ESRF	**Experiment title:** Influence of nanoscale dimensions on the elastic and plastic properties of *epitaxial* gold nanoparticles.	**Experiment number**: SI-1835
Beamline: ID01	**Date of experiment**: from: 8 April 2009 to: 14 April 2009	**Date of report**: 30 July 2009
Shifts: 18	**Local contact(s)**: Dr. Thomas Cornelius	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Marie-Ingrid Richard (1), Marc Verdier* (2), Marc De Boissieu* (2), J. Keckes (3), G. Dehm (3), Olivier Thomas* (1)*

(1) Université Paul Cézanne, IM2NP, Marseille, France (2) SIMaP-INPG, Saint-Martin d'Hères, France (3) Leoben University, Austria

Report:

X-ray diffraction is a powerful non-destructive tool which allows analyzing the strain field of nanoobjects. To study the mechanical behaviour changes as the size of the objects is reduced, nano-indentation - *i.e.* applying an external localised force to a nano-sized object – is also well established [1]. The study of the local strain field under external mechanical stress implies to perform *in situ* measurements to quantitavely analyze the undergoing physical mechanisms.
In this way, micro X-ray diffraction (μ-XRD) has been combined with an *in situ* atomic force microscope at ID01 with the AFM-tip used as a nano-indenter, to measure *in situ* the related strain response during nano-indentation of epitaxial gold grains of nano- or micrometer size in a 200 nm high polycrystalline film as shown in Fig. 1 (a).

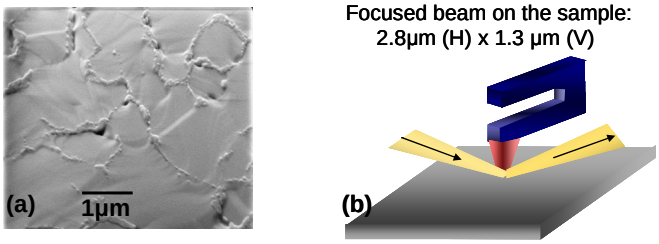

Fig. 1 (a) Scanning electron micrograph of the epitaxial gold nanofilm made of polycrystallites. (b) Scheme of the experiment: the micro x-ray beam and the AFM tip are located at the same position on the sample.

The x-ray beam with an energy of 7.75keV was focused with Be lenses. The beam size was approximately 3 μm (H)x1.22 μm(V) (see Fig. 1 (b)). A narrow slit in front of the Be lenses (60x20 μm^2) assured a transversal coherence of the beam. The scattered intensity was collected with a two-dimensional charged-coupled detector (CCD) (256x256 pixels) in a far-field position. Two selected Au grains were studied. *In situ* nanoindentation experiments were performed to peak depths ranging between 20 and 80 nm for the first studied grain and between 25 and 200 nm for the second grain. During *in situ* indentation, a 2D CCD slice was recorded around the Au(111) reflection to provide a real-time monitoring of the strain field. Before indentation and directly after unloading, the 3D diffraction pattern of the selected grain was collected around the Au(111) reflection to provide information on local elastic and plastic deformations. Figure 2 displays 3D diffraction patterns around the Au(111) reflection collected before indentation (a) and after compressive loadings characterized by a penetration depth of 20 (b), 50 (c) and 80 nm (d). Changes are clearly observed and explained by plastic deformation.

