ESRF	Experiment title: **“Brittle-ductile transition in low-dimensional silicon pillars studied by in situ 3D-XRD during mechanical compression tests”**	**Experiment number**: MA-1704
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The aim of this experiment was to study both the mechanical properties of individual nanostructures and the influence of size effects by coherent X-ray diffraction using nano-focused X-ray beams. While bulk silicon is brittle at ambient conditions and becomes ductile for elevated temperatures, Si nanopillars with diameters less than 300 nm are ductile at 300 K. A regular array of Si pillars with diameters of 2 µm, 400 nm, and 100 nm was prepared on a silicon-on-insulator (SOI) wafer employing electron beam lithography. The SOI (110) layer with a thickness of 340 nm is differently oriented than the Si (001) substrate allowing for disentangling the diffraction signals of the pillars and the substrate. An inter-pillar-distance of 20 µm ensures both to ambiguously locate a certain pillar and to avoid the contribution of neighboring structures. Nano-structures were mechanically deformed using our newly developed scanning force microscope for in-situ nanofocused X-ray diffraction studies (SFINX) and 3D reciprocal space maps were recorded in situ (see also experimental report of HS-4670).

The experiments were performed using the nanofocus setup at ID01. The incoming monochromatic x-ray beam with a photon-energy of 9.8 keV was focused down to a spot size of 600 x 300 nm^2 using a Fresnel Zone Plate (FZP). In order to probe ensemble properties using a conventional parallel x-ray beam, the FZP was removed from the optical axes. The measurements were conducted in coplanar diffraction geometry with the sample mounted horizontally on a piezo-stage for precise sample alignment and the diffracted intensity was monitored using a two-dimensional MAXIPIX detector.

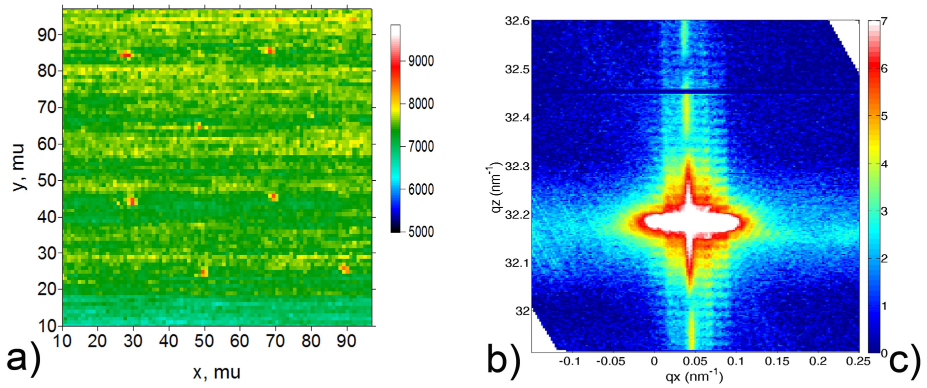

Figure 1. a) real space map of the sample surface recorded at Si 440 Bragg reflection ; b) RSM of micro pancake recorded at Si 220 reflection; c) RSM of 400nm diameter pillar recorded at Si 440 reflection

Figure 1a) shows a scanning X-ray diffraction map recorded at the Si 440 reflection using the k-mapping mode revealing the position of the 2µm sized structures. Figures 1b) and 1c) present vertical cuts from typical 3D reciprocal space maps (3D-RSM) for pristine structures with diameters of 2µm and 400nm, respectively. The 3D-RSMs reveal a superposition of the signals originating from the nanopillar and the remaining silicon layer (intense vertical streak observed along qz). The size fringes in qz-direction in Fig. 1b) correspond to a thickness of the remaining Si layer of 40nm and a height of the pillar of 300 nm. The comparatively large focal spot of the X-ray beam in comparison to nanopillars with a diameter of 100 nm illuminated a rather large area around the pillar resulting in a too strong background signal which circumvented the investigation of the smallest structures. Thus, the beam foci expected after the upgrade of ID01 will render it possible to investigate even the smallest pillars.

