EUROPEAN SYNCHROTRON RADIATION FACILITY

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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

	Experiment title: **Quantitative 3D analysis of In concentration in InGaN/GaN quantum dots using grazing incidence anomalous diffraction**	**Experiment number**: HS 2010
Beamline:	**Date of experiment**: from:16/04/2003 to:22/04/2003	**Date of report**: 21/08/2003
Shifts:	**Local contact(s)**: O. Plantevin	*Received at ESRF:*

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Report:

Preliminary note: Change of sample and scientific background

The sample initially chosen for this experiment were InGAN quantum dots in a GaN matrix, synthesized at the CEA. However, a new "modified Stranski-Krastanov" method was recently developped at the CEA/SP2M to improve (Gogneau *et al.*, *J. Appl. Phys.* **94** *(2003),* 2254) the control over the growth of quantum dots, and notably the density of quantum dots.

Our experiment was therefore modified, in agreement with ID1 scientists, to focus on GaN quantum dots in a AlN matrix grown using this new method, around the Ga K-edge. The fundamentals of the experiment were otherwise not changed.

Samples studied

During our experiment we studied 4 different samples:

- 1: single layer of GaN quantum dots (QD), uncapped
- 2: single layer of GaN QD, capped by AlN
- 3: 10 layers of GaN QD, capped by AlN
- 4: 78 layers of QD, capped by AlN

The purpose of these measurements was to study the ***evolution of the quantum dots as a function of the number of layers deposited***, notably because PhotoLuminescence results suggest that the size of the QD increases with the number of layers.

All diffraction data was measured in ***grazing incidence*** mode, so that we would only be sensitive to the first layers (penetration depth around 1000 Å) of the sample where the QD are located. Also, we used a PSD as detector so that each 1D scan produced a 2D map in reciprocal space.

Anomalous diffraction measurements (1D)

After orienting each sample, we measured theta-2theta scans near the (300) (in-plane) and [302] (out-of-sample-plane) reflection. In figure 1 is shown the evolution of the scan around the [302] reflection, for the different samples.

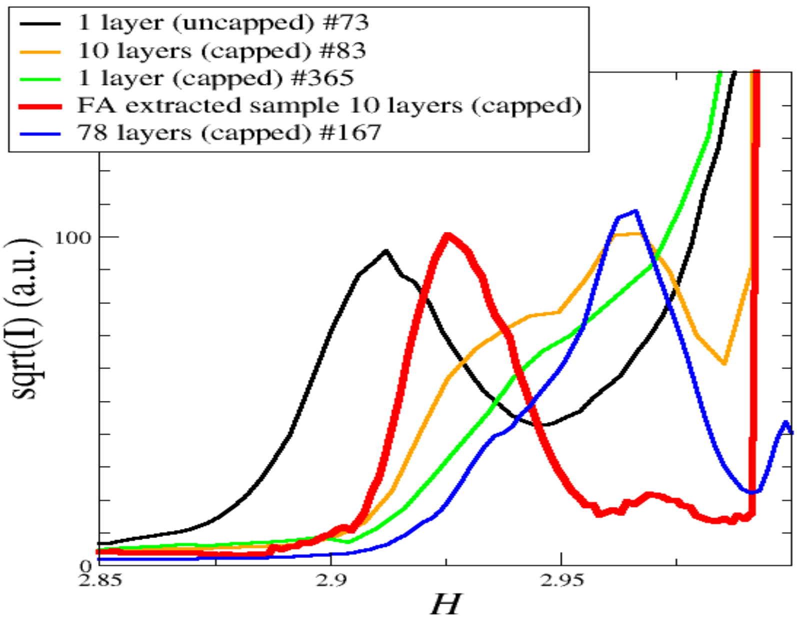

*Figure 1: theta-2theta scan near reflection [302]. Each scan consists of several peaks more or less separated. From left to right, the GaN peak (largest cell parameter, smallest diffraction angle), the AlN peak, and the intense peak beginning on the right is the SiC peak of the substrate. Also shown is the **extracted structure factor F_A** from the Ga, which can be obtained by analysing the evolution of the diffracted intensity as a function of the energy near the Ga K-edge. Only anomslous diffraction can thus separate the GaN diffraction from the AlN bulk.*

These scans where repeated at 9 different energies around the Ga K-edge, to measure the evolution of the diffracted intensity as a function of the energy. Analyzing these variations allows us to **extract the structure factor F_A of the resonant atom** (Ga), i.e. gives us the location of the diffraction from the QD in reciprocal space. In figure 1 is shown the extracted F_A for the 10-layers sample. From this extracted structure factor, we are able to **quantify the difference of relaxation between the uncapped QD and the buried ones**. With a standard diffraction measurement it would have been impossible to separated the contributions of AlN and GaN in the diffraction pattern.

