EUROPEAN SYNCHROTRON RADIATION FACILITY

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 via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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.

ESRF	**Experiment title:** Coalescence of GaN nanopillars grown by Nano-Pendeo Epitaxy	**Experiment number**: AO-2-878
Beamline: BM02	**Date of experiment**: from: 29/04/2022 to: 02/05/2022	**Date of report**: 28/02/2023
Shifts: 9 shifts	**Local contact(s)**: Gilbert Chahine	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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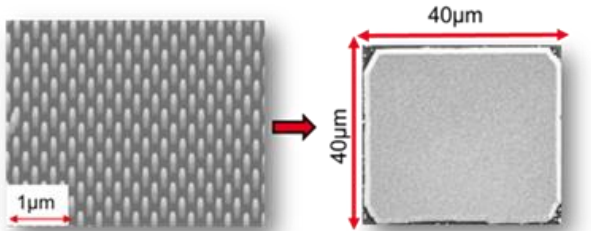
Report:

Introduction

Novel epitaxial structures of gallium nitride (GaN) are grown on top of GaN/AlN/Si/SiO_2 nano-pillars for optoelectronic applications such as microLEDs. The nano-pillars are intended to allow the independent GaN nanostructures to coalesce into a highly-oriented film thanks to the SiO_2 section becoming soft at GaN growth temperature. The nano-pillars as well as fully coalesced GaN platelets are presented in figure 1 (a) and (b). The objectives were to measure the variation of tilt and twist in both the GaN layers and silicon (Si) nano-pillars before and after coalescence and for different pillar to pillar distances (pitch).



(a) (b)

Figure 1: Scanning Electron Microscope images of (a) nano-pillars before coalescence, (b) single fully coalesced 40 × 40 µm^2 GaN platelet.

Experiments performed

To measure the lattice tilt in the Si planes, ω-scans of symmetric (111) reflections were performed as well as (331) reflections. To measure the twist in the Si planes caused by edge dislocations, the asymmetric (331) reflection was measured and the sample rotated around its normal axis Φ. For the GaN layers, we performed scans on the skew-symmetric (204) Bragg reflection to analyse the tilt and twist in the GaN crystallites. The incident beam arriving on the sample covered an area of two coalesced GaN structures each 40x40 µm^2 and these measurements were performed on a reference sample that has only nano pillars, i.e. prior to the pyramids growth, and on two coalesced 40x40 µm^2 GaN structures of pitch p=0.5µm and p=1µm respectively.

Figure 2: photo of the instrument at the BM02 beamline at the ESRF (red arrow is pointing at the studied sample).

Results and discussion

Our growth approach assumes a self-alignment of GaN layers during coalescence, which would cause a dis-orientation of the Si (111) nano-pillars. The results of the asymmetrical Bragg reflection Si (331) and the symmetrical Bragg reflection Si (111) measurements for reference sample A and coalesced sample B are presented in table 1 (a) and (b) respectively.

Full width at half maximum (FWHM θ) Si 111 /°	P=0,5µm	P=1µm
Sample A: GaN pillars	0,32	0,25
Sample B: Coalesced GaN structure	0,81	0,71

(a)

Full width at half maximum (FWHM Φ) Si 331 /°	P=0,5µm
Sample A: GaN pillars	1,1
Sample B: Coalesced GaN structure	2,5

(b)

Table 1: (a) Full width at half maximum (FWHM θ) of Si 111 measurements (b) Full width at half maximum (FWHM Φ) Si 331 measurements.

For the (331) reflection, the FWHM of the diffraction curve is broadened in sample B compared to sample A, from 1.1° for sample A to 2.5° for sample B for a pitch of 0.5µm. It is likely that the initial value of FWHM is strongly linked to instrumental resolution and the crystallite size and shape. The (111) reflection also shows an increase in FWHM from 0.32° for sample A to 0.81° for sample B for a pitch of 0.5µm and from 0.25° for sample A to 0.71° for sample B for a pitch of 1µm. The broadening of the two types of planes implies that the silicon layers are more twisted and tilted in the coalesced structures compared to the reference sample, as expected from our model.

It is interesting to also note that by measuring the GaN (204) reflection, we obtained values FHWM around 0.6° in the GaN layers of the coalesced structure. This relatively high value can be explained by the fact that with this technique we are scanning across two structures and not just one, thus the results presented correspond to clusters across two structures.

Figure 3 (a) shows the diffraction pattern from the 2D detector, with the y-axis corresponding to values of 2θ, and the x-axis corresponding to Φ. This image is from the GaN (204) Bragg reflection in the middle of a GaN platelet coalesced from pillars having a 0.5 µm pitch. We can clearly see that for a given value of θ and ω we have peaks at different values of Φ, as shown by the red arrows. This implies that we are diffracting zones or clusters of GaN with different values of twist.

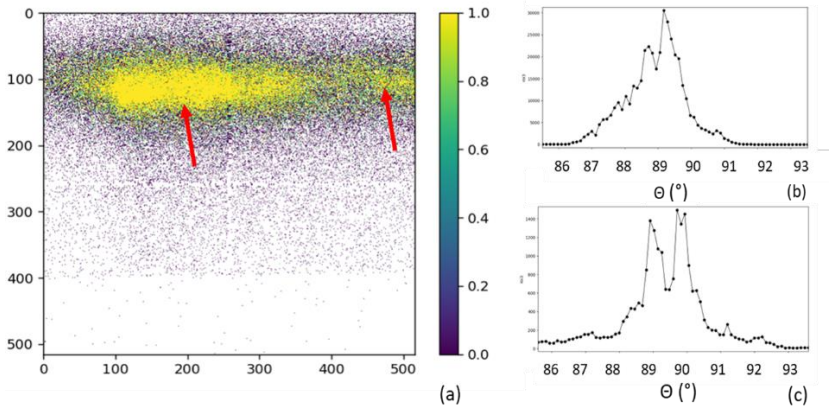


Figure 3: (a) 2D detector image of the GaN (204) Bragg peaks at one position in the GaN structure, with the x and y axis representing Φ and 2θ respectively. Intensity is displayed to the right of the image and two Bragg peaks at different values of Φ are shown with the red arrows, (b & c) rocking curves showing two Bragg peaks at different values of ω for pitch=0.5 µm (b) and pitch=1 µm (c).

In addition, figure 3 (b) shows the distribution of the intensity as function of ω for a given Φ value, and we can see the presence of more than 1 peak at different values of ω *e.g.* for pitch =0.5µm, we identify a peak at ω= 88.8° and at ω = 89.5°. This also implies that we are observing aligned clusters. Figure 3(c) shows similar results for the structures coalesced pitch of 1 µm.

These results are very beneficial to understand our growth approach and present an indirect proof of the self-orientation of the GaN pyramids during the coalescence. These findings along with others from the Dark Field X-ray microscopy measurements are the subject of an article, which has been submitted for publication in the journal of applied crystallography.