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ESRF

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: The Kinetics of the Stranski-Krastanow Growth of GaN Quantum Dots on Top of AlN in Excess of Ga (MSK growth)	**Experiment number**: 32 03 640
Beamline: BM32	**Date of experiment**: from: 04.12.05 to: 04.19.05	**Date of report**: 10.02.05
Shifts: 21	**Local contact(s)**: Dr. G. Renaud	*Received at ESRF:*

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Report:

Grazing incidence anomalous x-ray scattering was used to monitor *in situ* the molecular beam epitaxy growth of GaN/AlN quantum dots (QDs). The strain state was studied by means of grazing incidence Multi-wavelength Anomalous Diffraction (MAD) in both the QDs and the AlN during the progressive coverage of QDs by AlN monolayers. Vertical correlation in the position of the GaN QDs was also studied by both grazing incidence MAD and anomalous Grazing Incidence Small Angle Scattering (GISAXS) as a function of the number of GaN planes and of the AlN spacer thickness.

Samples were grown on 6H-SiC(0001) substrates in a plasma assisted molecular beam epitaxy chamber installed on the SUV apparatus at BM32. The nitrogen flux was supplied by a radio-frequency plasma cell ; Knudsen cells provided the Ga and Al fluxes. Wurtzite GaN QDs were synthetized on top of AlN buffers *via* the modified Stranski-Krastanow mode, at ~ 730°C, with a GaN equivalent amount of 6 MLs. A grazing incidence and exit setup was used to enhance the scattered x-ray signals from the QDs with respect to that of the substrate. Diffraction anomalous measurements were carried out around the Ga K-edge (10.367 keV). For the MAD experiments reciprocal space scans along the [10-10] direction around h=3 were systematically recorded at 12 energies around the Ga K-edge using a point detector. The detector slits were opened so as to measure the integrated intensity over the grazing exit angle. A 2D charge-coupled device detector placed perpendicular to the incident beam was used for GISAXS measurements.

The progressive capping of GaN QDs by AlN was analyzed *in situ* by means of MAD, that is the Ga and AlN structure factors could be extracted (cf. Fig. 1) from the measurement of the diffracted intensities at 12 energies across the Ga K-edge. Figure 2 depicts the evolution of the in-plane lattice parameters deduced from the position of the maximum of the partial structure factors, in both AlN and GaN, as a function of the AlN cap layer thickness. It is clearly demonstrated that the AlN capping initially stressed by the QDs is then progressively relaxed. Two regimes can be distinguished, in which QDs and AlN capping are mutually influenced. A rapid decrease is observed until 16 MLs, followed by a more gentle diminution probably lasting after 34 MLs. Jointly the partially relaxed surface QDs are gradually compressed by the AlN capping untill 16 MLs, leading to a plateau. Interestingly, the AlN strain state varies only very slowly above an AlN thickness of < 30 MLs (7.5 nm). Under this semi-quantitative limit, the AlN tickness is such that the presence of buried dots significantly affects the AlN strain state, and a GaN QDs vertical correlation is expected.

Another grazing incidence MAD study has been carried out for a gradual QDs planes stacking. The AlN spacer was chosen in a regime of mutual QDs and AlN influence and fixed at 25 MLs. From the width of the structure factors extracted for 1, 2, 3, 5, 7, and 10 capped QDs planes, the size of the QDs was estimated according to the Debye-Scherrer formula. A 50% increase in the size of QDs is put in evidence, as a consequence of a vertical correlation effect.

The vertical correlation effects were further investigated by anomalous GISAXS. The incident angle was set slightly above the critical angle, for a sensitive to the whole stacking. Figure 3(a) shows GISAXS measurements for a stack of 5 QDs planes separeted by 25 AlN MLs. The occurrence of Q_z satellites at $Q_x \neq 0$ is an evidence for the QDs position vertical correlation along the [0001] direction. The strong energy dependence at the Ga K-edge of these satellites (Fig. 3(c)) confirms that the scattering yield is mainly from the QDs. Finally, fig. 3(b) shows a GISAXS map measured for a QDs planes stacking with 53 MLs AlN spacers. Compared to the case of thiner (25 MLs) AlN spacers (Fig. 3(a,c)) 53 MLs AlN spacers (Fig. 3(b,d)) seem large enough to inhibit the vertical correlation effects, likely because they are out of the regime of QDs and AlN mutual influence. This is consistant with the above conclusion that the critical thickness of the AlN spacer requested to allow correlation lays around 30 MLs.

The results were submitted to Appl. Phys. Lett. (http://arxiv.org/abs/cond-mat/0508126).

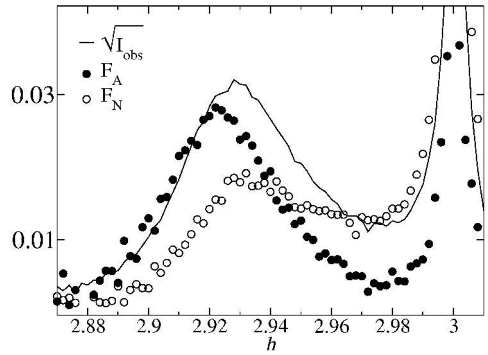

Fig. 1 : Square root intensity at 10.267 keV, Ga (F_A) and non anomalous (F_N) structure factors for GaN QDs covered by 8 MLs AlN.

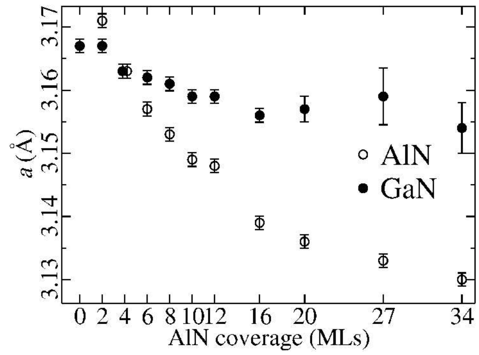

Fig. 2 : In-plane lattice parameter in AlN and GaN deduced from MAD measurements

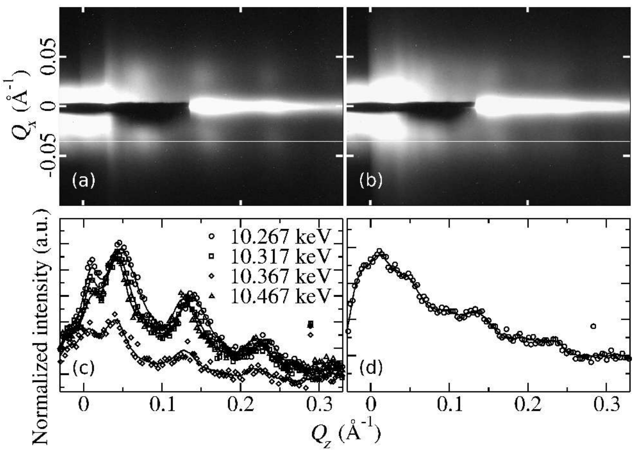

Fig.3 : GISAXS maps measured for GaN QDs / AlN multilayers with (a) 25 MLs and (b) 53 MLs AlN spacers, and corresponding ((c) and (d)) diffused intensity along Q_x=-0.033Å^{-1}, at different energies (c).