CRG France	Experiment title: Epitaxie et relaxation élastique dans des nanofils de semi-conducteurs – Epitaxial relationship and elastic relaxation in semiconductor nanowires	Experiment **32 02 643**
Beamline: BM32	**Date of experiment**: from: 15/03/06 (8h00) to: 19/09/04 (8h00)	**Date of report**: 05/10/07
Shifts: 12 allocated	**Local contact(s)**: J.S. Micha	*Received:*

Names and affiliations of applicants (* indicates experimentalists):

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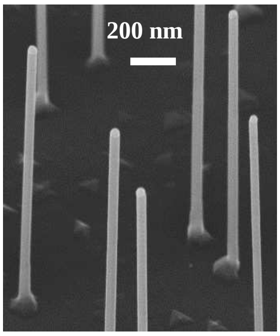
L. Fröberg, T. Mårtensson, L. Samuelson (Lund University, Sweden).

The aim of these first experiments on BM32 was to study epitaxial NW (NanoWires) arrays with random position of the catalysts to get average information about growth-related defects (particularly the staking faults-SF), orientation distribution of epitaxial relationships and strain. We have studied NW samples coming from the Lund University, one of the leader groups in Europe working in this field. The studied systems present interesting features for electronic applications (see the European project NODE http://www.node-project.com/) and are known for their high structural quality. The “multitechnique goniometer” has been used to test GIXRD and SAXS techniques to have insights on these objects that tend to impose as a real new building block for microlectronics.

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

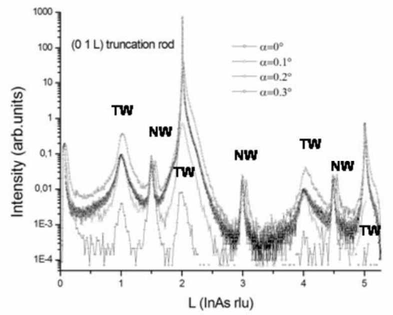
Quantitative information can be obtained concerning epitaxial relationships, orientation distributions, strain relaxation and stacking defects in samples grown by Chemical Beam Epitaxy (CBE) and Au aerosol catalysts. The sample is composed by the growth of 300 nm InAs/20x(20 nm InAs/10 nm InP)/100 nm 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 correspond surely 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.

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.



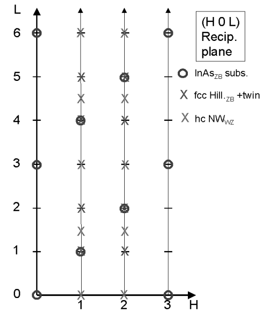
(a)

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



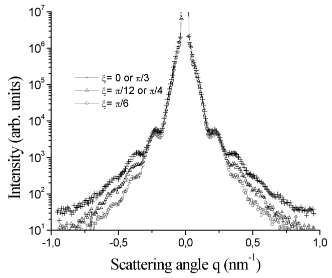
(b)

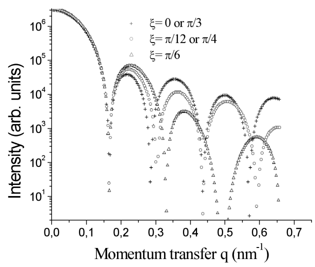
(0 1 L) truncation rod measurement for several grazing incidences (0-0.3° range). L is in surface lattice cell unit.



(c)

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





First SAXS measurements have been performed. They show the dependence of small angle scattering as the function of the azimuth ζ (left) in agreement with the hexagonal cross section (ζ=0 perpendicular to an edge) and with DBBA calculation for this shape (right).

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 SF density during a process. Such measurements should be continued, the problem of SF generation being observed in several systems (see French GDR Nanofils-Nanotubes).

Conclusions: We have shown 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. First SAXS experiments show that the shape (including complex faceting in heterostructures) of the NW should be studied by GISAXS to give an estimation of geometrical parameter fluctuations and facets indexation.

References related to this work:

- *Grazing incidence X-ray measurements of epitaxial InAs/InP nanowires.*
J. Eymery, F. Rieutord, V. Favre-Nicolin, O. Robach, 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, MaterialsToday **9** (10) (2006) 28-35.

- *Grazing incidence X-ray measurements of epitaxial nanowires.*
J. Eymery, F. Rieutord, V. Favre-Nicolin, L. Fröberg, T. Mårtensson, L. Samuelson. Poster presented in the Semiconductor Nanowires Symposium (http://www.extra.research.philips.com/nanowires/index.htm).