	Experiment title: *Observation of first plastic events during in-situ compression of InSb micropillars using coherent x-rays*	Experiment number: MA1706
Beamline: ID01	Date of experiment: from: 10/04/2013 to: 16/04/2013	Date of report: 28/06/2016
Shifts: 18	Local contact(s): Gerardina Carbone	*Received at ESRF:*

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Report:

The goal of the experiment was to make a coherent x-ray diffraction study of InSb micropillars, during in-situ compression with a micro-compression device to observe the elastic and plastic deformation of these objects. An important question we tried to address in this experiment was to identify when the first plastic event appears during during deformation. Indeed, coherent x-ray diffraction can detect single dislocations in bulk sample, and is compatible with in-situ studies [1,2].

Here, many InSb micropillars (pillar axis: [213]) were available, with different sizes and diameters to determine the influence of the size on the mechanical behaviour under load. Around 20 of such pillars were prepared by Focused Ion Beam (FIB) milling, with diameters in the range 1-6 µm and heights of ~10µm (Fig. 1 shows an example of such single crystalline InSb micropillars)

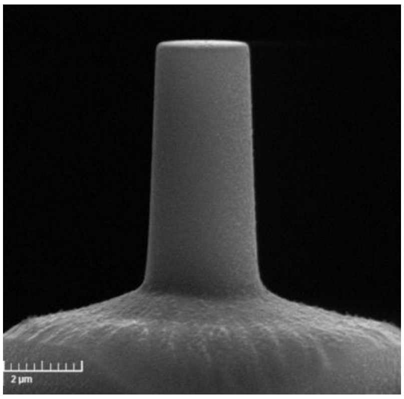

Figure 1: InSb micropillar with 2µm diameter.

The sample was installed in the micro-compression machine, specially designed for in-situ x-ray diffraction experiments. It is a compact setup that allows accurate mechanical studies, as the force and deformation are both measured, allowing to retrieve stress-strain curves. A diamond flat punch tip was used to perform the mechanical deformation, at a speed of few nm/s. Pictures of the compression machine, of x-ray absorption maps to align tip/sample/beam and a force/displacement curve are shown in Fig. 2.

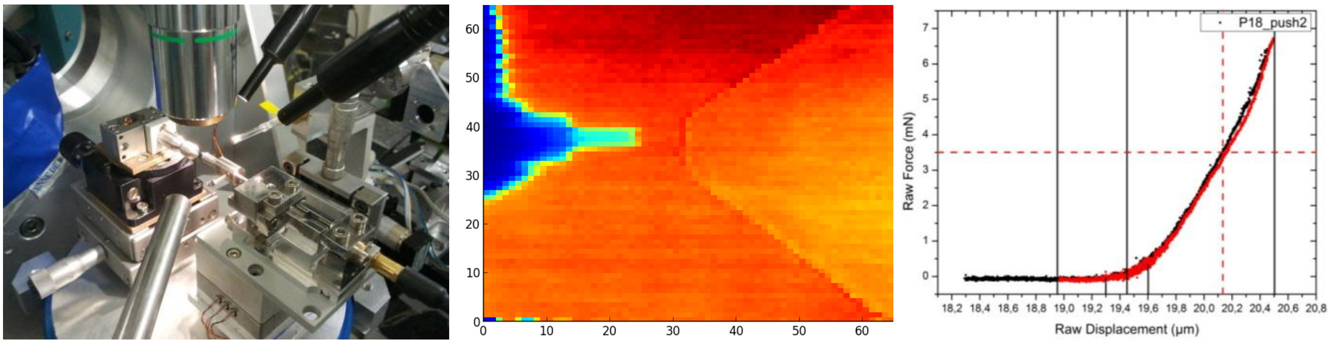

Figure 2: Left: micro-compression device mounted on the diffractometer of ID01. A microscope is used for precise alignment, and Fresnel Zone Plates are mounted on piezomotors to perform KMAP. Center: x-ray absorption image of InSb pillar (left) and diamond flat punch tip (right). Right: Force/displacement curve measured in-situ.

An 7keV beam was generated, and Fresnel Zone Plates (FZP) used to focus the beam down to 200(v)x500(h) nm². Coherence was achieved using slits before the FZP. Detection was performed with the Maxipix pixel detector.

Complete maps (rocking curves at each position of sample) were recorded before and after compression on the 11-1 and 202 Bragg reflections. The evolution of the Bragg reflection have been continuously followed during compression, at fixed angles to prevent vibrations that would destroy the sample when tip and sample are in contact.

Different deformation stages were detected (see Fig. 3 and Fig. 4): first a bending of the micropillar when the tip gets in contact, to accommodate for the eventual slight misorientations of the tip and pillar surfaces. Then, elastic compression was seen both on the in-situ compression curve and displacement of Bragg reflections, and finally plastic defects were created at a load of 6.3mN, observable through the appearance of speckles on the coherent diffraction patterns, and deviation on the stress/strain curve. The tip was then retracted and the unloading path was also recorded both on the diffraction patterns and stress/strain curve.

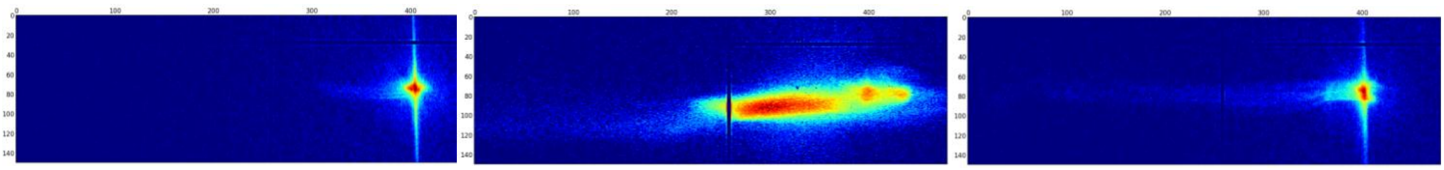

Figure 3: Left: 11-1 reflection before load; center: during load the elongation is due to the pillar bending and vertical deviation to elastic deformation. Defects appear through speckle along a direction which is out of the camera plane; right: 11-1 reflection after compression at 6.7mN.

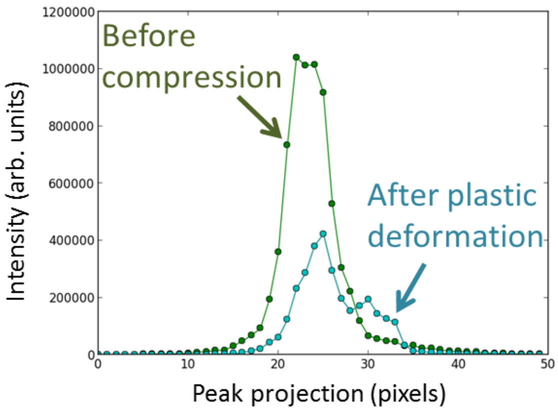

Figure 4: The peak is not similar to the one measured in the pristine state, because of the irreversible plastic deformation of the pillar.

These original experiments have been very succefull and the data are still being processed for the several loading-unloading sequences that have been performed for each micropillar.

[1] V. Jacques et al. PRL 106, 065502 (2011)

[2] V. Jacques et al. PRL 111, 065503 (2013)