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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 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:** Study by nano-XRD of the coalescence of GaN nanopillars grown by Nano-Pendeo Epitaxy	**Experiment number**: MA-5376
Beamline: ID01	**Date of experiment**: from: 14/06/2022 to: 18/06/2022	**Date of report**: 28/02/2023
Shifts: 12	**Local contact(s)**: T. Schulli E. Zatterin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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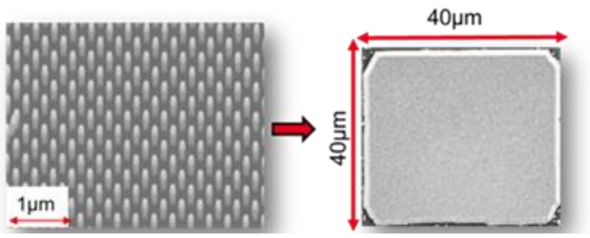
Report:

Introduction

The objective of the experiment was to comprehend a novel heteroepitaxial approach that relies on the coalescence of GaN pyramids grown on top of GaN/AlN/Si(111)/SiO_2 nano-pillars of 100nm diameter (figure 1a) into GaN 40 x 40µm^2 platelets (figure 1b). Our growth approach assumes a twist/tilt of the nano-pillars and a self-alignment of GaN layers during coalescence preventing the formation of grain boundaries and causing a dis-orientation of the Si(111) layer at the top of the nano-pillars. Therefore, we determined using k-map technique the orientation of the Si sections in the nano-pillars as well as the GaN layers before and after coalescence to understand the coalescence process. In addition, it is crucial to get the strain distributions of GaN to gain insight on the quality of the GaN layers for optoelectronic applications such as microLEDs.



(a) (b)

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

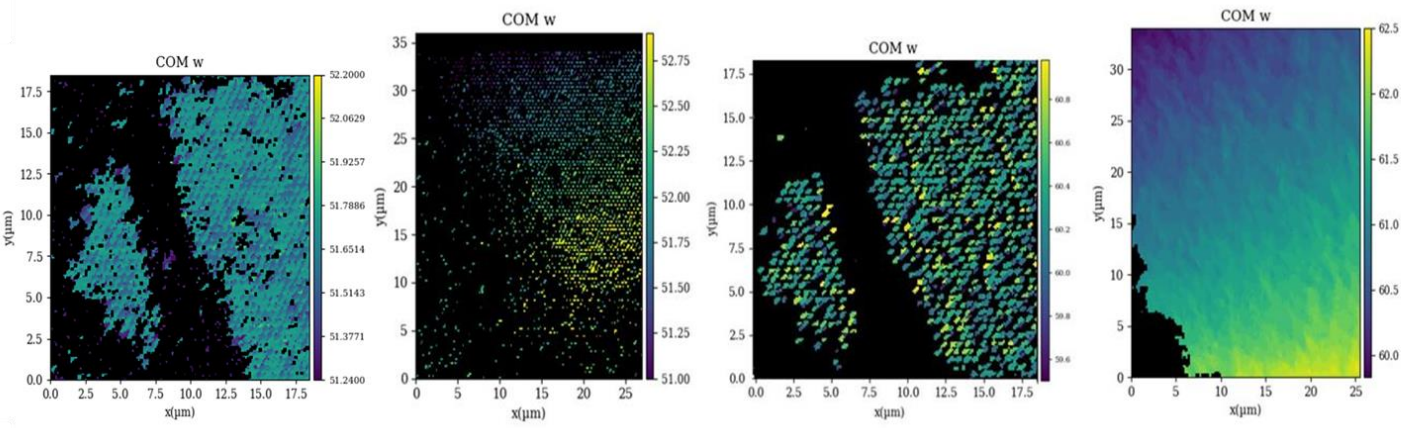
Experiments performed

To determine the orientation of the nano-pillars induced by coalescence, a sample area of 20 x 20 µm^2 was mapped and the asymmetrical Bragg reflection Si (331) in this top silicon (111) layer was measured for sample A which is a reference sample that has only nano-pillars, i.e. prior to the pyramids growth. Same measurements were repeated for sample B (figure 1b) where an area of 30 x 40 µm^2 was studied. For both samples, we performed rocking curves and took diffraction images every 150nm in x direction and 200nm in the y direction nm for different incidence angles (51° < w < 52.75°).

Then, to determine the GaN orientation as well as the strain distribution in the GaN layers, measurements were performed around the GaN (105) Bragg peak for varied incidence angles (59.4°<w<60.8°) for both samples.

