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Names and affiliations of applicants (* indicates experimentalists): *T.W. Cornelius, ESRF, Grenoble, France *T.H. Metzger, Max Planck Institute of Colloids and Interfaces, 14424 Potsdam, Germany		
*A. Davydok, Siegen University, Fachbereich 7, 57068 Siegen, Germany		
*R. Grifone, Siegen University, Fachbereich 7, 57068 Siegen, Germany		
U. Pietsch, Siegen University, Fachbereich 7, 57068 Siegen, Germany		

Report:

We studied the mechanical deformation of GaAs nanorods by combining the new *in-situ* "Two-Tower" atomic force microscope of ID01 and nanofocused X-ray diffraction. While in previous works, *in-situ* deformation studies were performed at one fixed Bragg angle only, we repeated the deformation studies at different rocking angles. These experiments at various rocking angles will pave the way to obatin *in-situ* the three-dimensional intensity distribution of mechanically deformed nanostructures and, thus, gaining more detailed information of the mechanical properties of individual nano-objects.

For our current studies, the X-ray beam was focused to 250 x 350 nm² by means of a Fresnel zone plate. GaAs nanorods with a diameter of 600 nm and a height of 300 nm (Fig. 1(a)) were grown on a GaAs substrate by means of selective-area metal organic vapor phase epitaxy. For that purpose, a SiN_x layer of thickness 15 nm is deposited on the substrate and subsequently structured by means of both electron beam lithography and wet chemical etching. The circular openings in the SiN_x layer define the position of the rods grown afterwards.



Fig. 1: a) Scanning electron micrograph and b) scanning X-ray diffraction map at the GaAs(111) Bragg peak of GaAs nanorods. c) Scanning absorption image of the AFM-tip.

The two stacks of piezo stages of the new *in-situ* AFM allowed for the independent alignment of the sample and the AFM-tip with respect to the nanofocused X-ray beam. A scanning X-ray diffraction map (SXDM) of the sample and an absorption image of the AFM-tip at the GaAs (111) Bragg angle are presented in Fig. 1(b) and (c), respectively. The SXDM shows both the quadratic arrangement of the GaAs nanorods and a clear separation of neighbouring pillars enabling us to select an individual rod with the nanofocused X-ray beam. When scanning the AFM-tip through the X-ray focus, the tip may either block the direct beam or the X-rays diffracted from the sample. Thus, two shadows of the AFM-tip are visible in the absorption image. In Fig. 1(c), the upper shadow originates from the absorption of the direct beam while the lower one is caused by the absorption of the X-rays diffracted from the sample. The position of the X-ray focal spot is located in the center between the two shadows being marked by a cross. After alignment, the AFM-tip is brought into contact with the selected nanorod and, subsequently, it is moved down stepwise increasing the force applied on the pillar. After reaching a certain deformation the AFM-tip is retracted stepwise. This compression test was repeated at different rocking angles.

Figure 3 presents two sequences of nano-XRD images for a GaAs pillar during compression taken at two different rocking angles. The two series were taken close to the GaAs (111) Bragg peak of the nanorods and the GaAs substrate. In the first images of the first sequence, the crystal trunction rod (CTR) of the substrate, the Bragg peak of the GaAs nanorods, and the diffuse Bragg signal of the GaAs substrate are visible. For the second serie, the substrate CTR and the substrate Bragg peak overlap and cannot be distinguished anymore. While increasing the pressure applied on the nanorod the intensity of the nanorod's Bragg signal decreases, the intensity of the substrate Bragg peak increases, and, finally, an additional flare appears at a q_z position larger than for the substrate Bragg peak. Thus, the atomic lattice of the nanorod is compressed during the experiment to the same value as the substrate and even beyond. During the experiment the substrate CTR stays at the same position proving that the sample does not tilt under pressure application.



Fig. 2: Two sequences of XRD images taken at two different rocking angles during the compression of a single GaAs nanorod.

Every two-dimensional diffraction image represents only a cut through reciprocal space whereas every sample is three-dimensional. Consequently, the ultimate goal is the *in-situ* measurement of the three-dimensional intensity distribution in the reciprocal space of the nanostructure under investigation. Thus, our new approach of performing *in-situ* compression tests at various rocking angles will enable us to record a complete three-dimensional reciprocal space map and gain information about the deformation of single nanostructures *in-situ*.