EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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 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:** DFXM study of the coalescence of GaN nanopillars grown by Nano-Pendeo Epitaxy | **Experiment number**: MA5175 |
| --- | --- | --- |
| **Beamline**: ID06-HXM | **Date of experiment**: from: 04/07/2022 to: 09/07/2022 | **Date of report**: 28/02/2023 |
| **Shifts:** 14 shifts | **Local contact(s)**: Can Yildirim | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

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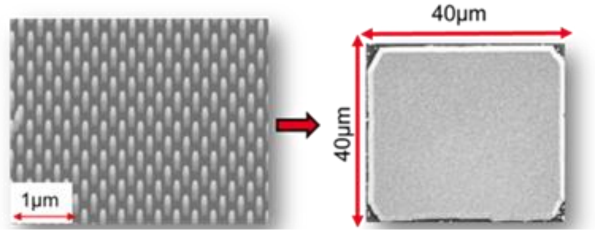
**Report:**

**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 a fully coalesced GaN platelets are presented in figure 1 (a) and (b).

The GaN coalescence process and the GaN layer quality were studied using Dark-field X-ray microscopy (DFXM) in order to understand optimize this growth approach for different patterns of pillars.




(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 understand the process of coalescence in a 2D array of crystallites, we start by studying the coalescence along lines (i.e. a 1D array) of 10 pillars with a pitch of 0.5µm shown in figure 3(a). Then, two 40 × 40 µm$^2$ GaN platelets were anaylzed, each with a different pitch value (distance pillar-pillar).

A strain ($\omega$-2$\theta$) and mosaicity ($\omega$- $\Phi$) map were performed with the $\omega$ incidence angle, 2$\theta$ the angle between the detector and the incidence ray and $\Phi$ the angle around the normal axis. A photograph of the experimental setup is shown in figure 2. The spatial resolution was 100 nm and the beam energy 16 keV. We measured the GaN (101) Bragg reflection to study the GaN structures using a diffraction angle $\theta$=9.16°.

For the ($\omega$- $\Phi$) scan we typically performed 40 rocking curves with a step $\Delta\Phi$ of 0.08° and $\Delta\omega$ of 0.02°.

For the ($\omega$- 2$\theta$) scan, we performed 35 rocking curves with a step $\Delta 2\theta$ of 0.004° and $\Delta\omega$ of 0.06°.

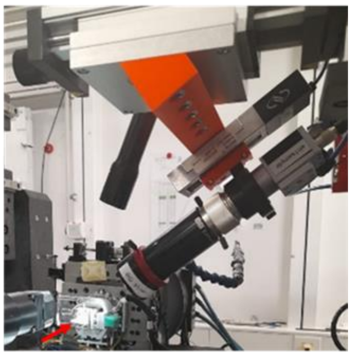


Figure 2: photo of the instrument at the ID06 beamline at the ESRF (red arrow is pointing at the studied sample).

**Results and discussion**

Figure 3(b) shows the center of mass (COM) map for the incidence angle ω obtained for the five lines obtained from a set of ω-Φ scans and extracted using the *darfix* library [1].

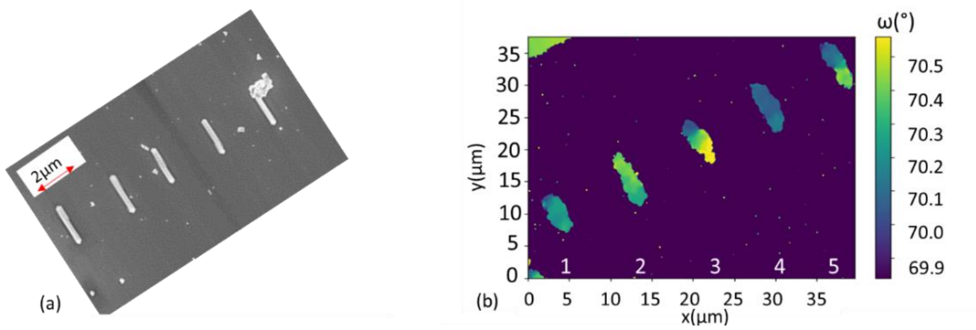



Figure 3: (a) SEM image of five fully coalesced GaN lines, (b) Centre of Mass map for ω of the 5 lines

