ESRF	**Experiment title:** Quantitative diffraction on single nano-structures for mechanical, photonic and electronic applications	**Experiment number**: HS-4670
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Names and affiliations of applicants (* indicates experimentalists):

T.W. Cornelius*, O. Thomas*, *Im2np (UMR 7334) CNRS, Aix-Marseille University, 13397 Marseille, France*

M. Dupraz*, G. Beutier*, M. Verdier*, *SIMaP, Grenoble University, Grenoble, France*

V. Favre-Nicolin, *CEA Grenoble, Grenoble, France*

E. Rabkin, *Technion, Haifa, Israel*

Report:

The goal of this experiment was the study of the mechanical properties of single gold crystals by *in situ* nano-indentation in combination with coherent Bragg diffraction imaging. For this purpose, the new *in situ* AFM "SFINX" which had been developed within the framework of the ANR MecaniX project as well as this LTP was installed on the diffractometer of the upgraded ID01 beamline.

The gold crystals used in this experiment were prepared at the Technion in Haifa (Israel). A gold film with a thickness of 45 nm was magnetron sputtered on a sapphire (0001) substrate. During the annealing of this film at 930 °C for 24 hours, it was dewetted from the substrate, agglomerated, and formed a large number of hexagonally shaped Au islands with a flat top facet. The islands are all oriented with the Au(111) direction normal to the sapphire surface. By using a mask during the deposition of the initial gold film, a regular array of Au crystals was prepared where an indvidual Au island was located in the centre of a square formed by Au crystals (Fig. 1(a)). The side length of this square amounted to 50 µm allowing for studying a single Au island and image it before and after the experiment by SEM.

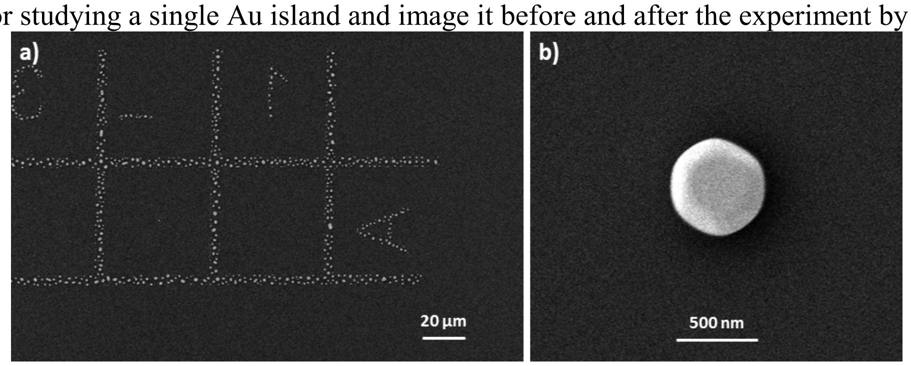

Fig. 1: SEM image of a) a regular array of Au crystals and b) a single Au crystal which was measured by *in situ* CBDI.

For coherent Bragg diffraction imaging, the 8 keV X-ray beam was focused down to 300 x 500 nm^2 using a tungsten Fresnel zone plate with a diameter of 300 µm. The high precision slits installed in front of the FZP were closed to 60 x 300 µm^2 matching the lateral coherence lengths at the upgraded ID01 beamline.

For *in situ* nano-mechanical tests, the AFM-tip, the Au islands, and the focused X-ray beam were aligned with respect to each other by simultaneously recording an AFM topography image and a scanning X-ray diffraction map. After alignment, the AFM-tip was positioned above a selected Au island (Fig. 1(b)), the feedback loop of the AFM as well as the excitation of the cantilever were switched off, and the tip was moved down with a speed of 2 nm/s indenting the island. At pre-defined loads, the AFM-tip was retracted with the same speed until it reached a position of 1 µm above the island top facet. During the loading and unloading of the island, 2D coherent X-ray diffraction patterns were recorded using a 2 x 2 MAXIPIX detector which was installed at a distance of 1.3 m downstream the sample. After retracting the AFM-tip, coherent Bragg diffraction images were recorded rocking the sample by +/- 0.5° (Δq = +/- 1.34 nm^{-1}). Vertical cuts through the coherent Bragg diffraction images for the pristine gold island, the island after loading to 250 nN and to 2 µN as well as after a 24 hours of studying the crystal by CBDI are presented in Fig. 2(a) – (d). All four diffraction patterns show distinct size fringes fitting to the crystal size measured by SEM. The inner structure as well as the diffuse scattering around the diffraction peak changes for the four different CBDIs. From the measurement, the real space image of the Au island was reconstructed (Fig. 2(e) – (h)). For the pristine as well as the indented crystal, the reconstructed shape coincides with the SEM image presented in Fig 1(b). However, after experimenting 24 hours the crystal shape changes showing an increase of the size of the (110) facets.

