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</td><td>Experiment title:
3D reciprocal space mapping during in situ compression tests on single GaAs nanorods</td><td>Experiment number:
MA 1318</td></tr>
<tr><td>Beamline:
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18</td><td>Local contact(s): D. Carbone, V. Jacques</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
*T.W. Cornelius, IM2NP, Marseille University, France
*A. Davydok, Siegen University, Siegen, Germany
U. Pietsch, Siegen University, Siegen, Germany
T.H. Metzger Kleinberg</td></tr>
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Report:

We aimed on studying the mechanical deformation of single GaAs nanorods *in situ* combining the *in situ* AFM a the ID01 beamline and nanofocused X-ray diffraction. Due to problems with the GaAs samples we used for our current studies SiGe islands grown on Si(001) instead. To record a full 3D reciprocal space map *in situ* during the deformation, the energy of the incident X-ray energy was scanned in a pre-determined range. These experiments using the energy scanning approach paves the way to obatin the three-dimensional intensity distribution of mechanically deformed nanostructures *in situ* and, thus, gaining more detailed information of the mechanical properties of individual nanoobjects.

For our studies presented in this report, the X-ray beam was focused to 250 x 350 nm^2 by means of a Fresnel zone plate. The SiGe islands have a base width of 1000 nm and a height of 500 nm as shown in Fig. 1(a).

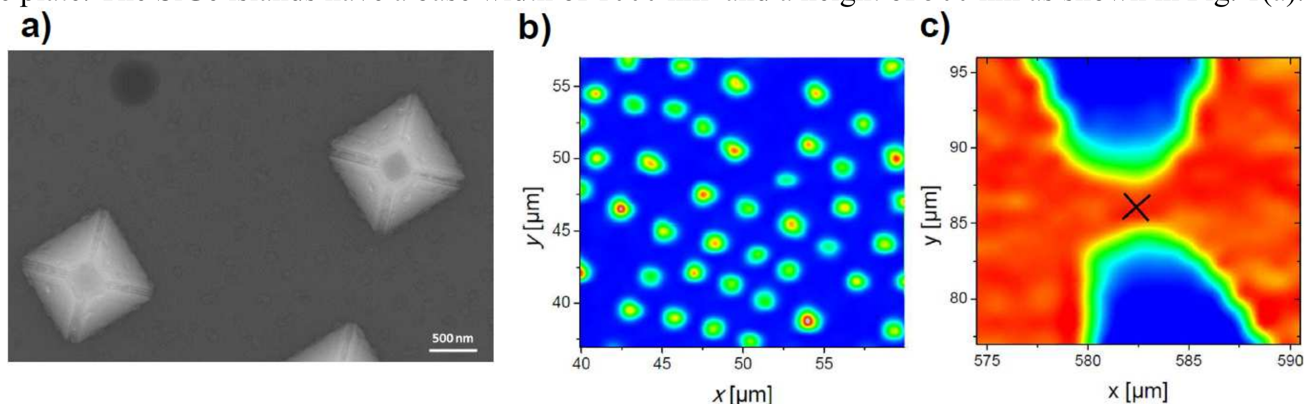

Fig. 1: a) Scanning electron micrograph and b) scanning X-ray diffraction map at the Si(004) Bragg peak of SiGe island array. c) Scanning absorption image of the AFM-tip at the Si(004) Bragg angle.

The sample and the AFM-tip were aligned separately with respect to the focused X-ray beam by scanning X-ray diffraction mapping (SXDM) and scanning absorption imaging, respectively. A SXDM of the sample and a scanning absorption image of the AFM-tip at the Si(004) Bragg angle are presented in Fig. 1(b) and (c), respectively. The SXDM shows the SiGe islands array and a clear separation of neighbouring islands enabling us to select an individual island 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 lower shadow

originates from the absorption of the direct beam while the upper 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 centre between the two shadows being marked by a cross. After alignment, the AFM-tip was brought into contact with the selected island and, subsequently, it was moved down consecutively increasing the force applied on the structure. Due to technical problems with experimental setup, the step size could not be controlled accurately. At each deformation step the energy was scanned by ± 100 eV in steps of 1 eV and 2D X-ray diffraction images were recorded simultaneously. This collection of 2D X-ray diffraction patterns as a function of energy allowed us to reconstruct the *in situ* 3D reciprocal space maps for different deformation stages.

