Nanowire Semiconductor Heterostructures diffraction

Exp. SI1309, Starting date: 30/06/2006, 12 shifts:
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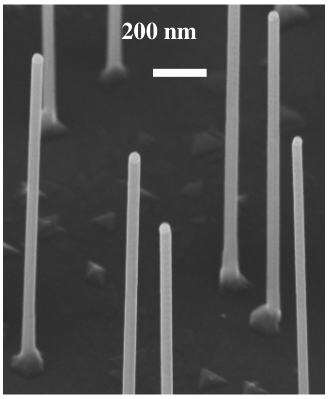
The development of vertical semiconductor nanowires (NW) as new building blocks for future nanoscale electronics and photonics devices is strongly dependent on the growth control and size parameters. To characterize the structural properties, Transmission Electron Microscopy (TEM) is a standard tool that gives first-rate information about individual objects or small assemblies including the determination of the growth directions, heterostructure cross-sections, surface facets, dislocations or stacking faults. In spite of their very strong complementarities with TEM, X-ray diffraction techniques are up to now very little used. The aim of these experiments was to study on epitaxial NW arrays (with random position) the average information about growth-related defects (in particular staking faults), orientation distribution and strain. GIXRD mappings have been performed with anomalous measurements using the nice equipment of ID01.

We have studied the following systems that present interesting features for electronic applications (see the European project NODE http://www.node-project.com/).

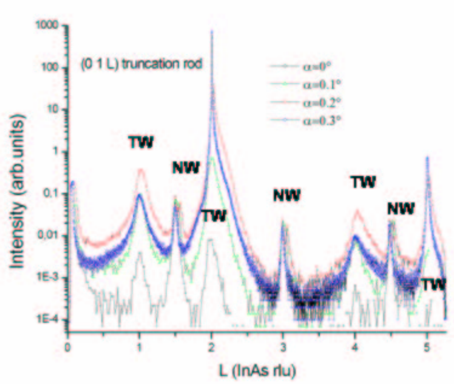
1. NW Heterostructures of InAs/InP on InAs(111) substrate (CBE, Lund, L. Fröberg).

We have shown that quantitative information can be obtained concerning epitaxial relationships, orientation distributions, strain relaxation and stacking defects in samples grown by Chemical Beam Epitaxy (CBE) and Au aerosol catalysts. It consists of 300 nm InAs/20x(20 nm InAs/10 nm InP)/100 nm InAs on InAs$(111)_B$ substrate (see Fig. 1 (a)). The measurements of the crystal truncation rods (CTR) allow determining the phases present in the sample. Fig. 1 shows a CTR example (b) as the function of the grazing angle and the reciprocal lattice (c) deduced from all the CTR measurements with (H K L) Miller indexes corresponding to the InAs $(111)_B$ surface unit cell. The InAs substrate has the standard ABCABC… stacking of the fcc (zinc-blende) structure and the NWs are hcp (wurtzite) without stacking defects according to TEM observations. The TW-peaks shown in Fig. 1 (b) are consistent with twins in a cubic phase similar to the InAs substrate. They probably correspond to the pyramidal hillocks observed by SEM. Interesting effects of multiple scatterings leading to the peak doubling as the function of the angle have been observed and explained with the Distorted Wave Born Approximation (DWBA). No superlattice contribution (resonance) has been observed. The lost of phase between the InAs/InP periods comes probably from the catalyst particle size distribution (affecting the period) and from different starting time for the NWs (a position-sensitive nucleation delay may occur before the NW growth). These causes are also worsened by growth defects like disorientations, stacking faults, section variations and strain relaxation distributions. This lack of signal constitutes a severe drawback for anomalous measurements analysis; we will submit a new proposal with optimized sample to have insight on the interdiffusion in nanowire heterostructures.
The strain measurements are obtained by in-plane and out-of-plane Bragg peaks measurements (radial-scans) and standard epitaxy is checked with $<11\bar{2}>$ side facets (referred to the cubic substrate). The average NW disorientation can be measured with this technique. The two mosaicities corresponding to the tilt and twist are estimated by in-plane and out-of plane transverse scans.



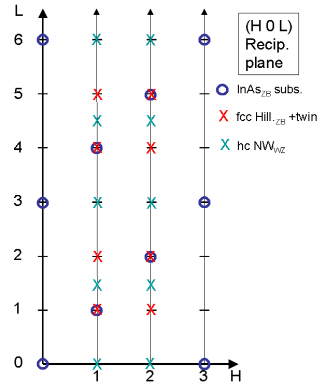
(a)

Fig. 1: SEM image of NW heterostructures grown by Lund (CBE).



(b)

(0 1 L) truncation rod measurement for several grazing incidences (0-0.3° range). Reciprocal space is indexed with respect to the substrate lattice unit cell.



(c)

Schematics of the (0 K L) reciprocal plane of the nanowires assembly.

Complementary experiments (under analysis) concerning the disorientations and relaxation effects have been performed with GIXRD reciprocal space mapping and anomalous measurement.

Fig. 2 shows an example of mapping close to the (0 1 L) CTR in agreement with Fig. 1 (c). The nature (simulation) of the streak doublings (for example at L=1.5) is under analysis.
Fig. 3 shows the same behavior with larger L variation.
Such measurements have been performed at different energies (11.85, 11.876, 11.89, 11.926 keV) close to the Au L3 edge energy.