Reciprocal space mapping

The reciprocal space maps collected allowed us to separate the contributions from the different parts of the sample (GaN QD, GaN from the wetting layer, AlN from the bulk, and SiC from the substrate- all have different cell parameters and therefore diffract at different positions in reciprocal space).

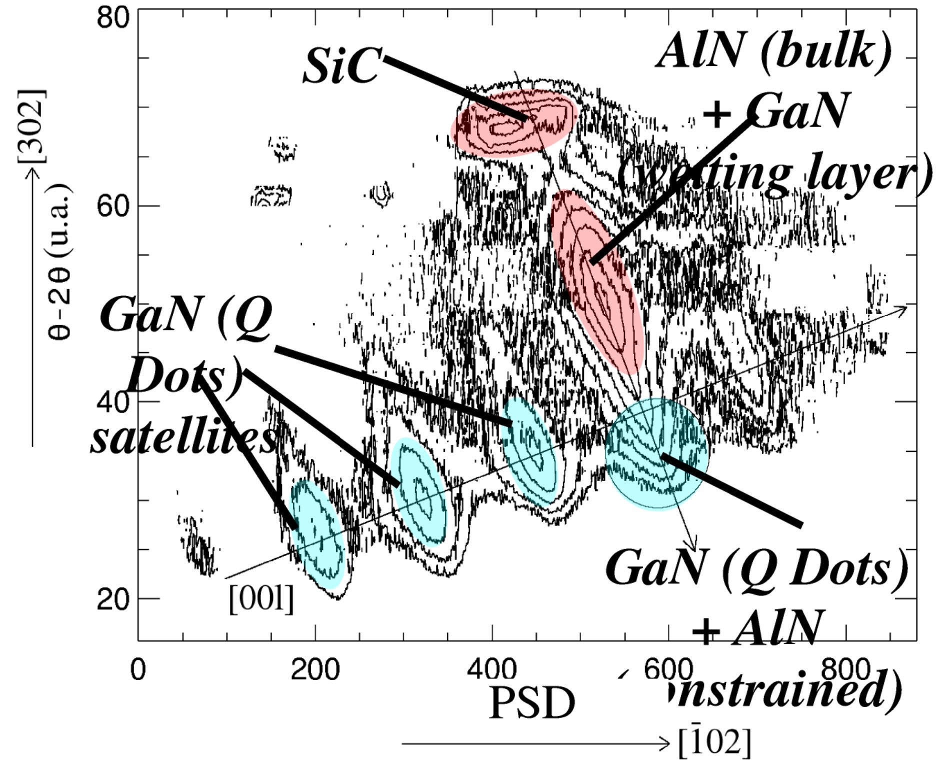

Figure 2 : reciprocal space map in the vicinity of the [302] reflection, for the sample with 78 layers of QD. The different peaks corresponds to parts of the structure with different strains, and at the bottom can be seen a series of stallite peaks indiciating a correlation between successive layers of QD

On the 78-layers sample we collected a "large" map around the (*302)* reflection, using several scans.This has revealed the existance of **correlation peaks** corresponding to the GAN (QD) part of the structure. Although a correlation was expected due to the short spacing between successive layers, the absence of associated correlation peaks corresponding to the AlN parts of the sample is yet fully unexplained, it may be due to a thickness effect : the AlN+wetting layer regions being thicker than the GaN/AlN regions.

Conclusion

We have measured anomalous diffraction scan and successfully shown that it can be used to extract the sub-structure of the QD thanks to the selectivity of resonant diffraction. The analysis is now focused on the determination of the strain values for the buried QD in the different samples (using the average diffraction angle), as well as the evolution of the size (using the size of the extracted F_A peaks). A careful analysis is required to take into account the complex absorption and transmission effects due to the grazing incidence geometry, which is dependent on the energy and therefore influences on the anomalous signal.

The correlation peaks observed will be analyzed using structural models taking into accounts strains in the material, and it will be necessary to perform another anomalous experiment to investigate the nature of the correlation peaks, specifically to determine if no AlN constrained by the GaN contributes to these peaks.