ESRF	**Experiment title:** Onset of plasticity in Si nanopillars studied by *in-situ* coherent X-ray diffraction	**Experiment number**: HC-2911
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Report:

The aim of this experiment was to monitor *in-situ* the nucleation and glide of dislocations appearing during compression of semiconductor Si pillars using coherent X-ray diffraction (CXRD). Previous studies showed that a clear interference signal is measured on the coherent diffraction patterns when lattice defects (dislocations, stacking faults) are induced by deformation in the pillars. Recent compression tests on InSb micropillars using a custom built device adapted for the ID01 diffractometer demonstrated the feasibility of *in-situ* mechanical tests. While the aforementioned experiments focused on micropillars, we aimed at studying the first lattice defects in nanopillars during mechanical deformation and to determine the stress at which the first events of the plastic deformation appear. This study will open new perspectives for the study of plasticity in nano-objects. This experiment is a part of the ANR BiDuL project (ANR12-BS04-0003).

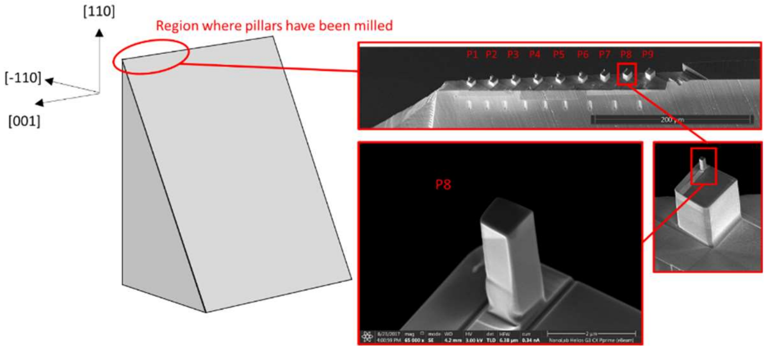

Figure 1. Si wedge with crystallographic orientations (incident X-ray beam along [-110]) and SEM images showing the series of Si pillars (with different dimensions) on their pedestals and, at highest magnification, example of pillar P8 with height of 2.8µm and lateral size of 0.9µm.

The samples consisted in [110]-oriented Si pillars with different sizes. They were carefully machined by Focused Ion Beam (FIB) milling, using very low ion beam currents to limit the creation of lattice defects and/or amorphization. Figure 1 presents some pillars standing at the top of a Si wedge, in a geometry that favours X-ray transmission for easier alignment of the pillars and Bragg diffraction for the in-situ CXRD tests. Prior to the experiment some pillars were deformed *ex-situ* by compression to directly study the influence of lattice defects on the diffraction patterns. The Fresnel Zone Plate used for beam focusing was installed on a piezostage facilitating the sample mapping by scanning the focusing optics through the incident X-ray beam and, thus scanning the focal spot across the sample surface.

In a first step, Coherent Diffraction Imaging (CDI) was performed on all pillars (virgin and ex-situ deformed). For the virgin pillars, the excellent quality of the crystal after FIB milling was verified through the quality of the **220** and **001** Si Bragg reflections (Fig. 2).

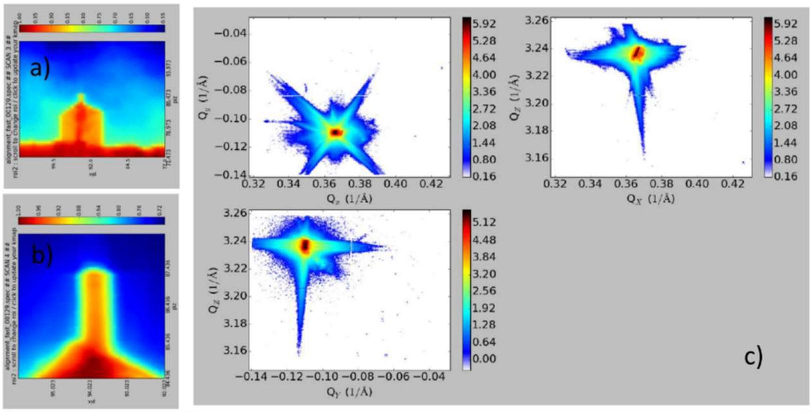

Figure 2. Diffraction maps collected at the maximum of the **220** Bragg reflection showing a) pillar P8 sitting on its pedestal and b) pillar P8 alone. c) X-ray reciprocal space maps around the **220** Si reflection obtained on pillar P8.