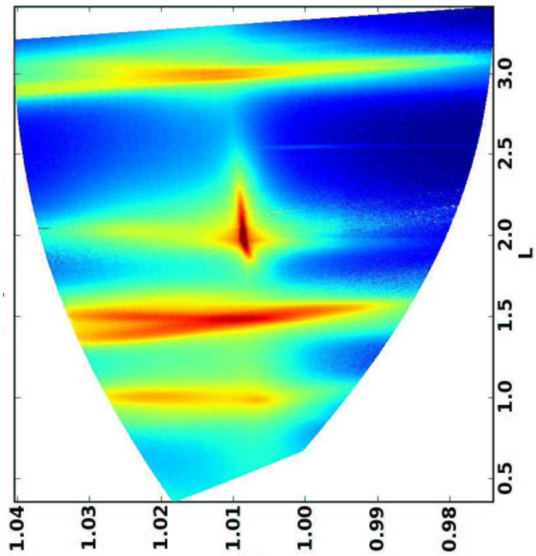


Fig. 2: Example of K-L mappings along the (0 1 L) CTR for an angle of incidence of 0.2° and E=11.850 keV. (Color is needed).

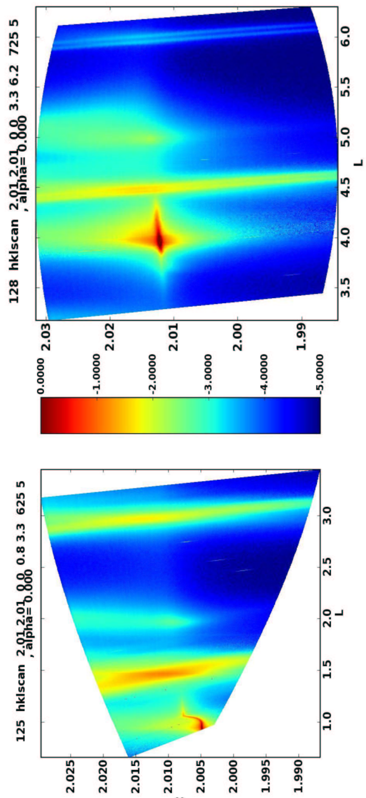


Fig. 3: Example of K-L mappings along the (2 0 L) CTR for an angle of incidence of 0.2° and E=11.850 keV. (Color is needed).

These data should give a deeper insight about stacking faults in the NWs and open the way to a real quantitative analysis of the nanowire growth-related defects with the GIXRD mappings.

2. GaP on Si(111) substrate (MOCVD, Lund, T. Mårtensson).

In this system, the NWs have the cubic structure with a lattice parameter very different from the silicon substrate. They are grown by MOCVD with Au catalysts. We have performed GIXRD to obtain epitaxial relationship, phase determination (including overgrowth that is larger than in the first case) and orientation distribution as presented before. The originality of this system comes from the stacking fault occurring in GaP NW that give local hcp structure (the stacking fault energy is small). For a very low stacking fault probability in the fcc structure, the reciprocal lattice correspond to the superposition of twinned orientations (similar to the overlayer contribution of the previous system). For larger probabilities of stacking faults, CTR corresponding to H=1, 2 are strongly affected and the Bragg peaks are transformed to diffuse streaks whereas CTR are H=3*n remain invariant. We have worked on the measurement and quantitative analysis to evaluate the fault probability on a very large number of NW. This statistical information, which cannot be obtained by TEM measuring a small number of NWs is very important to understand how to minimize the stacking fault density during a process. The first part of the experiments (simple HKL scans) was very successful, but a sudden power shutdown of a large part of the ring has abruptly stopped the mapping experiments about 2 shifts before the end (the good one of course…). It could be nice to have beam time to finish the study of this system.

Conclusions: We have shown with two systems that epitaxial nanowires can be studied by GIXRD, and that their signals can be well separated from quasi-2D overgrowth that can occur on the substrate. Important information is obtained about epitaxial relationships and stacking faults. The study of average strain has been performed, but we need to optimize the heterostructure features to have superlattice peaks in order to analyse strain distribution by anomalous scattering.
Such measurements will be important in the future to check the average quality and distribution of assembly of nanowires, for example in resonant devices (sensors or optical) where high quality factors are required, but also in transistors where a slight fluctuation of structural features has a strong impact on the device.

We have already started to publish the work related to this experiment:

- *Grazing incidence X-ray measurements of epitaxial InAs/InP nanowires.*
J. Eymery, F. Rieutord, L. Fröberg, T. Mårtensson, L. Samuelson, submitted to Appl. Phys. Lett.
- *Nanowire-based One-Dimensional Electronics.*
C. Thelander, P. Agarwal, S. Brongersma, J. Eymery, L. F. Feiner, M. Kamp, A. Forchel, M. Scheffler, W. Riess, and L. Samuelson, to be published in MaterialsToday November (2006).

A poster will be presented in September about this work in the Semiconductor Nanowires Symposium (http://www.extra.research.philips.com/nanowires/index.htm):

- *Grazing incidence X-ray measurements of epitaxial nanowires.*
J. Eymery, F. Rieutord, V. Favre-Nicolin, L. Fröberg, T. Mårtensson, L. Samuelson.