The results show significant differences between the lines. For lines 1, 2 and 4, we have homogeneous lines with a very small ω variation along the line itself (Δω<0,1°) and values of the standard deviation in the order of 0.04°, implying that the GaN crystallites are very well oriented and that few, if any, coalescence boundary dislocations are required to accommodate any mis-orientation which is  a significant improvement. In contrast, lines 3 and 5 have strong ω variations (Δω>0,1°) along their length, implying the generation of dislocations at the interfaces between pillars.

Next, two GaN platelets were analyzed: structure 1 (figure 4(a)) with a pitch of 0.5 µm and structure 2 (figure 4(b)) with a pitch of 1µm. Figures 4(a) and (b) show the COM of the two structures for ω using the GaN (101) reflection from ω-Φ scans.

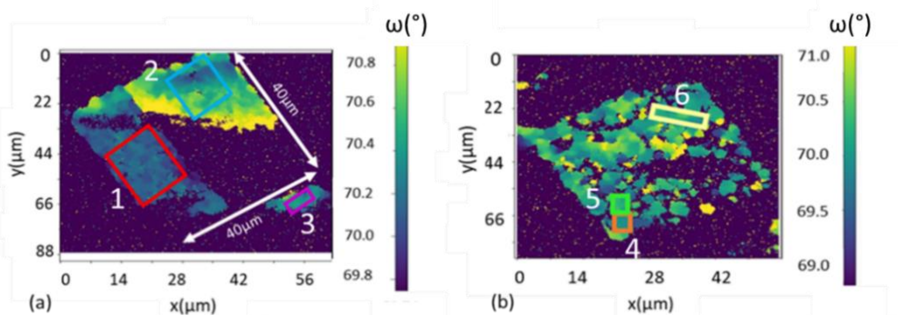



Figure 4: (a) Centre of mass map for ω of GaN structure 1 with pitch=0.5µm. (b) Centre of mass map for ω of GaN structure 2 with pitch=1µm.

From structure 1, we can see three separate areas. Clusters 1 and 3 in structure 1 are very well oriented areas, with standard deviations  of 0.04° and 0.07° respectively. These values are similar to those found in the homogenous lines discussed above. Cluster 4, 5 and 6 have standard deviation values of 0.07°, 0.08° and 0.11° respectively.

Although not uniform across the platelets, these results are extremely promising since we are able to achieve high quality GaN across areas greater than 10 × 10 µm$^2$ (Structure 1, cluster 1) with our growth approach. A paper discussing these results has been submitted for publication in the journal of applied crystallography.

The COM map of 2θ was extracted from a set of ω-2θ scans and a post treatment analysis allowed us to obtain the relative strain along the GaN (101) in the 40 × 40 µm$^2$ GaN platelet a shown in figure 5. We can clearly see that GaN platelets presents small values of strain with a maximum value around 2%. Further analysis for other GaN structures and lines will provide us with necessary information in order to understand the growth process.

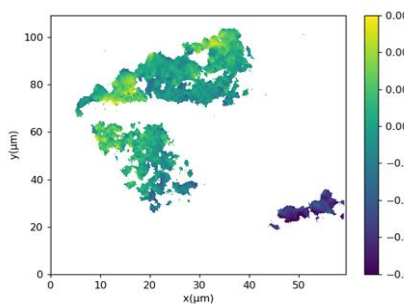



Figure 5: Relative strain in GaN along (101).

**Reference:**

[1] J. Garriga Ferrer *et al.*, “darfix: Data analysis for dark-field X-ray microscopy,” *arXiv e-prints*, p. arXiv-2205, 2022.