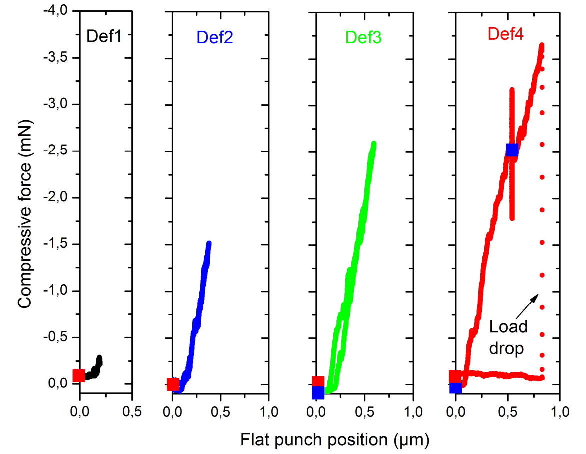

Figure 3. Force-position curves recorded during the 4 loading sequences imposed to pillar P8. Red squares are associated to the collection of k-maps while blue squares are associated to the collection of energy k-map (see text).

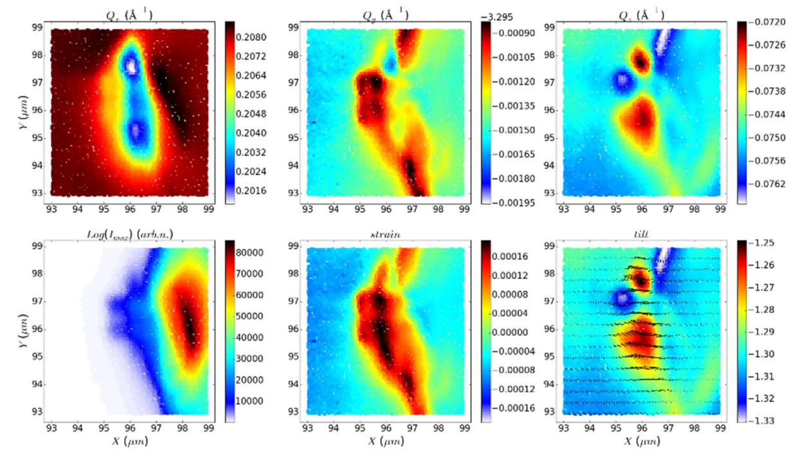

Figure 4. Q_x, Q_y, Q_z, intensity, strain, and lattice rotation measured by in-situ k-mapping using the energy tuning approach.

Figure 3 presents the mechanical curves recorded during the in-situ compression of pillar P8, for which 4 loading sequences were performed (def1, def2, def3, def4). Sequences def1 to def3 correspond to the incremental elastic loading of P8 (linear force-position curves) with collection of X-ray diffraction maps, using the k-map approach, before and after loading (red squares). For sequence def4, we developed a novel k-mapping approach by varying the energy of the incident X-ray beam instead of rocking the sample: these energy-k-maps (or “E-k-maps”) prevent any vibrations induced by moving diffractometer motors and can thus be performed also when the pillar is under load. Ordinary k-maps and E-k-maps (Fig. 4) were performed before and after the def4 loading sequence (red and blue squares in Fig. 3). During sequence def4, the loading sequence was paused in order to collect an E-k-map under load (blue square on figure 3) after which the loading sequence was resumed and a large load drop occurred, as a probable footprint of large plastic deformation of pillar P8: the analysis of k-maps and E-k-maps performed after this event should bring more information on its nature (nucleation and storage of dislocations?). Similar tests were successfully performed on pillars with different diameters: the analysis of the collected data is under progress.

The proposed experiments was very successful since several complex loading-unloading sequences could be in-situ performed on [110]-oriented Si pillars with different dimensions. In addition to ordinary k-maps, a new approach based on variable energy was also successfully developed and should provide access both to the rotation of the crystalline lattice and to the strain field inside the pillars under load. This novel experimental approach opens very promising avenues for the *in-situ* evaluation of the mechanical behavior of nanostructures.