EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** Nanopendeoepitaxy: analyzing strain and mosaicity	**Experiment number**: MA-3588
Beamline: ID01	**Date of experiment**: from: 2017/10/20 to: 2017/10/25	**Date of report**: 01/03/2022
Shifts: 14	**Local contact(s)**: T. Schulli	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Introduction:

Gallium nitride (GaN) is a III-V direct band gap semiconductor and well suited for a range of applications such as optoelectronics and high-power electronics due to its special properties. However, GaN still presents low efficiency and this mainly due to the high dislocation density generated in the GaN epitaxial layer. Therefore, improving GaN heteroepitaxy methods is essential for producing more efficient GaN platelet. Figure 1 illustrates the four steps of an origin approach for GaN heteroepitaxy. It consists of growing GaN platelets on patterned Si-On-Silicon Oxide pillars (so-called SOI, for silicon on insulator). The viscoelastic properties of SiO_2 are expected to help in the process of growing low density GaN platelets.

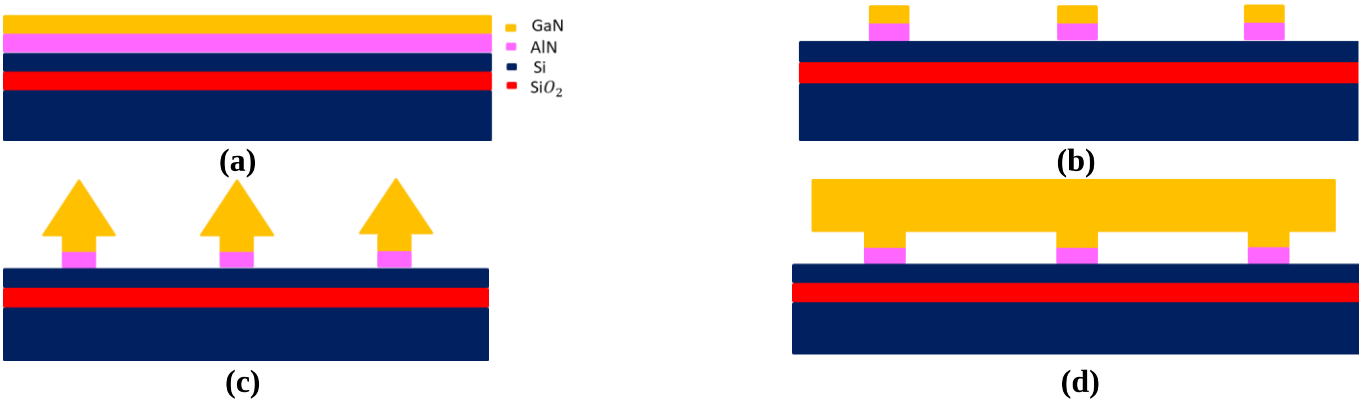


Figure 1: (a) SOI substrate with thin layers of AlN and GaN, (b) nano-patterned pillars, (c) grown GaN pyramids on top of the pillars and (d) fully coalesced GaN platelets.

To further understand and optimize this procedure, as well as to characterize the obtained GaN layers, different samples were analyzed under the beam line ID01.

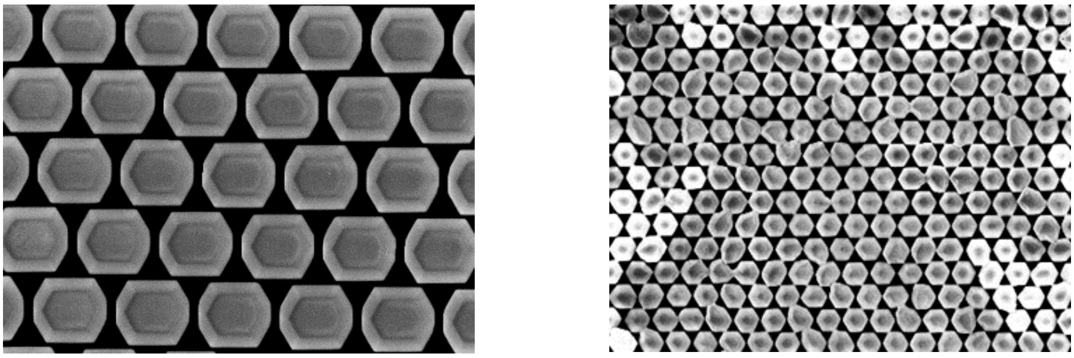
Experiment:

Due to the lack of time only two samples were characterized under the beam line ID01 with a scanning step $\Delta x = \Delta y = 100nm$. At 9 keV, the beam spot size was 100 nm (V) and 130 nm (H) (FWHM), however, the effective spatial resolution also depends on the incidence angle and the sample thickness. Each sample had varied pitch and distinct plot diameter summarized in table 1.

