ESRF	Experiment title: "Analysis of strain-accommodation in the initial stage of self-induced MBE growth of InAs nanowires on Si [111] using a nano-focussed X-ray beam "	Experiment number: SI-2207
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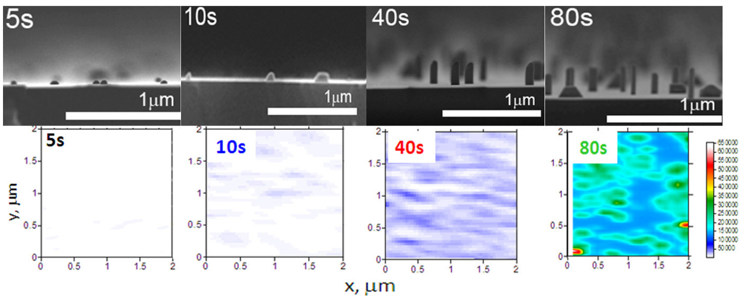
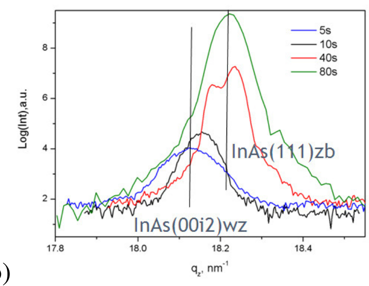
Report: The aim of this proposal was to characterize the structure of single InAs nanowires (NW) grown on Silicon (111) substrate using the nano-focus setup at ID1. In particular, we were trying to understand the process of lattice parameter accommodation at the NW to substrate interface considering the huge lattice mismatch of 11%. Samples have been grown by catalyser-free MBE at Paul-Drude-Institute for Solid State Electronics in Berlin with number density of about 1 NW/µm². However, due to statistical character of growth each sample consists of NW with different heights and diameters. Since the strain release may depend on NW size we had to perform measurements at single NWs.

For the reporting experiment we used samples with growth time of 5s, 10s, 40s and 80s. The respective SEM pictures are shown in Fig.1a(top).

Nano-focussing at ID1 was achieved using Fresnel Zone Plate focusing the incident beam to a spot size of about 300x300 nm². This was sufficiently small in order to separate individual NW´s in coplanar symmetric scattering geometry. For recording the diffraction intensity, we used a 2D pixel detector, MAXIPIX with pixel size of 55x55 μm^2. In order to identify the NW's positions we scanned the whole sample along x- and y- direction at fixed Bragg angle of the InAs(111) peak. As shown in Fig.1a, bottom we found positions with enhanced scattering intensity which we identified by NW positions. At these positions Rocking curves have been recorded. Fig 1b shows typical Bragg peak profiles taken at single NWs of the four samples under investigation. It reveals a shift of Bragg peak position as function of growth time. For samples with short growth times the intensity maximum appeared at smaller q_z- position, corresponding to a lattice parameter which we associate with that of wurtzite InAs (c=6.94Å). For longer growth time the peak shifts to a position known for zinc-blende type InAs. No additional peaks or slope intensity were found between the InAs and Si Bragg peaks. Thus we conclude that InAs grows already with its own lattice parameter onto the silicon substrate based on the inclusion of misfit dislocations at the NW to substrate interface. Additionally we found that InAs grows initially in wurzite phase followed by zinc-blende phase material for later growth time. Both phases are separated by stacking faults.



a)



b)

Figure 1. a) SEM pictures and position maps recorded at InAs(111) Bragg reflection
b) rocking curves measured from single wires

In order to determine phase content within individual NWs we measured asymmetrical reflections, specifically sensitive to either wurzite or zinc-blende phase units. By intensity reasons these measurement have been performed for the sample with 80s growth time. In order to identify these units across the sample again we recorded lateral space maps at the zinc-blende 331 and wurtzite 10i5 Bragg peak. Unfortunately the reproducibility of the sample

goniometer was not stable enough to scan exactly the same sample position for both reflections. The measurement of spatial intensity maps similar to 111 at these reflections required a positional stability of the diffractometer seen as small offset in the overlay of both maps shown in Fig. 2. However, one is able to identify NW positions fulfilling both zinc-blende and wurzite Bragg condition together but also those showing up a peak in one of the diffraction maps only. Figure 3 a,b shows reciprocal space maps recorded at the position indicated by red circle in Fig.2 measured at

