ESRF	Experiment title: **"Strain relaxation in GaAs – InGaAs nanowire heterostructures on Si(111)"**	**Experiment number**: SI-2302
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Aim of the reported experiment was to study the relaxation behavior and structure of GaAs/InAs heterostructures in semiconductor nanowires (NWs) grown by molecular beam epitaxy (MBE). To this end, radial heterostructures ("core-shell" NWs) have been grown consisting of a GaAs core nanowire, overgrown by an InAs shell of varying thickness. A set of samples has been prepared prior the experiment with the NWs grown on GaAs (111) and Si (111) surfaces. Contrary to the initial proposal, at the present stage only radial heterostructures could be obtained instead of axial ones.

Experiments have been performed using the nanofocus setup of ID01. The incoming monochromatic x-ray beam with a photon-energy of 10keV was focused down to a spot size of 300 x 300 nm 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. Measurements have been performed in coplanar diffraction geometry with the sample mounted vertically on a piezo stage for precise sample alignment and the diffracted intensity was monitored using a two-dimensional MAXIPIX detector.
Figure 1 shows scanning electron micrographs from two of the inspected samples, both composed of GaAs nanowires on a GaAs substrate, overgrown with different thickness of InAs: sample (A) has a shell thickness of ~10nm, sample (B) has a shell thickness of ~25nm. In both cases, the core diameter is ~85nm.

Figure 2 shows reciprocal space maps (RSMs) around the symmetric (111) reflection of the two samples, obtained using a broad x-ray beam, i.e. representing average information over an ensemble of NWs.
Besides the Bragg peak of the GaAs substrate, located in the upper part of the RSM, several features can be identified, labeled by "1" – "3". Peak "1" measures a lattice parameter slightly larger than the bulk GaAs. With increasing shell thickness, the lattice mismatch compared to the bulk GaAs increases from 0.25% (A) to 0.56% (B) (arrow). For sample (A), a second peak "2" is found between the expected positions of GaAs and InAs, showing an

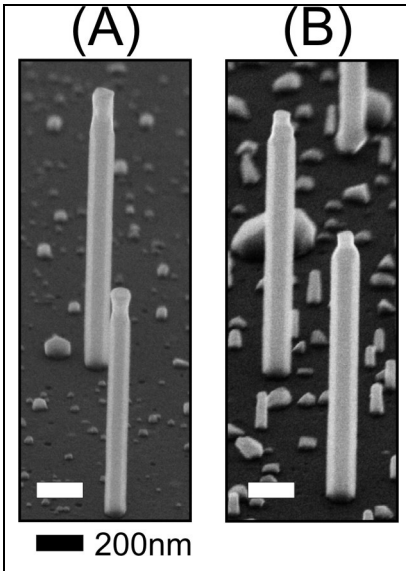

Fig. 1: SEM images of GaAs / InAs core-shell NWs with thin (A) and thicker (B) shell

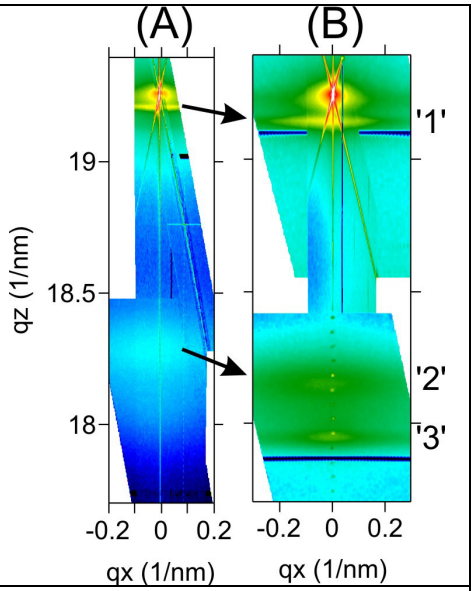

Fig. 2: Reciprocal space maps around the symmetric (111) reflection for samples (A) and (B)

asymmetric shape towards the pure InAs position (q_z=17.95nm^{-1}). For sample (B), this peak shifts further towards the pure InAs position (arrow). A possible scenario to explain the positions and shift of peaks "1" and "2" is an intermixing process of Ga and In at the GaAs / InAs heterointerface during growth, forming an In-rich InGaAs compound in the shell and a Ga-rich GaInAs core. For sample (A), the peak position of peak "2" indicactes a relatively high Ga content of 30% using Vegard's law. The asymmetric shape of this InGaAs-type peak for sample (A) indicates a gradient in lattice parameter, explained by a gradual decrease of Ga content towards the outer part of the shell. Whereas the peak shift of peak "2" towards InAs for sample (B) measures the increased mean lattice parameter of the considerably thicker shell for this sample, the simultaneous shift of the core's Bragg peak "1" further away from the bulk GaAs position is interpreted by enhanced interdiffusion of In from the shell into the core (which does not grow anymore) during the increased growth time at elevated temperatures.

