



GLANCING-INCIDENCE DIFFRACTION ANOMALOUS FINE STRUCTURE OF INAs/INP SELF-ASSEMBLED QUANTUM WIRES

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Anomalous X-ray diffraction at glancing angles is shown to be an appropriate technique to study the inner part of nanostructures self-assembled in a very thin epilayer. The site and chemical specificity of anomalous diffraction has permitted us to probe atoms of the quantum wires only and not those belonging to the oxide layer or to the substrate.

InAs quantum dots or quantum wires provide attractive quantum properties for optoelectronic devices like micro-laser sources [1]. They are grown by molecular beam epitaxy and formed during the first steps of growth by a "spontaneous" self-ordering process driven by the strain due to the lattice mismatch between the substrate and the epilayer. The resulting objects are on the nanometre scale. They are characterised mainly in terms of optical (luminescence, photoreflectance) and vibrational properties (Raman), while AFM (atomic force microscopy) and TEM (transmission electron microscopy) give an image of the shape and an idea of the spatial homogeneity. However, the inner composition is still not well known, as mixing with the substrate

atoms and segregation mechanisms of the impinging species could occur during the epitaxial growth.

We have studied InAs/InP(001) quantum wires by Diffraction Anomalous Fine Structure (DAFS). This spectroscopic method offers the advantage of providing the local information of the chemical selective Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy with the spatial and site selectivity of X-ray diffraction. In such a way, we are able to obtain local information about the atoms that belong to the nanostructures.

Our sample consists of an array of quantum wires (Figure 1) aligned along

the [1-10] direction with a typical length above 5 μm , a height between 0.6 and 2 nm, a period of 20 nm with an equivalent InAs coverage of about 2.5 monolayers. The DAFS measurements were carried out at the French Collaborative Research Group beamline BM2 (D2AM). We measured the intensity at the maximum of two Bragg reflections of the quantum wire array, near the 420 and 440 InP substrate Bragg peaks, as a function of energy, around the As K-edge (11.867 keV) (Figure 2). The measurements were performed in glancing-angle geometry, with an incidence angle kept constant close to the critical angle of InP (about 0.2°). This lets us collect the diffracted photons from an extremely thin quasi-surface layer enhancing its contribution to the diffracted intensity in comparison with the substrate contribution.

The data analysis is a two-step process. First, we performed a simultaneous crystallographic fit to the lineshape of the smooth part of the two DAFS spectra (Figure 2) refining the P concentration, $1-x$, for $\text{InAs}_x\text{P}_{1-x}$, Debye-Waller factors and experimental factors. We introduced corrections to the calculated diffraction intensity in the framework of the Distorted Born Wave Approximation (DBWA) [2] such that, in the total reflection regime, the thin epilayer is a small perturbation for the evanescent X-ray wave that penetrates a few nanometres into the refracting medium. Another fit parameter was the

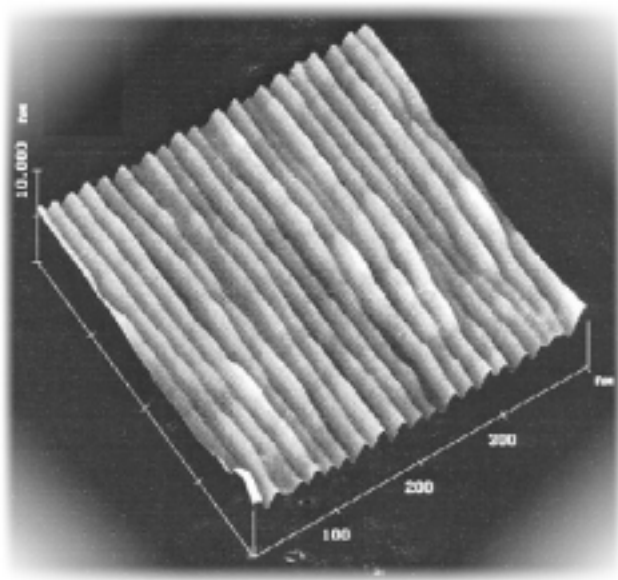


Fig. 1: AFM tridimensional view of InAs quantum wires on InP buffer [7].