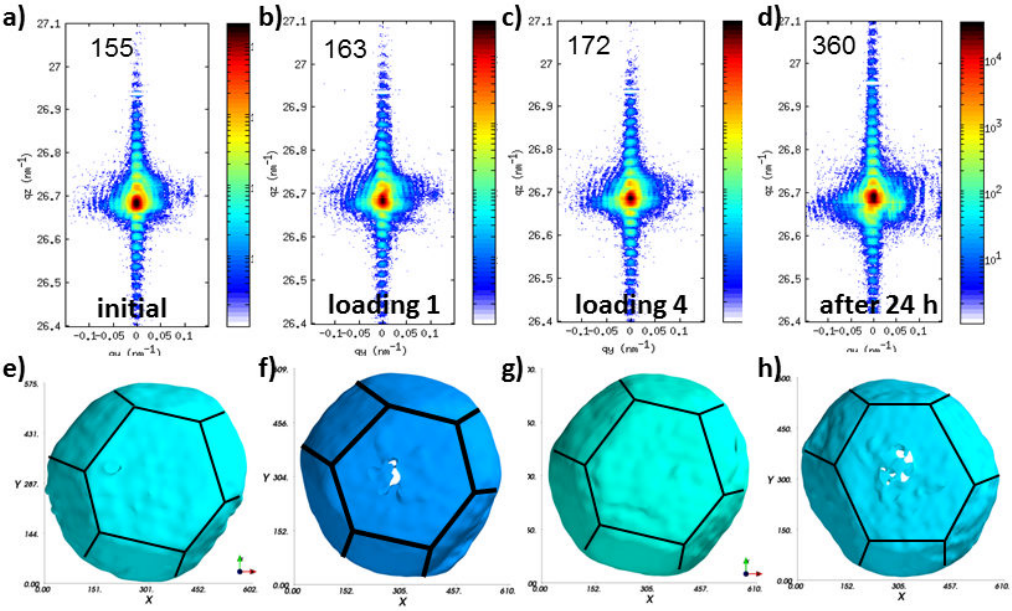

Fig. 2: Coherent Bragg different patterns of a) the pristine and the indented Au crystal loaded b) up to 250 nN, c) to 2 µN, and d) after 24h of measurement. e) – h) Reconstructed images from the CBDI shown in a) – d)

From the CBDI the phase in the crystal was reconstructed from which the strain in the crystal was calculated being presented in Fig. 3. At the island-substrate interface a tensile strain of ~6.5 x 10^{-4} was found for the pristine crystal (Fig. 3(a)). For the indented crystal the strain becomes more homogeneous which may originate from a relaxation of the residual strain by the nucleation of defects. For the crystal loaded up to 2 µN, a prismatic loop can be evidenced which has been induced by the mechanical loading (Fig. 3(b)). Note that in most cases no presence of defects could be evidenced after the indentation which may be caused by the diffusion of the defects to the surfaces where they were annihilated.

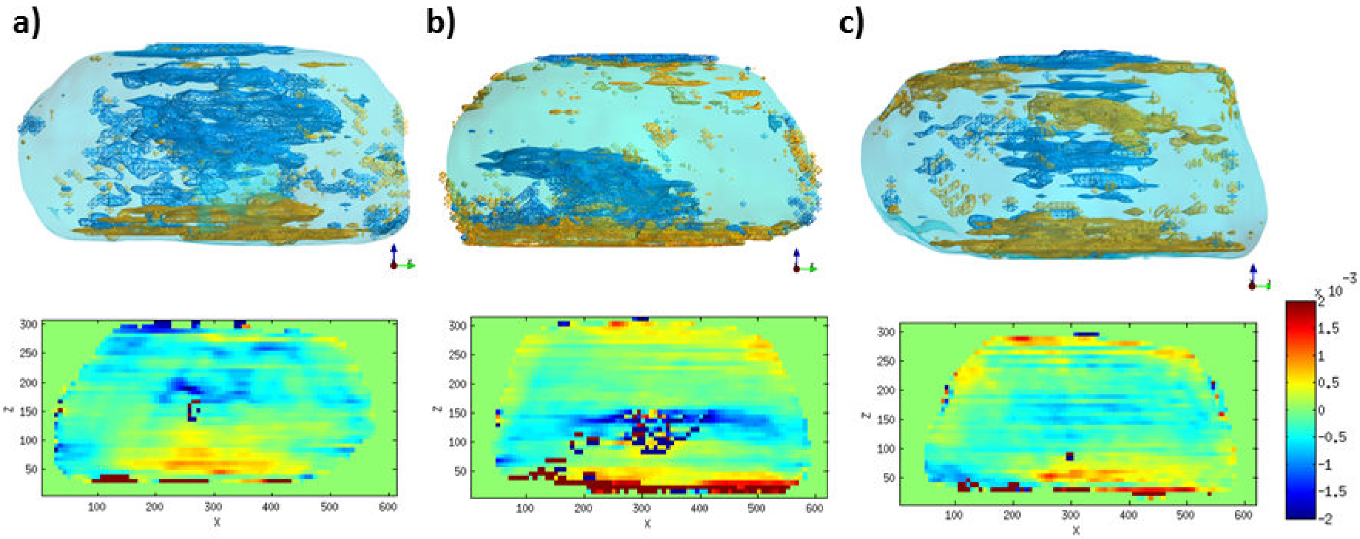

Fig. 3: Strain field calculated from the CBDI presented in Fig. 2 for a) the pristine Au crystal, b) after loading up to 2 µN, and c) after measuring for 24h