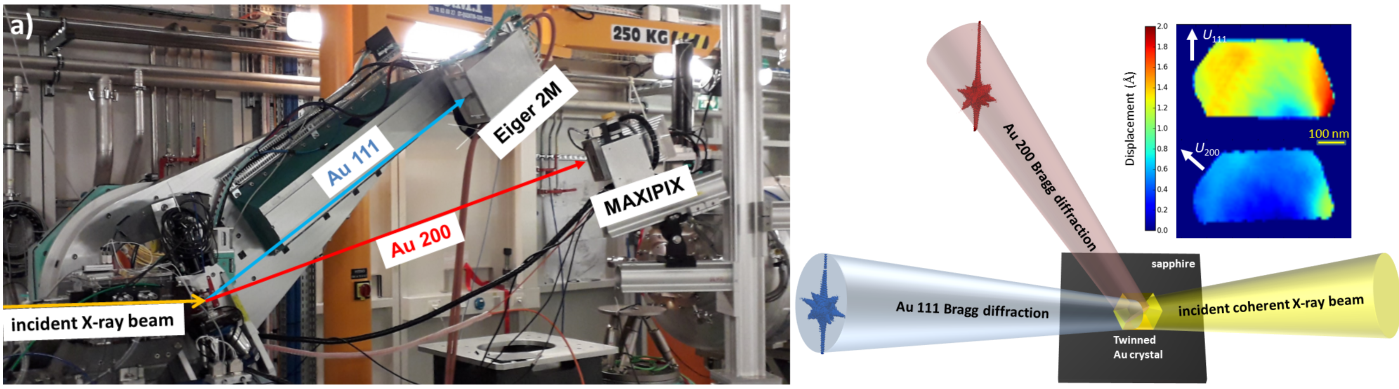

Fig. 2: Photograph of the experimental setup at the ID01 beamline at ESRF using two 2D detectors simultaneously.

Three-dimensional reciprocal space maps of the Au 111 and the Au 200 Bragg peak were recorded by multi-wavelength BCDI by scanning the energy of the incident X-ray beam from 8.75 to 9.25 keV in steps of 2 eV (Fig. 3(a)). The Bragg electron density and the phase were reconstructed from the 3D Bragg coherent diffraction patterns (BCDPs) using the PyNX software. An isosurface representation of the three-dimensional shapes of the crystals reconstructed from the Au 111 and the Au 200 Bragg peaks are presented in Figure 3(b). Both crystals have similar shapes. However, the thickness of the crystal reconstructed from the Au 111 Bragg peak is 370 nm whereas the crystal reconstructed from the Au 200 Bragg reflection measures only 330 nm. While the Au 111 Bragg peak is not sensitive to the twin boundary parallel to the substrate interface and thus represents the diffraction signal of the entire crystal, the Au 200 reflection corresponds to the upper part of the twinned crystal, thus explaining the difference in the thickness. Therefore, the second part of the twinned crystal measures 40 nm in height. The vertical cross-sections through the reconstructed crystals are shown in Figure 3(c) displaying the displacement along the respective *q*-vector. The displacement field is different for the Au 111 and for the Au 200 indicating that the displacement field fluctuation is larger for the component along the 111 direction.

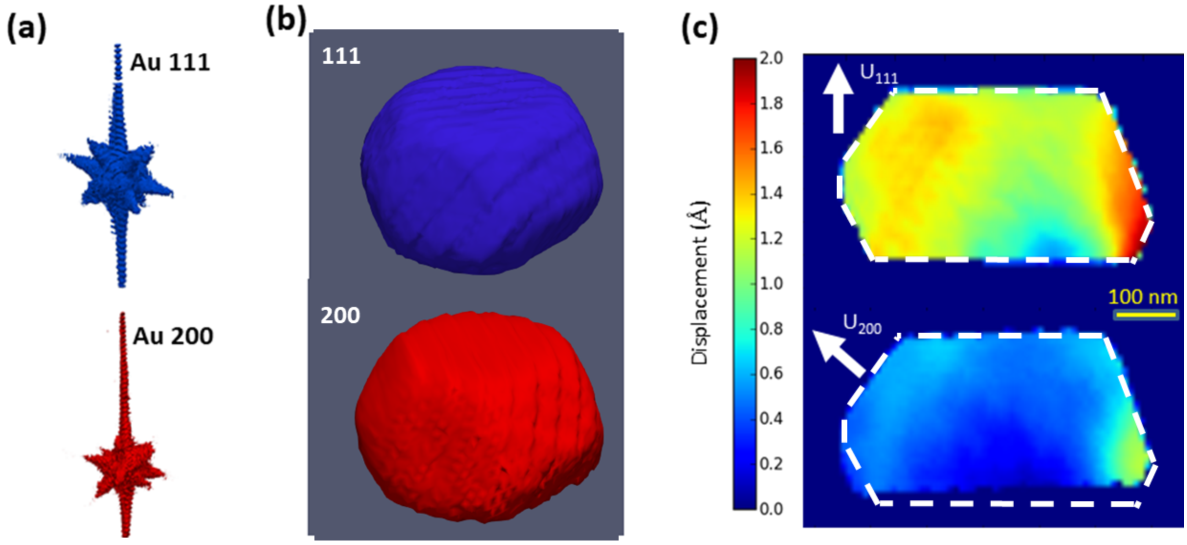

Fig. 3:

Publications:

F. Lauraux, S. Labat, S. Yehya, M.-I. Richard, S. Leake, T. Zhou, J.-S. Micha, O. Robach, O. Kovalenko, E. Rabkin, T.U. Schülli, O. Thomas, T.W. Cornelius, *Simultaenous multi-Bragg peak coherent X-ray diffraction imaging*, Crystals (2021) under review