thickness of an oxidized dead layer that was found $15 \pm 5 \text{ \AA}$ thick. To reproduce the shape of the anomaly at the edge, we had to include P atoms, $1-x = 0.5 \pm 0.1$: a considerable amount of P atoms is contributing to the satellite diffraction peaks, showing the same periodicity of the wires. However, at this stage of the analysis, we are not yet able to discern whether the P atoms belong to the wires or to the InP substrate which could show a periodic modulation as well as periodic strain spots due to the strain with the InAs wires.

In a second step, we extracted the Extended DAFS oscillations that appear after the edge (Figure 3) and we analysed them according to an EXAFS data processing scheme to get local parameters such as distances and populations [3]. Theoretical multiple-scattering EXAFS signals were calculated by the FEFF code [4] simulating the fine structure signal from an As atom inside a cluster containing In atoms, As (x) and P (1-x) atoms. The polarisation of the incoming photons was perpendicular to the surface, so the As and P next nearest neighbour atoms contribution to EDAFS is due only to the out-of-plane atoms. Refinement of the data was completed by the least-square fit procedure of the FEFFIT program [5] (Figure 3a and 4a). The relevant results are the P concentration, $(1-x) = 0.4$, and the As-P distance, found at 4.17 \AA . While the As-As distance was fixed at 4.29 \AA , the P content is, within the error, equal to the value found by the crystallographic fit of the anomalous diffraction lineshape.

The two independent analyses detected a consistent amount of P atoms: $(1-x) = 0.4-0.5$. In a previous study of $\text{InAs}_{0.5}\text{P}_{0.5}/\text{InP}$ superlattices samples [6], the out-of-plane As-As and As-P distances were found much closer to each other, about 4.28 \AA and 4.25 \AA respectively. In our analysis, the As-P distance is 4.17 \AA , close to the P-P distance in bulk InP (4.15 \AA), therefore, we could exclude the hypothesis of a fully relaxed InAsP epilayer. The P atoms contributing to EDAFS have to belong to the interface region, 0.5-2 monolayers, and the core of the quantum wire is essentially strained InAs. Two types of interface or a combination of both can explain the results: a) an abrupt InAs/InP interface with periodical strain strips generated in the InP buffer layer due to the interface mismatch strain; b) a

Fig. 2: Glancing-angle DAFS spectra of quantum wires, for (440) and (420) Bragg peaks, at the As K-edge (dotted curves) and crystallographic fits (continuous curves).

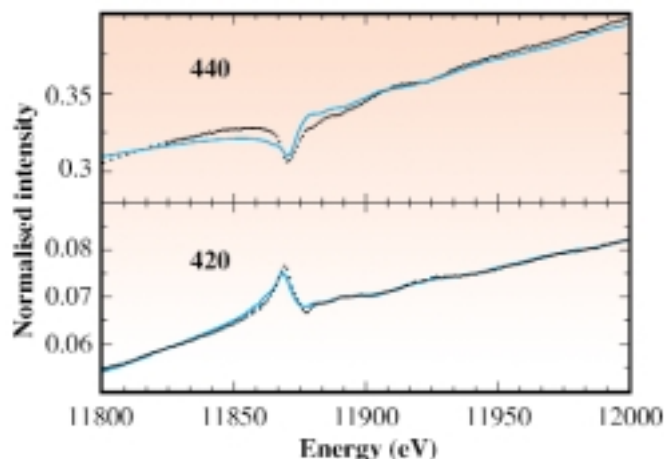


Fig. 3: (a) InAs quantum wire Glancing-angle EDAFS oscillations, after background subtraction, with best fit (continuous line), (b) EXAFS of the quantum wires. The curves have been rescaled for clarity.