The second part of the beamtime was dedicated to in-situ compression tests of 400 nm diameter nanopillars. For this purpose, the AFM-tip of SFINX was positioned above one of the pillars, the feedback loop was switched off, and the AFM-tip was lowered with a defined speed of 3 nm/s corresponding to a strain rate of 10^{-2}. During the mechanical loading 2D diffraction patterns were recorded with an exposure time of 10 s, thus averaging over a movement of the tip of 30 nm. Besides the in-situ 2D diffraction images, 3D-RSMs were recorded before and after the compression test. The force sensor of SFINX is a self-sensing cantilever with a stiffness of 5 N/m. For this experiment, the AFM-tip was cut to flat bunch with diameter of 2 μm employing focused ion beam milling. For the time being, the force applied on a structure cannot be directly measured. It is approximated by the vertical movement of the piezo-stage during force application assuming that the complete movement is eventually converted into a deflection of the cantilever. Nanopillars were tested by applying forces ranging from 2.5 to 40 μN.

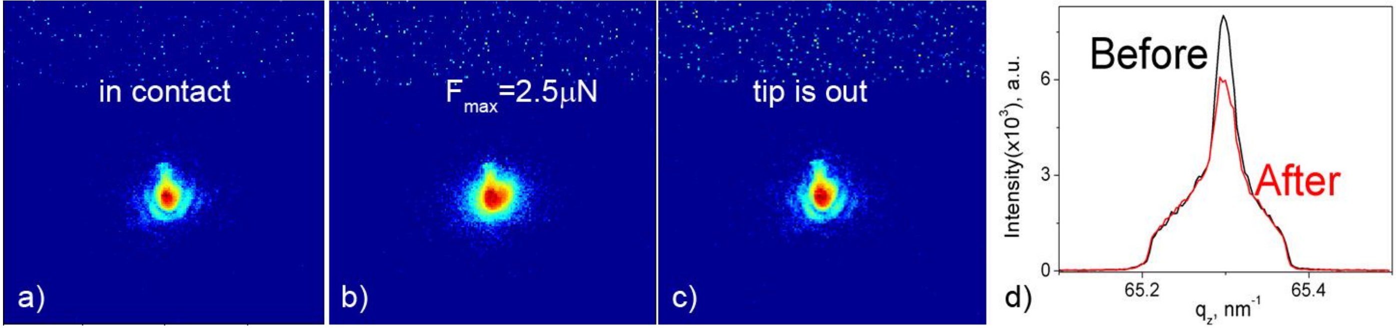

Figure 2. a) Detector image at Bragg peak position when AFM-tip is in contact with pillar surface; b) detector image at Bragg peak position when applied force is 2.5μN (maximal during particular compression test); c) detector image at Bragg peak position after compression test; d) vertical profiles from RSMs recorded before and after compression test

Two-dimensional diffraction images for three stages of a compression test are shown on Figs. 2a-c. The shape of the diffraction signal on the detector is the same before and after compression being characterized by concentric circles (shape of the nanopillars). At the maximal applied force of 2.5 μN the signal is strongly blurred. The effect of the mechanical loading on this nanopillar was investigated by recording rocking curves before and after the compression test. Vertical line profiles of the 3D-RSMs are displayed in Fig 2d showing a lower maximum intensity for the nanopillar after the compression test compared to before while the signal originating from the remaining Si layer (lower wider part) remains unchanged. For larger applied forces the signal of the nanopillar completely disappeared (not shown here). These experimental findings may be explained by an inclination of the pillars or even a detachment from the substrate.

In this experiment, we demonstrated the feasibility of in situ coherent X-ray diffraction studies on Si nanopillars with diameters down to few hundreds of nanometers in combination with mechanical loading using a self-developed scanning force microscope SFINX. A variation of the Bragg reflection is observed depending on the force applied on the Si pillars. However, the applied pressures are below the onset of plasticity for bulk Si. The observed changes in the 3D-RSMs may be caused by an inclination of the mechanically tested pillars originating from the fact that the SiO2 layer below the pillars is comparatively soft. Thus, this layer is deformed before the plastic regime for pure Si is reached. For future in situ experiments, the sample configuration will be improved avoiding an inclination of mechanically tested pillars due to a softer layer beneath and a direct measurement of the applied force and the displacement will be integrated into SFINX.