L	Pitch P	Plot Diameter D
L1	10 µm (isolated plots)	D_{max}
L2	0.5 µm	100 nm
L3	1 µm	100 nm
L4	2 µm	100 nm
L5	1 µm	200 nm
L6	2 µm	200 nm
L7	1 µm	500 nm
L8	2 µm	500 nm

Table 1: Varied pitch and diameter of the samples.

Different coalescence levels within the same sample can be observed due to the variation in the pitch and diameter of the pillars. Figure 2 show images of sample TS0438 with GaN pyramids at a level of no coalescence at all (L7) and others at the start of coalescence (L2).



(a) (b)

Figure 2: TS0438 with GaN pyramids (a) not coalesced (L7) and (b) at the beginning of coalescence (L2).

Figure 3 shows GaN pyramids that have completely coalesced in sample TS0440.

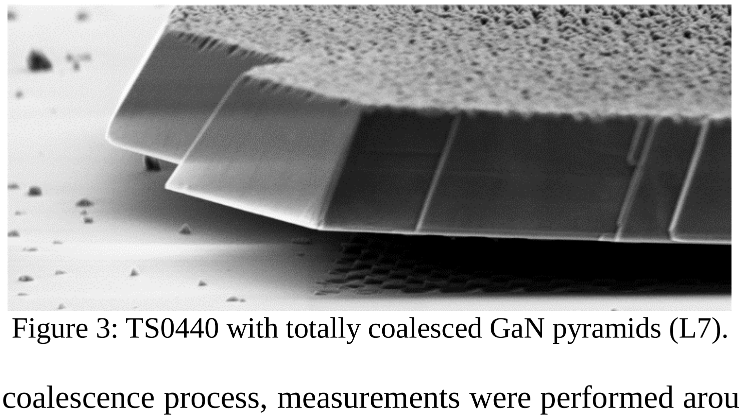


Figure 3: TS0440 with totally coalesced GaN pyramids (L7).

In order to understand the coalescence process, measurements were performed around the Si(111) Bragg peak for varied incidence angles w. Rays from Si substrate were also detected, along with rays from different GaN planes. Table 2 presents the different rays detected for the two samples.

Sample	matrix	material	ray
TS0438	L2	GaN	006
			204
		Si plot	111
		Si substrate	400
	L7	GaN	006
			204
		Si plot	111
TS0440	L5	GaN	006
			204
	L2	GaN	006
			204
	L7 center	GaN	006
	L7 corner	GaN	006
			204

Table 2: Different studied rays of the two samples.

In the following paragraph, the results of these experiments are analyzed and reported.

<u>Results and discussion:</u>

Using X-SOCS software, the data from the ID01 beam was examined to create a reciprocal map at each position of the sample in order to calculate the lattice parameter and estimate the in-plane and out-of-plane strain distributions. We present in this report the examination of the acquired data.

TS0438_L2_Plots Si (111)

A rocking curve was performed at each position of the sample and 80 scans were registered. The Bragg peak was detected for a Bragg angle w= 13.3°.

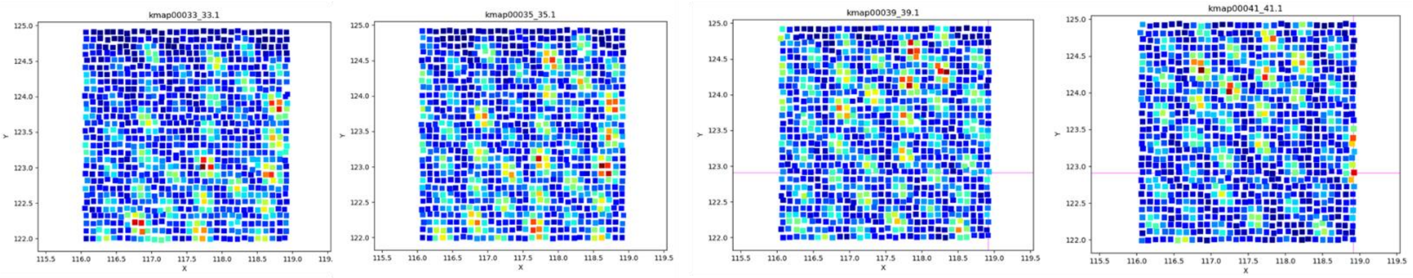


Figure 4: Diffraction spots from rocking curve scan on TS0438_L2_plots Si (111).

