ESRF	**Experiment title:** Investigation of semiconductor nanowires using extremely focused x-ray beams	**Experiment number**: SI-1785
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Report:

Aim of the experiment was the investigation of a single InAs or InAsP nanowire grown epitaxially on a Si(111) substrate using a focused x-ray beam. With a focus diameter in the order of the wire diameter, it should be possible to move up and down the NW and see the different strain state in the relaxed main part of the NW and the strained contact area to the substrate. The experiment has several critical aspects regarding sample and setup:

- focus size of few 100 nm. We used a KB mirror optics, resulting in a beam spot FWHM of 300×300 nm^2. This fits well 100-200 nm diameter of the NWs, the setup worked very well.
- Alignment of the sample with 10 nm or better "precision", meaning that both stepping accuracy and stability during measurement times of typically one to few hours need to be in that range. The precise alignment is achieved with piezo stages, so the available step width was even much better. The stability of the spot was problematic, especially during scans of the sample rotation, or if one piezo stage was moved along the wire while in the other direction the sample should not move. Here the position was sometimes lost by some 100 nm, but effects were not reproducible. This point will very probably improve once the final stage setup is available.
- Stability of the sample (against radiation damage) during the measurement time. Here several effects have to be considered: there might be oxidation/etching of the sample due to ozone produced in the intense beam. Ionization of electrons by the x-rays might facilitate defect nucleation or propagation within the NWs, especially in regions where strain does exist. In previous experiments, the samples have been kept in a He environment. This was not possible this time, as so far no He environment compatible with the restrictions concerning space, required sample movements, and elimination of vibrations, was available. As a workaround we used a small pipe to flush a small current of He onto the sample during the experiment. How much this reduces the O_2 content in the sample environment is, however, unknown.

We have performed x-ray diffraction for two different samples: Pure InAs NWs on Si(111) and InAs NWs on Si(111) covered by a InAsP shell with about 2.5% mismatch. First we aligned the Si (333) and InAs (333) reflection, and scanned the sample to locate individual wires. Then the position in direct space was "fixed" to one NW at a time, and rocking scans were performed. Figure 1 shows two such scans, taken in the center of

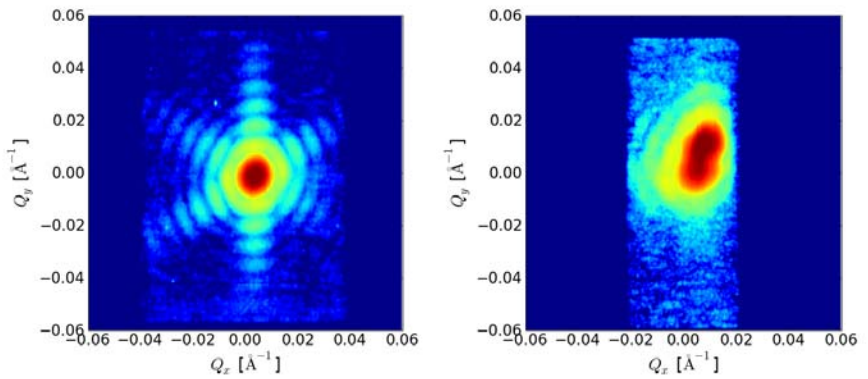

Figure 1: left: Reciprocal space maps taken from a single NW, exhibiting streaks due to the hexagonal wire cross-section and finite size fringes due to the finite NW diameter of about 150 nm. In this case, the focused beam was aligned to hit the middle part of the NW. right: the same scan with the beam aligned in to bottom region of the NW, i.e., close to the NW/substrate interface, where strain fields and/or defects lead to distortions of the crystal lattice.

the NW and at the wire/substrate interface. Clearly there are some differences visible. This is expected, since at the interface strain due to mismatch and due to defects accomodating the mismatch are likely to occur. However, it turned out that changes occur also if the same scan is repeated several times. We believe that sample movements in the order of 100-200 nm are responsible for these irreproducibility. While such movements are rather uncritical if the beam hits the homogenous part of the NWs, they cannot be neglected if the beam should stay at the wire/subtrate contact region. In addition, beam damage as stated above and illustrated in the SEM image of two investigated NWs in Fig. 2 certainly has an effect on reproducibility of measurements.

Conclusion

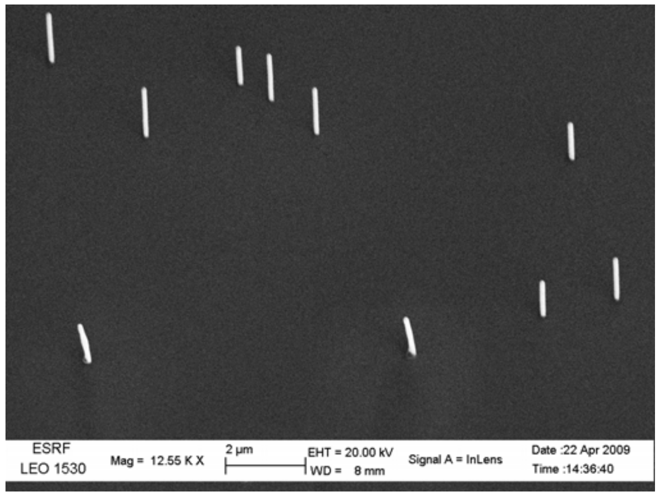

Figure 2: SEM image of the region scanned with the focused x-ray beams. Wires no. "1" and "2" are clearly bent. They were in the x-ray focus for several hours, while the other wires have "seen" the intense x-ray beam only shortly during alignment scans.

The current setup of ID13 is already very stable and reproducible compared to other beamlines. Still, several things need to be improved in the experimental setup to allow for delicate nanodiffraction experiments:

- Sample environment: a small He-filled or evacuated chamber would prevent beam damage to the samples, at least as far as oxidation is concerned. Since such a chamber will limit the accessibility of the sample by the optical microscope, and chamber designs with a constant gas flow will probably not be compatible with vibration minimization, a good design, eventually based on a one-time evacuated and sealed chamber, has to be worked out.
- The origin of residual mechanical instabilities needs to be clarified. Possible reason are temperature fluctuations in the hutch, mechanical influences between the motors, or sub-micron "play" in the bearings. Particularly suspicios during this beamtime was a rather stiff cable to the uppermost piezo-stage, which shall be replaced once the hexapod design has been implemented. The latter will also give important degrees of freedom for sample alignment, as tilt and azimuth can be aligned. A small stage for larger azimuth variations would be very convenient for x-ray diffraction experiments and should be included into the setup.
- During this experiment, no detector arm was available, but the FRELON camera was mounted on a X95 frame setup, and had to be mechanically shifted in order to change Bragg reflections. This is not very problematic for the FRELON, as this detector has a large field of view. However, especially for experiments exploiting coherence, small area pixel detectors like the MAXIPIX (recently available at ID13, small area: 14x14µm^2) will be used in future experiments. In this case manual detector setup will become practically impossible. A setup with at least some linear stages, preferrably also a rotation for the detector, would facilitate diffraction experiments and reduce considerably alignment times.

The aim of the experiment was not completely reached. While the alignment of single NWs into the focused beam and the recording of 3D reciprocal space maps was accomplished successfully, scanning along the NWs could not be realized reproducibly due to limited stability and radiation damage effects.