For longer InAs growth times (sample (B)), a third peak appears at the position of pure InAs. Comparison with SEM images (fig. 1) suggests that pure InAs crystallites and smaller NW´s have been formed in-between the original NWs. In order to probe this attribution, the spatial distribution of peak "1" – "3" has been determined.

To this end, a newly developed tool could be used, allowing continuous scanning of the sample through the focused x-ray beam together with a fast readout of the MAXIPIX detector. Compared to the previously used method to fix the sample position at dedicated positions and acquiring images (conventional mesh-scan), the new tool dramatically decreases the time necessary to map large sample areas. Figure 3(a) shows the spatially resolved intensity distribution of peaks "1" (GaInAs = core?, left) and "2" (InGaAs = shell?, right) in a surface area of 25x25µm², measured with 250nm lateral step size. For both maps, elongated maxima are visible, caused by the "shadow" of the rather long NWs in the x-ray beam. Figure 3(b) shows an overlay of both intensity distributions, where the InGaAs-shell peak has been represented by grey contours. As visible, both maps coincide nicely, except a small offset caused by the sample rotation necessary in order to choose the appropriate detection volume in reciprocal space.

A similar position map has been recorded for the pure InAs signal simultaneously with the shell's peak. In this case, both diffraction signals could be recorded exactly simultaneously by choosing appropriate ranges of interest on the 2D detector. Figure 3(c) shows the comparison of the InAs map (color) with the shell-signals already shown in Fig. 3(b). Obviously, both intensity distributions do not match, but the pure InAs signal is mainly located in between the nanowires. In addition, the elongation of the InAs signal along the diagonal direction is smaller, in agreement with the assumption that the InAs is growing as smaller crystallites and wires in-between the initial NWs.

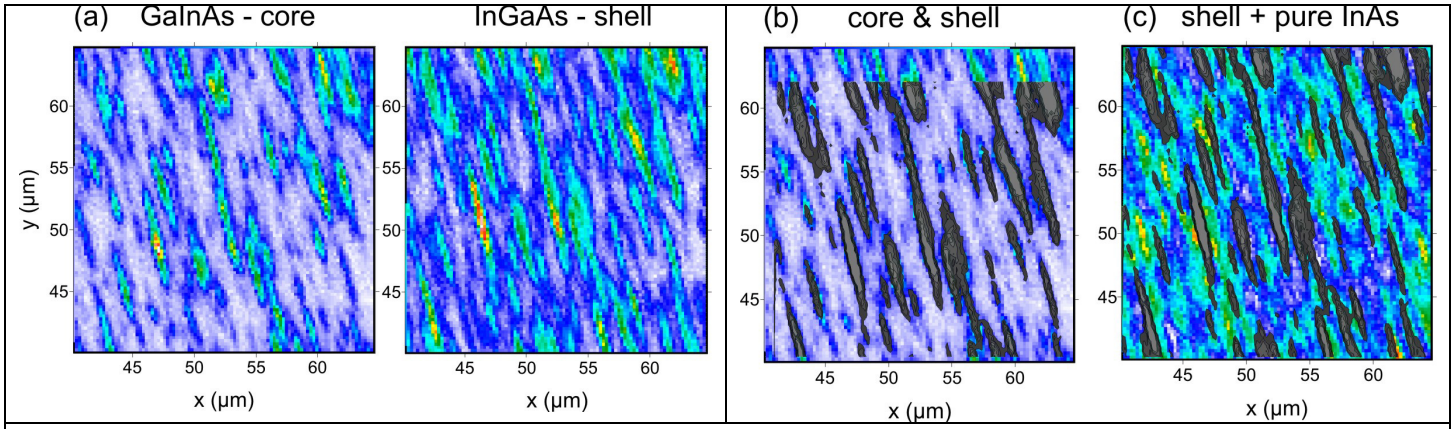

Fig. 3: (a) Spatially resolved diffraction intensity of the GaInAs core (left) and InGaAs shell (right) – signals. (b) Overlay of the core and shell diffraction signals. Both distributions overlap nicely. (c) Comparison of the distribution of InGaAs from the NWs shell (grey) and the pure InAs signal. The InAs is growing on the surface in between the NWs.

In summary, the results show an intermixing of Ga and In at the heterointerface in GaAs / InAs core/shell nanowires. With increasing shell thickness and therefore longer growth time, larger amounts of In are incorporated into the GaAs core.

Using the nanofocussed beam together with fast scanning of large sample areas, pure InAs was identified to grow in between the initial nanowires.