Diffraction spots from Si(111) plots are clearly detected and the hexagonal structure as well. However, we notice that the position of the sample is changing throughout one rocking curve; from scan number 33 to scan number 41, the sample moves progressively upward as illustrated in figure 4. This shift was also visible in the experiment notebook from the images taken during the experiment. Because the shift was not linear from one scan to the other, a correction of this shift using one scan as a reference was difficult.

From the data obtained in K-Map, X-SOCS software can perform numerical Gaussian fitting for the Bragg peak to determine its position in reciprocal space (Q_x, Q_y, Q_z) as seen in figure 5 below for TS0438_L2_Si (400).

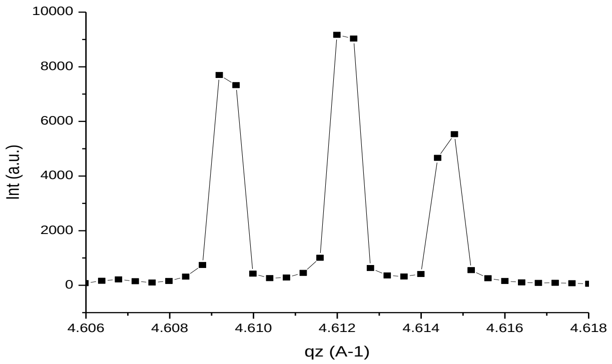


Figure 5: Diffraction peaks of Si (400) in the reciprocal space.

This unclear result of three diffraction peaks in the reciprocal space is probably due to the shift in the samples position.

Furthermore, the incident beam does not fall perpendicularly on the sample, which means the diffracted ray might originate from multiple plots causing more complexity in the results. Therefore, to improve the spatial resolution, we analyzed the data from the diffraction of GaN (204) of the sample TS0440 where the direct beam falls perpendicularly on the substrate.

TS0440_L5_GaN (204)

Results from the measurements of GaN (204) in TS0440_L5 are expected to be at a high resolution since a Bragg peak was detected at an incidence angle w=90° and the plots are well separated (P=1µm). Figure 6 shows the findings of three scans out of a total of one hundred.

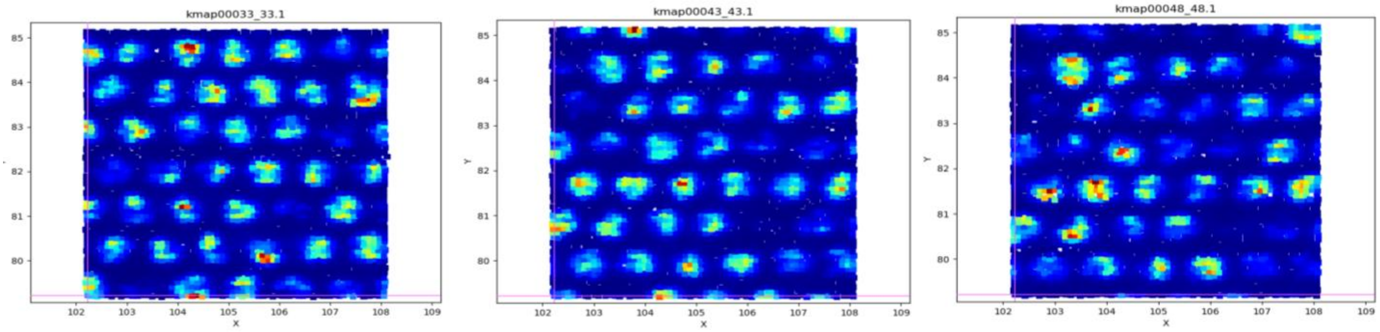


Figure 6: Diffraction spots from rocking curve scan on TS0440_L5_GaN (204).

Similar results were observed and the shift in the sample's position remains. This issue could be caused by a variety of factors, like a sample fixation problem or a motor problem.

<u>Conclusion:</u>

In order to describe the process of GaN heteroepitaxy on SOI pillars and characterize the obtained samples, rocking curve scans were performed under the beam line ID01 for two different samples. Diffraction spot were detected for Si (111), Si (400) and different GaN planes. The construction of the reciprocal space in order to obtain the tilt and strain distribution was not accurate because of a shift in the sample's position. Therefore, no further improvements in the analysis was possible. This issue will be considered in the future, and efforts to improve the sample quality will continue in order to be obtain GaN platelets with low dislocation density.