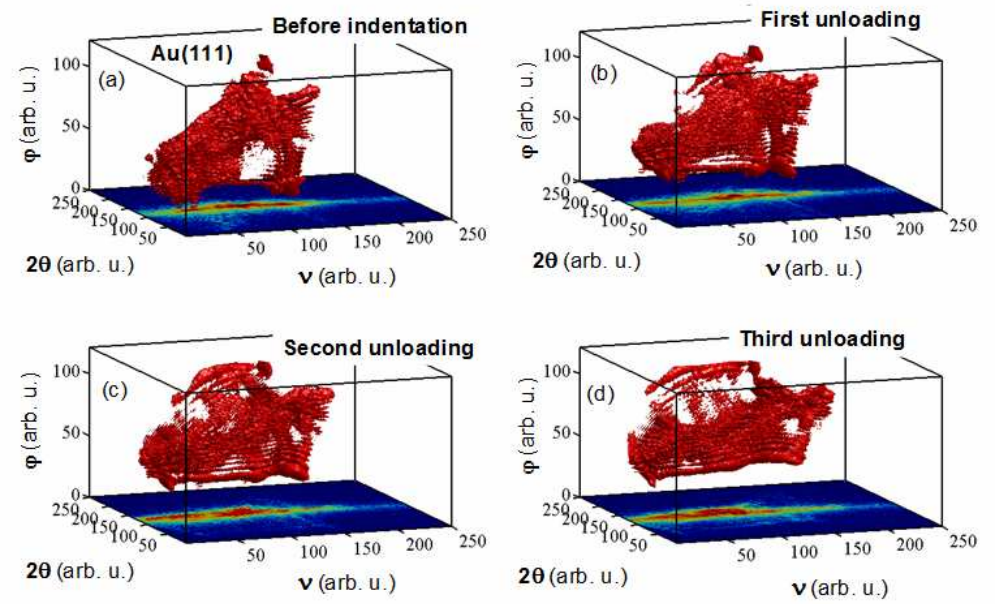

Fig. 2 Three-dimensional diffraction patterns around the Au(111) reflection before indentation (a) and after loading (c)-(d). The penetration depth of the AFM tip was 20 (b), 50 (c) and 80 nm (d).

Figure 3 shows 2D CCD slices which were recorded around the (111) reflection before indentation and after loading. The scattered signal is totally different and vertical fringes can be observed after loading. These interferences may be induced by phase field deformation caused by dislocations [2]. In this case, plastic deformation proceeds through dislocation nucleation. Figure 4 displays the intensity integrated along θ and ν as a function of δ = 2θ (angle between the primary beam and the detector). Broadening of the diffraction peak is observed (see inset of Figure 4), which is an expected consequence of the presence of defects such as dislocations. The peak position is also shifted after loading. This indicates that the mean lattice parameter of the crystallites is changing due do strain-field deformation.

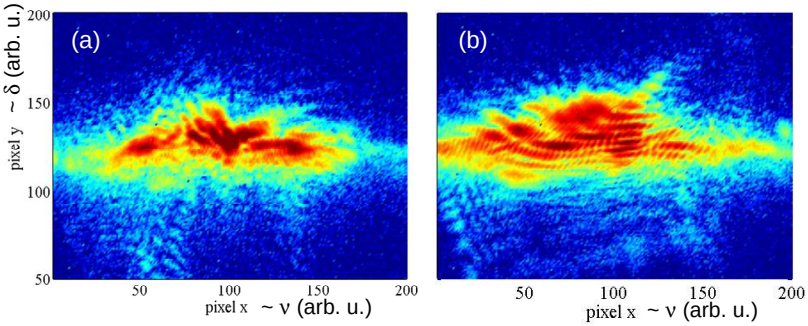

Fig. 3 (left) 2D CCD slices before indentation (a) and after loading with a penetration depth of 80 nm (b).

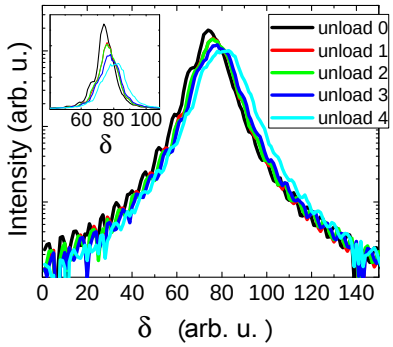

Fig. 4 (right) Intensity integrated along θ and ν for the second grain. The intensity is plotted vs δ= 2θ before indentation (unload 0) and after unloading. The penetration depths of the AFM tip for unload 1, 2, 3 and 4 were 28 nm, again 28 nm, 30 nm and 60 nm, respectively.

These preliminary results show the great potential of combining micro x-ray diffraction with an *in situ* atomic force microscope to examine defect nucleation in real time. The further step to clarify and quantify the effects of plastic deformation is to succeed in isolating the scattered signal of one grain. The measured data will be processed using the phase retrieval [3], from which we intend to reconstruct the displacement field around the inserted nucleations to provide insights for defect nuclation.

<u>References:</u>

[1] A. Gouldstone *et al.*, Acta Materialia **55**, 4015 (2007).
[2] D. Le Bolloc'h, Phys. Rev. Lett. **95**, 116401 (2005).
[3] J. R. Fienup, Appl. Optics 21, 2758 (1982).