Figure 5 presents 2D diffraction patterns, *in situ* 3D reciprocal space maps, and central q_x-q_z cuts through the *in situ* 3D-RSMs for a SiGe island at different deformation stages. The images for the pristine sample show the Si(004) Bragg peak, the substrate CTR, and the signal of the island including the streaks originating from the island side facets which are highlighted in the central vertical cuts by dotted lines. During pressure application on the island top facet the substrate CTR on the 2D diffraction patterns remains at the same position proving that the sample does not tilt in the experiment. Thus, all changes revealed by the *in situ* 3D diffraction mapping originate from the *in situ* deformation of the specimen. With increasing mechanical load, the central part of the diffraction signal of the SiGe island vanishes while the Si(004) Bragg reflection becomes more diffuse. Hence, a part of the island signal is superimposed with the substrate Bragg peak. In addition, the Si(004) Bragg peak develops a sub-structure for increasing load which may be caused by a superposition with the SiGe island signal, by strain induced in the Si substrate, or by defects created in the Si.

As observed from the *in situ* 3D-RSMs all 4 streaks originating from the island side facets turn to steeper angles with increasing load. The angle of elevation increases from 36 ° for the pristine sample to 60 °. This behavior is in good qualitative agreement with FEM and FFT calculations (not shown here). In the last stage (Fig. 2(d)) the island either is plastically deformed or experienced brittle fracture and no clear island signal is obtained anymore. The fringes observed around the position of the SiGe island signal and along the substrate CTR indicate the presence of defects created during the *in situ* compression. The island facet CTRs show the same angle as the pristine sample indicating a relaxation of the specimen.

This combination of the energy tuning approach and mechanical deformation of a single structure paves the way to novel *in situ* mapping of the 3D intensity distribution during mechanical testing.

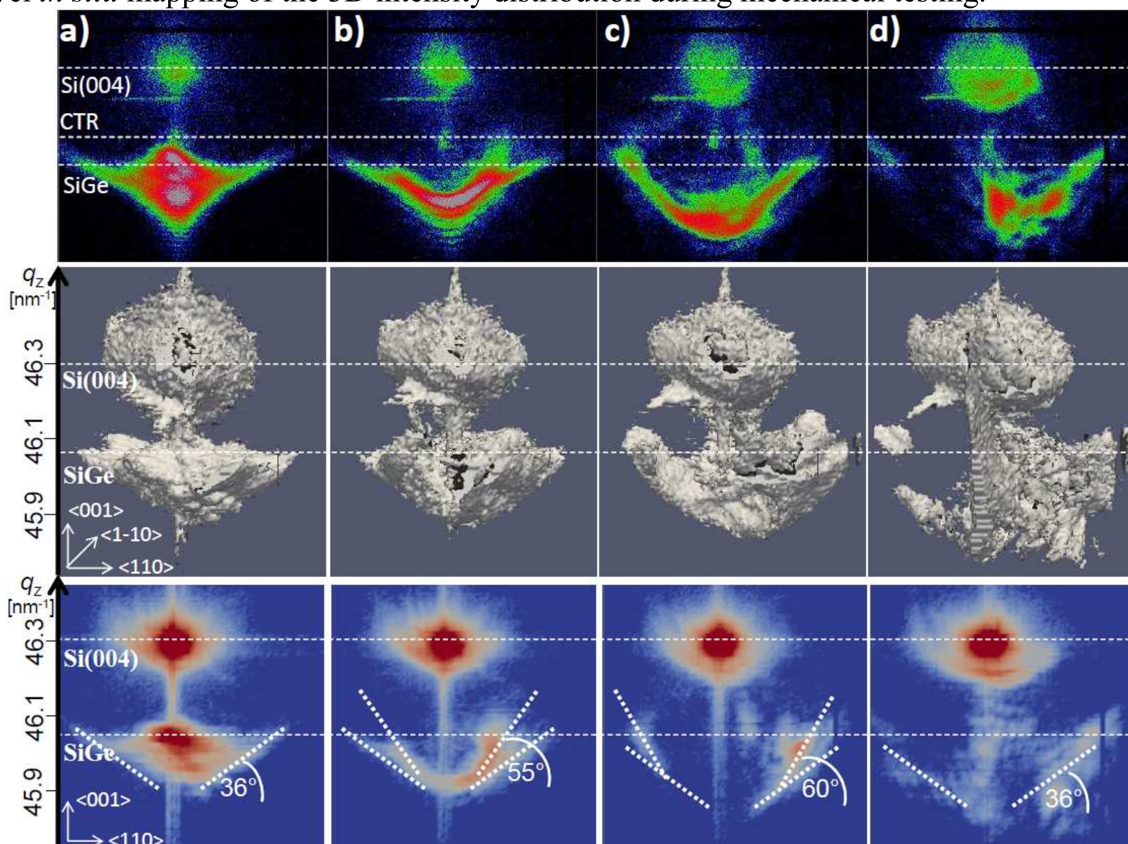

Fig. 2: 2D diffraction patterns, *in situ* 3D reciprocal space maps, and central q_x-q_z cuts through the *in situ* 3D-RSMs for a) a pristine SiGe island and b)-d) for the same island at different deformation stages.