Standard Project

Experimental Report template

Proposal title: Study of the strain in GaN on Si(111) induced by ion implantation.		**Proposal number:** 32-02-790
Beamline: BM32	**Date(s) of experiment:** from: 20 July to: 25 July 2105	**Date of report:** 17/09/2016
Shifts: 15	**Local contact(s):** Jean-Sébastien Micha, Samuel Tardif, François Rieutord	*Date of submission:*

Objective & expected results (less than 10 lines):

The goal of the measurements was to observe the infuence of several parameters on the structural relaxation in a top GaN layer grown on Si(111) substrate induced by Mg implantation. GIXRD is a well-known and very reliable method to study the strain in the implanted materials. The important parameters to evaluate correspond to the role of the implantation dose in low and high conditions as well as the temperature, surface preparation, controlled atmosphere and duration of the annealing necessary to heal the damages and to activate electrically hole dopants. Combination of GIXRD and non-grazing incidence methods will allow us to discriminate the damaged thickness and to get the evolution of the stress tensor of these materials.

Results and the conclusions of the study (main part):

The crystal healing of Mg-implanted GaN layer has been studied by X-Ray diffraction. We measured several bragg reflections for samples having different protective cappings with different fluencies and annealing steps (see table below in French).

PDM ESRF

Capping	Dose implantée	Recuit post implant: 1h sous N2 1100°C	Echantillons disponibles	Mesure XRD basique	Evolution en température	Evolution en temps de recuit
Fin: AlN+SiN	1.00E+13	oui	3	1		
	1.00E+13	non	3	1	1	1
	1.00E+14	oui	3	1		
	1.00E+14	non	3	1	1	1
	1.00E+15	oui	3	1		
	1.00E+15	non	3	1	1	1
Fin: AlN+SiN	aucune	non	3	1		
AlGaN+SiN+Si3N4 désposé	3.00E+14	oui	3	1		
	1E+15	oui	2	1		
	3.00E+15	oui	3	1		
	aucune	oui	3	1		

The GaN layer is obtained by MOVPE growth on Si(111) wafer with intermediate AlGaN layers as shown in Figure 1. For post implantation samples oscillation fringes appear on the low-angle diffraction side of the main GaN peak. This lattice distorsion has been previously studied in Si implanted materials [i] and similar analysis and interpretations apply. Oscillation fringes result from the lattice variation parallel to the free surface (z-axis) induced by the implantation defects and can be analysed quantitatively from the profiles shapes that are related to defect density and strain, increasing with the implanted doses. They have been measured as the function of the dose for (002) to (008)

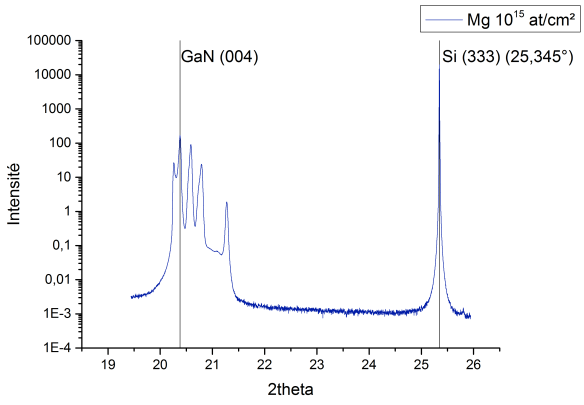

Fig. 1: Mesurement of the (004) reflection of implanted

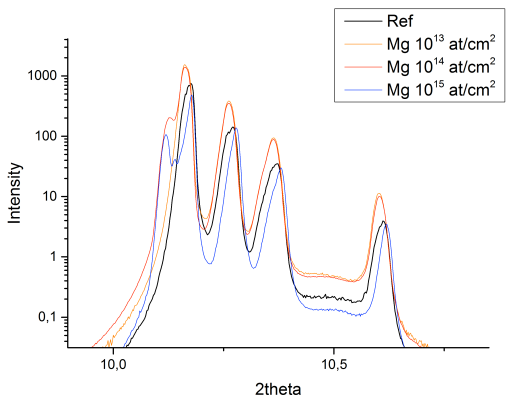

Fig. 2: Influence on the implantation dose measured at

sample stacking. *the (002) reflection.*

Bragg peaks in order to have several experimental data to analyse in order to improve the accuracy (see Fig. 2 and 3).