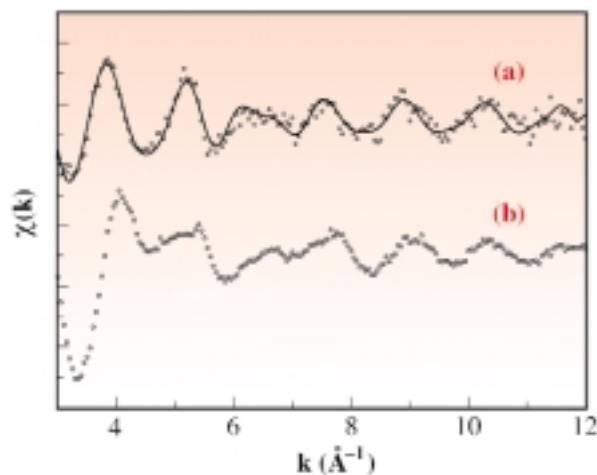
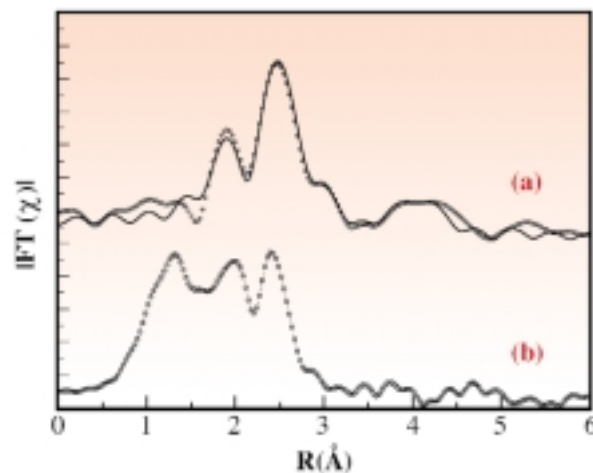


Fig. 4: (a) FT of quantum wire EDAFS, with best fit (continuous curve), (b) FT of quantum wire EXAFS. The curves have been rescaled for clarity.



corrugated InAs/InP interface with the same wire's periodicity.

We have also measured a glancing-angle EXAFS spectrum at the As K-edge (Figure 3b) at beamline BM8 (GILDA). The spectrum shows a clear As oxide shape with a strong low-frequency component that corresponds to a huge peak at 1.2 \AA in its Fourier Transform (FT) (Figure 4b). The oxide layer causes

a significant loss of information in particular for shells beyond the first one, whereas, for a DAFS spectrum, it lowers the overall diffracted intensity and the jump at the edge but it does not perturb the fine structure signal of the interesting atoms.

In conclusion, we have shown for the first time that DAFS can be applied in the glancing-angle regime to the study of



auto-organised nanostructured materials with an extremely low equivalent coverage. As the interface effects on the EDAX spectrum are remarkable due to the low epilayer thickness and to the X-ray beam polarisation, we were able to show, as a preliminary result, that the inner composition of the quantum wires is not a relaxed InAsP structure. Furthermore, we have detected a P-rich region at the interface with the same periodicity as the quantum wires. ■

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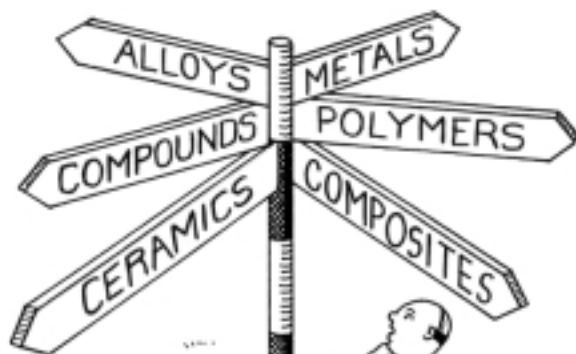
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