	Experiment title: In-situ Coherent X-ray Diffraction Imaging of Core-Shell Metallic Nanocubes during Nano-Indentation.	**Experiment number:** MA-4450
Beamline: ID01	**Date of experiment:** From: 11 Feb 2021 to: 16 Feb 2021	**Date of report:** 06/09/2021
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Report:

The main goal of this experiment was to study the influence of the interfacial microstructure of Core-Shell metallic nanocubes on the mechanical properties. Hence, study the Au-core/Ag-shell nanocubes with 0.2% lattice mismatch (coherent interface) by in-situ nanoindentation in combination with Bragg Coherent X-ray Diffraction Imaging (BCDI) measuring symmetric as well as asymmetric peaks to obtain a 3D image of the strain fields and defects nanostructure. The incident X-ray beam at ID01 was monochromatized to 9 KeV and focused down to 500 x 500 nm^2 using KB mirrors. Before mounting the AFM for in-situ indentation, Bragg coherent X-ray diffraction images of Au@Ag core-shells dropped on 100 µm of Sapphire substrate were recorded by rocking scans in order to measure the initial state of the structure of our nanocubes.

Due to the fact that the Au@Ag core-shell nanocubes are not attached to the substrate but are only deposited they are usually not stable under the beam. However, here we succeeded to record some diffraction patterns of Au@Ag core-shell nanocubes and to obtain the 3D reconstruction showing the Au@Ag core-shell structure (cf. figure 1). This was done before indentation where the Au@Ag nanocubes appear to be stable on Sapphire substrate.

For in-situ nanoindentation, the *in-situ* AFM "SFINX" was installed on the diffractometer. The nanocubes, the AFM tip, and the focused X-ray beam were aligned with respect to each other while recording AFM-topography and scanning x-ray diffraction mapping. Nevertheless, the isolated particles moved a lot under the beam for which BCDI combing in-situ indentation was not possible.

Due to this instability of these core-shells nanocubes, we decided to change the sample and perform BCDI combing in-situ indentation on Platinum nanoparticles which are most usually stable under the beam in the aim of inducing defects in these Pt crystals thanks to the AFM.

The Platinum nanocrystals were prepared on a Sapphire substrate by dewetting magnetron sputtered Pt thin film with a thickness of 30 nm at 1100°C for about 24 hours under Air. During the dewetting process, the thin film agglomerates forming a large number of well-facetted crystals which are all oriented in the Pt(111). Thanks to the stability of the Pt nanocrystals under the beam, the 3D Bragg coherent diffraction pattern (BCDP) of the Pt 111 Bragg peak was measured for several crystals before indentation to determine their initial state. This latter allows us to select the defect-free Pt nanocrystals by reconstructing the electron density and the phase from the measure BCDP alongside running the PyNX phase retrieval software using beamline computer.

The selected particles were then indented using "SFINX", where once again, the Pt crystals, the AFM tip, and the focused X-ray beam were aligned with respect to each other, while recording AFM-topography and scanning x-ray diffraction mapping. Moreover, energy scans were recorded during indentation in order to obtain a thorough follow up of the indentation process. Once the AFM tip was retracted a rocking scan was recorded for the indented NPs to obtain their final state. Figure 2 shows the 3D reconstruction of the electron density of two indented Pt nanoparticles. Two types of defects were induced: dislocation loops (cf. Fig 2-b) and defect blocked at the interface (cf. Fig 2-d).

We were not able to perform the originally proposed experiment due to the instability of the Au@Ag core-shell nanocubes under the beam. However, we got promising results while indenting the Pt nanocrystals where the stability of the induced defects can be explored by future synchrotron experiments that would consist of combing BCDI with thermoelastic strain by heating the sample using a custom-made furnace. These results shall soon be published in a high impact journal.

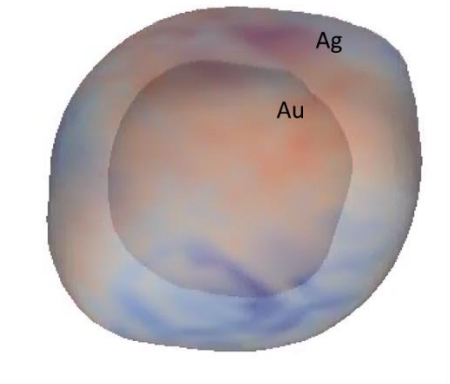

Figure 1: *Reconstructed shape of Au@Ag nanocube in 3D, colored with out-of-plane displacement field*

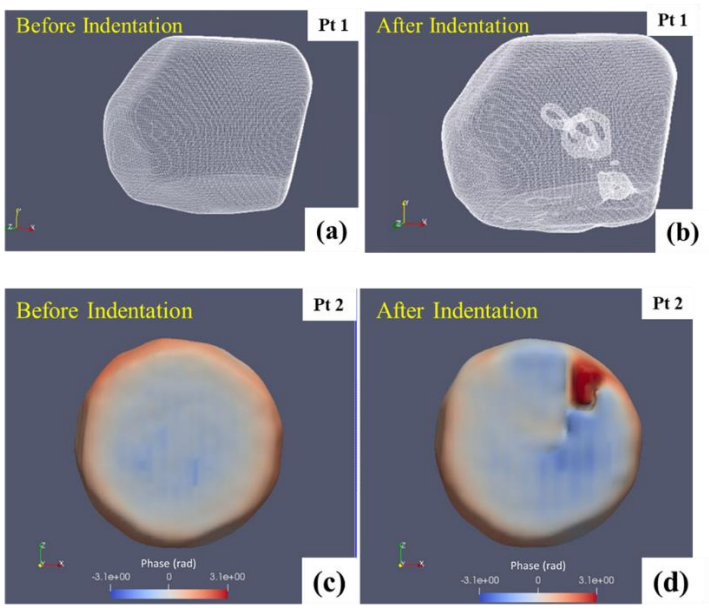

Figure 2: *Semi-transparent 3D reconstruction of the electron density of Pt 1 nanocrystal (a) before indentation evidencing no defects. (b) after indentation with induced defects inside. (c) bottom view of the 3D reconstruction of Pt 2 nanocrystal before indentation with defect-free surface, (d) bottom view of the 3D reconstruction of Pt 2 after indentation with a defect blocked at the interface.*