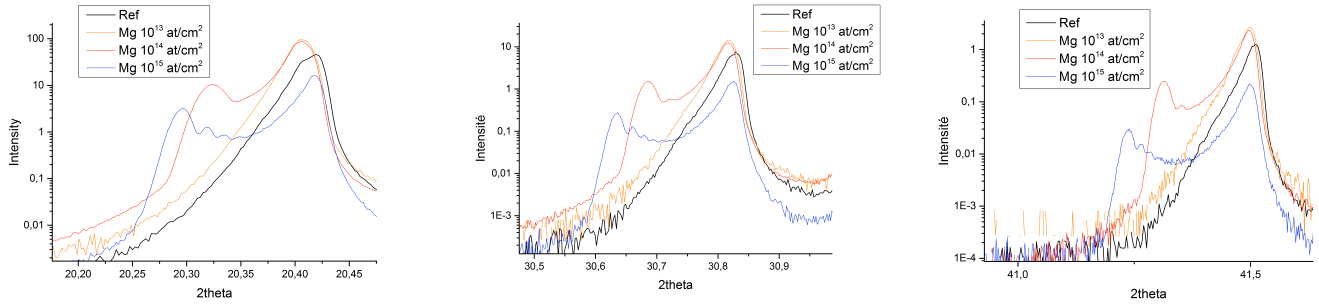

Fig. 3: *Influence on the implantation dose measured at the (004),(006) and (008) reflections.*

Oscillation fringes collapse with the GaN peak with an annealing of 1 h at 1100 °C under N_2 as shown in Fig. 4. It confirms the crystal lattice recovery on the z-axis for this thermal budget. We observe moreover a change of the relaxation in the satcking from the signature of the background signal (see red circles in Fig. 4).

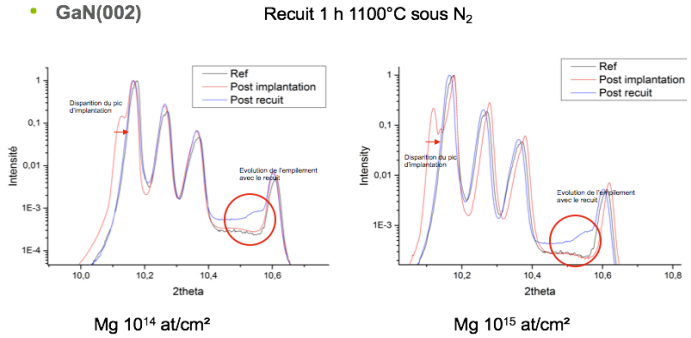

Fig. 4: Comparison of reference, post implantation and 1h annealing at 1100 °C under N2 Bragg peak measurement around the (002) GaN reflection.

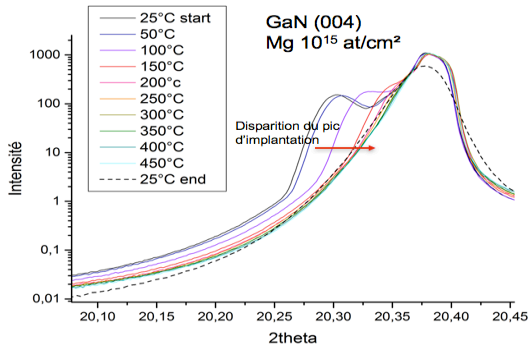

Fig. 5: Evolution of the (004) Bragg peak under annealing between 25 and 450 °C (thermocouple values).

A example of the kinetic evolution of the (004) Bragg peak under annealing between 25 and 450 °C (thermocouple values) is shown in Fig. 5. The real temperature of the sample will be adjusted from the shift of the Silicon Bragg peak also carefully measured and from the knowledge of expansion coefficient. We used the special furnace developed in the QMAX ANR (coordinator Limoges University), which work well before an intensity burst that killed the filament. The switching on of the furnace should be limited in current and corrective procedures are under way.

Justification and comments about the use of beam time:

- The GIXRD signals show a strong mixing of contributions and the broadening of the peaks will be studied. Most of the measurements have been focused on symmetric reflections.
- During this experiment, we mainly studied ex-situ prepared samples and we have successfully determined the influence of most parameters in a very qualitative way. Only one in-situ annealing study has been performed and we discovered that crystal healing occurs at much lower temperature than electrical activation. This point will be specifically investigated in the future.
- These measurements provide very good data for quantitative analysis of the deformation profiles. X-ray diffraction profiles are under analysis using the RaDMax program (M. Souilah, A. Boulle, A. Debelle, "RaDMaX: a graphical program for the determination of strain and damage profiles in irradiated crystals", *J. Appl.Cryst.* **49**, 311-316 (2016)) to get deformation and Debye Waller coefficients as a function of depth. As said before, the measurement of different orders will provide a better accuracy of the quantitative results.
- These experiments are original in the nitride community and give important outputs for the development of implantation process in these materials.

Publication(s):

- One publication is presently under revision in Physica Status Solidi to publish the description of sample preparation.
- A more complete publication dedicated to X-ray results will be submitted when all the results will be analysed.

[i] N. Sousbie, L. Capello, J. Eymery, F. Rieutord, C. Lagahe, Journal of Applied Physics **99**(10) 103509 (2006).