Results and discussion

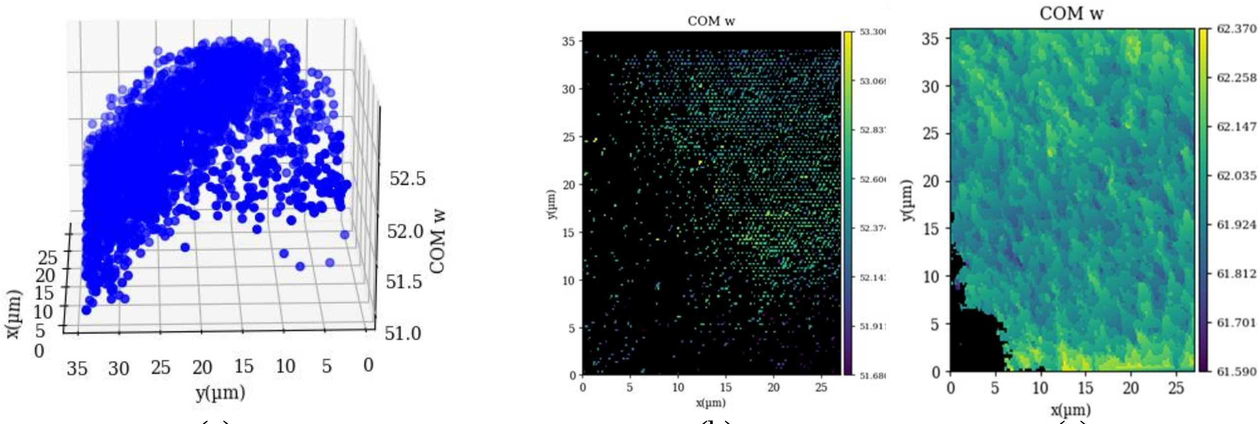
Figure 2 shows the individual silicon pillars and GaN layers before and after coalescence (after sample shift correction) measured on ID01. In the reference sample A, the Si layers are well oriented with maximum ω variation of Δω=0.96° (figure 2a) and the GaN layers are completely disoriented with Δω=1.5° (figure 2c) which is expected due to the heteroepitaxial growth of Nano-pillars GaN layers.



(a) (b) (c) (d)

Figure 2: Omega ω variation of silicon layers in nano-pillars before (a) and after (b) coalescence. Nano-pillars GaN layers before coalescence (c) and GaN layer after coalescence (d).

In sample B, we notice a gradient in ω along the diagonal over a length scale of ~ 44 μm for both Si and GaN layers (figure 2b and 2d). This gradient is probably related to a curvature (figure 3a) of the sample that occurs during the cooling phase of the growth process. To fully comprehend the behavior of the Si and GaN layers after coalescence, it is necessary to eliminate this curvature and its influence on the orientation of the layers. Therefore, Figure 3(b) was derived from a post-processing of the data presented in figure 2, it illustrates the variation of the Si and GaN layers omega (ω) after the removal of the curvature.



(a) (b) (c)

Figure 3: (a) 3D plot showing the curvature present in the Si nano-pillars of sample B, ω variation of (b) Si layers in nano-pillars and (c) GaN layer after coalescence and curvature removal.

In figure 3(b), we notice that the Si in the nano-pillars is more disoriented with a maximum Δω=1.62° compared to the reference sample (Δω=0.96°) , this means the coalescence of GaN pyramids causes dis-orientation of the silicon in the nano-pillars, implying that the growth occurs as intended.

Also, in the GaN layers (figure 3c), the variation of ω after coalescence and after curvature removal is relatively small (Δω=0.78°) compared to the GaN in the reference sample (Δω=1.48°) indicating that the obtained GaN layers are well oriented.

These results are beneficial as they prove that the self-realignment of GaN on Si(111), help in understanding the coalescence process and identifying the quality of the GaN layers. For a more detailed study of the quality of GaN layers, GaN measurements were also examined by creating a reciprocal map at each position of the sample and determining the components of the scattering vector Q, to extract the relative strain of 0.06% (figure 4) as function of the beam position for the GaN before coalescence. Further analysis of the rest of the datasets will allow us to obtain the tilt and strain map in the Si and GaN layers before and after coalescence.

These findings along with others [1] are promising for the fabrication of small highly oriented islands of GaN on silicon suitable for micro-LEDs. Therefore, the capacities of this powerful diffraction technique at ID01 opens the door for future investigation on newly improved high-quality samples with different pillars pattern.

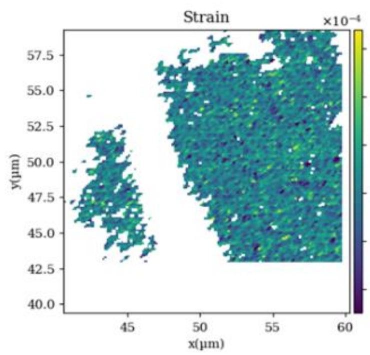


Figure 4: relative strain distribution along the GaN (105) as function of the beam position.

Reference:

[1]M. Wehbe *et al.*, "Study of GaN coalescence by Dark-field X-ray Microscopy at the nanoscale," *J. App. Crystallography*, submitted 2023