331 and 10i5 Bragg reflection, respectively. Both exhibit a speckle like scattering pattern along the q_z direction through (331)ZB and (10i5)WZ reflections which is explained by a stacking of thin wurtzite and larger zinc-blende units within the NWs probed by the coherent x-ray beam. On the other hand, Fig. 3c shows the RSM taken at the position indicated by blue circle in Fig.2. The recorded RSM can be recorded at 331 zinc-blende Bragg peak only and shows a complete different shape compared to those shown in Fig.3a,b. From oscillations along q_z (Fig. 3c) with Δq_z= 0.06 nm^{-1} we determined the height of the object to be 104 nm which corresponds with the average height of parasitic island seen in between the NWs in Fig.1a.

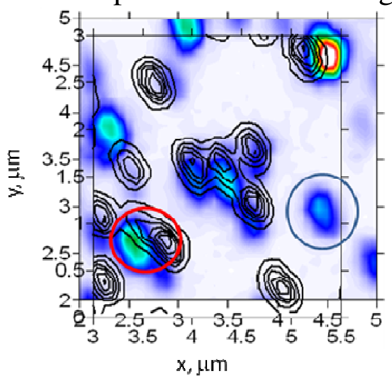


Figure 2. Overlay of position maps recorded at (331)ZB reflection (color map) and at (10i5)WZ reflection (counter map)

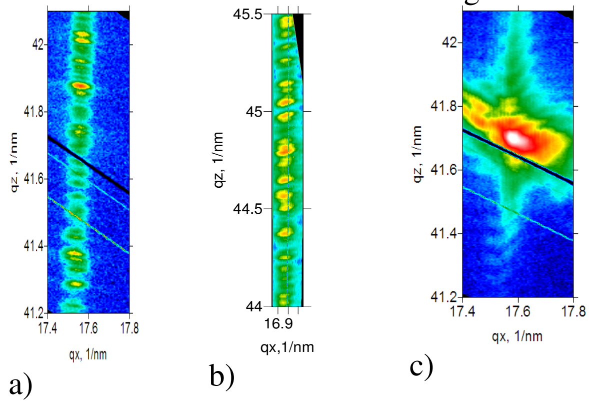


Figure 3. Reciprocal space maps re recorded: a) object exists on both position maps (marked with red circle on Fig. 3- (331) reflection) ; b) the same object (10i5) reflection; c) object exits only on position map recoded at (331)ZB reflection (marked with green circle)

Using RSM measurements at asymmetric reflections we could identify two types of nano-object - NW's and parasitic islands.

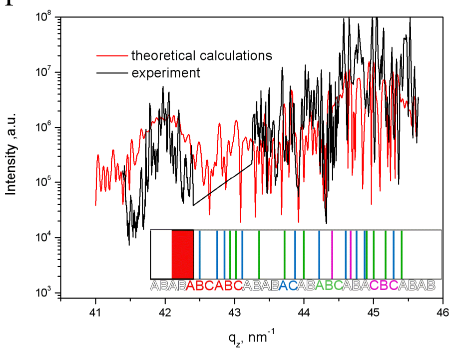


Figure 4. Theoretical calculations of intensity distribution along the NW and schematic representation of calculated model

Following Favre-Nicolin [3] the speckle maps can be retrieved to determine the stacking sequence of wurtzite and zinc-blende unit along the growth direction. The fact that the pattern differ from NW to NW tells us that the stacking differs among the NWs. Figure 4 represented first results of speckle simulation at one of the measured NWs and the respective scheme of phase composition according calculations containing wurtzite and zinc-blende units with different stacking sequences separated by stacking faults. The calculation is not finished yet.

Our experiment demonstrates the capability of phase analysis at single NWs without additional sample preparation. This is an essential contribution for the better understanding of the growth mechanism of MBE grown semiconductor nanowires.

Reference

[1] E. Dimakis, J. Lähnemann, U. Jahn, S. Breuer, M. Hilse, L. Geelhaar, H. Riechert, *Cryst. Growth Des.*, 2011, 11 (9), pp 4001–4008

[2] A. Davydok, E. Dimakis, A. Biermanns, S. Breuer, L. Geelhaar, U. Pietsch, PDI Topical Workshop on MBE-grown Arsenide Nanowires, Berlin, 8-9.09.2011, poster

[3] V Favre-Nicolin et al., New Journal of Physics 12 (2010) 035013