INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON



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:	Experiment
ESRF	embedded in GaN Matrices	HS- 3781
Beamline:	Date of experiment:	Date of report:
ID01	from: 12.06.2009 to: 16.06.2009	01.03.2011
Shifts:	Local contact(s):	Received at ESRF:
12	Dr. Rogerio Magalhaes- Paniago (email:paniago@esrf.fr)	
Names and affiliations of applicants (* indicates experimentalists):		
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Report:

InGaN quantum dots are used for high emittance photonic devices with a tunable output in the blue, green and ultraviolet spectral range. They are mostly grown by Metal-Organic Vapour Phase Epitaxy (MOVPE). The Indium incorporation in the InGaN quantum dots is very important for improving their luminescence efficiency and for tuning the wavelength. For a good device a GaN capping layer with high crystalline quality is required. One difficulty is that InGaN quantum dots grown in Stranski-Krastanow (SK) mode are dissolving during overgrowth [1]. Therefore, a new method has been introduced to form the quantum dots directly on the top of the buffer layer. A thin nucleation layer is deposited with a layer thickness below the critical thickness for island formation in SK mode [2].

We have measured a series of samples in order to study the growth evolution of InGaN quantum dots in different stages of formation process. The samples consist of a 2μ m thick GaN(0001) buffer layer deposited by MOVPE on the c-plane sapphire. On top of this buffer, about 2nm InGaN is deposited which evolutes into island-like structures (fig.1). Several samples have been capped by either InGaN formation layer with lower concentration of indium, or GaN. Additionaly, one sample has a 36nm thick capping layer of GaN on the surface. The growth conditions during growth are varied [2].

The experiments have been performed at the beam-line ID01, ESRF (synhrotron radiation energy of 7.75keV), using a MaxiPix 2D detector with evacuated flight tube in front. To avoid air scattering and damage of the samples due to reactions with ozone, the samples have been measured under helium atmosphere.



Fig. 1. Sample structure of a) freestanding and b) overgrown quantum dots. (an example)

We have performed grazing incidence diffraction (GID) which is sensitive to the lateral lattice parameter distribution. Measurements were done on the reflections from the planes sitting perpendicular to the sample surface. Samples were mounted vertically to use σ -polarization of the synchrotron radiation which gives more intense beam. Therefore, "hphi" motor was used as a sample "theta" and detector motor "del" as a "two thetha". In detail, radial and angular scans in grazing incidence geometry have been measured. Radial scan means performing the " θ -2 θ " scan in the direction of chosen reflection moving simoultaneously "hphi" motor by half of the step of "del" motor. Angular scan means scanning by moving just one motor "hphi", while "del" motor

stays at the fixed position fullfilling the Bragg condition. Position of "hmu" motor defines the incidence angle and "nu" brings detector in the position above the exit angle to collect intensity integrated in the z-direction. Furthermore, penetration depth dependend measurements in GID geometry have been performed in a sense that we have performed the same radial and angular scans applying a different angle of incidence. Additionaly, reciprocal space map $(q_{\text{radial}} - q_{\text{angular}})$ on few of the samples have been measured.

In GID geometry, (10-10) and (20-20) reflections were measured to compare two orders of diffraction. Each reflections was repeated in three different azimuthal orientations.

Fig. 2. shows two radial scans measured at different incidence angle, below (black line)



Fig. 2. GID (20-20) radial scan on the sample with InGaN FL measured at different angle of incidence, black – below and red – above the critical angle.

and above (red line) the critical angle. Below the critical angle, at α_i =0.25°, penetration depth was 2nm and above the critical angle, at α_i =0.5° was 200nm. Scattering from the vicinity close to the surface form a separate peak named InGaN(1) which is getting less pronounced changing the shape by getting deeper in the film.

On the figure 3. it is shown the comparison between the samples with same growth temperature, but different layer structure. Blue and green lines mark Bragg positions of pure GaN and pure InN, respectivly. On both diffraction profiles one can observe a sharp GaN peak coming from the thick buffer layer and well separated InGaN(2) peak corresponding to the alloy with nominaly 85% of indium. Scan on the sample with overgrown quantum dots (orange line) have a broad intensity distribution between these two peaks coming from overgrowing material of InGaN, which is even forming separate peak. Scan on the sample with freestanding quantum dots (black line) shows just a second component below the GaN peak what may indicate that quantum dots, which suppose to have 10-20% of In, are not completely strained to the substrate.

InGaN(2) peak is shifted to the position of GaN in both cases of overgrowing either by GaN or by InGaN.

We have measured angular scans (rocking curves) at the positions of the GaN, InGaN(1) and InGaN(2) peaks on all of the samoples and for both (10-10) and (20-20) reflections. strain. size The and misorientation components are overlaped with the change of composition and therefore we will develop a model of freestanding and overgrown InGaN quantum dots including different shape, size misorientation and fit calculated and intensity distribution to the measured one. Further, comparing the results of GID study to EXAFS and AFM results measured on the samples should help to answer on the real structure of investigated materials.



Fig. 3. GID (10-10) radial scans on the samples with different layer structure grown at the same temperature at 600°C: freestanding QDs – black line, overgrown QDs -orange line.

<u>References</u>

- [1] Pretorius A., et al., Phys. Status Solidi C 3(2006), 1679
- [2] Tessarek, Ch. et al., Phys. Status Solidi C, 1-4 (2009) / DOI 10.1002/pssc.200880913