

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://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

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



	Experiment title: Proposal title: Characterization of AlP and GaP nanolayers on silicon for photonic and photovoltaic applications	Experiment number: MA-2067
Beamline: MB02	Date of experiment: from: February 6, 2014 to: February 10, 2014	Date of report: August 31, 2015
Shifts: 12	Local contact(s): Nathalie Boudet, Nils Blanc	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): UMR FOTON, CNRS, INSA Rennes, Rennes, F35708 France A. Létoublon*, Y. Ping Wang*, O. Durand*, C. Cornet* Univ. Grenoble Alpes, Institut Néel, F-38000 Grenoble CNRS, Institut Néel, F-38000 Grenoble, France T. Nguyen Thanh *		

Report:

In order to extend light emission III-V semiconductors on Si for very large scale and low cost integration, the growth of GaP and related ternary and quaternary (GaAsPN) materials lattice matched to Si are studied. The best optical properties can be obtained with GaP as starting layer on Si. But crystal defects such as antiphase boundaries (APB) and microtwins (MT) are observed on GaP/Si interface, which are detrimental for optical properties and must be eliminated or at least confined near the Si interface. The characterization and optimization of interface structural properties are of primary importance and is the main motivation of the present project [1,2].

A first experiment on D2AM has been carried out november 2011, showing the advantage of using a large 2D low noise pixel detector for detection of both MT and APD [1,2]. The results showed the elimination of MTs at high growth temperature [3]. During the second experiment on november 2012, GaP/Si samples with differences on prelayer, miscut, and buffer of silicon have been studied. An original integration method has been developed to quantify deeply the MT relative density and CLs, and demonstrated the anisotropy MT ratio for growth on miscut surfaces and benefit of Al prelayer [4].

In this new experiment, 5 samples of GaP layer grown on Si (001) substrate miscutted 6° towards the [1 1 0] direction have been studied. Group 1 with S1: 45 nm GaP grown by MEE at 350°C and S2: 10 nm GaP grown by MEE at 350°C followed by 35 nm GaP grown by MBE at 500°C , both with a 0.9 Monolayer (ML) Ga prelayer. Group 2 contains 3 samples of 5 nm GaP grown by MEE at 350°C with a Ga prelayer; the difference is on the amount of Ga prelayer: S3 with 0.85 ML Ga, S4 with 0.93 ML and S5 with 1.1 ML. Snapshot images centered on (002), (004), (006), (008) and (0 0 10) GaP reflections for 2 azimuths (azim.1 X-rays parallèle to [1 -1 0] direction of the substrate; azim.2 X-rays parallèle to the [1 1 0] direction) have been performed on the first two samples. Images on GaP (002) and (006) have been acquired and a research of MT around the (002) reflection has been carried out on samples of Group 2.

For Group 1, transverse scan profiles extracted from the (002), (006) and (0 0 10) GaP reflections images display two components: the thin peak originated from the long-range lateral order of crystal structure of the

layer and the broad peak related to the short-range defects. The quality factor (QF) defined as the area ratio of the thin peak to the broad peak. Higher QF indicates better crystalline quality with lower density of defects. The Williamson-Hall Like analyses [5,6] have been carried out to evaluate the correlation length (CL) related to mean APB distances. The results reveal that S2 represents higher crystalline quality than S1. The CL of S2 for two azimuths are respectively 31.3 ± 2.1 nm and 22.6 ± 1.5 nm while that of S1 are 18.6 ± 0.1 nm 14.8 ± 0.1 nm. The QF measured from the (002) reflection is 16.6 for S2 and 3.4 for S1. This indicates a limitation of defects generation by applying the two-step growth sequence. For Group 2, due to the lack of intensity on the (006) reflection, only the (002) transverse scans are studied. Table 1 lists the integral breadth (IB) of broad peak, CL and QF for each azimuth of the three samples. CL is calculated according to the Scherrer's Law: $CL=1/IB$.

Table 2. Integral breadth (IB), correlation length (CL) and quality factor (QF) for two azimuth of Group 2

	Coverage Ga (ML)	Azim. 1			Azim. 2		
		IB (nm ⁻¹)	CL (nm)	QF	IB(nm ⁻¹)	CL(nm)	QF
S3	0.85	0.094	10.70 ± 0.08	49.3 ± 1.3	0.155	6.47 ± 0.05	6.8 ± 0.3
S4	0.93	0.148	6.67 ± 0.08	1.1	0.221	4.53 ± 0.07	0.6
S5	1.1	0.116	8.64 ± 0.04	0.4	0.128	7.82 ± 0.04	0.2

The table evidences the influence of the coverage of Ga on the CL and QF of GaP/Si layer. S3 represents larger CL and QF than the other samples. However, the r.m.s. roughness measured from AFM shows a most smooth surface for S4, as shown on Fig.1 a). Moreover, a strong anisotropy has been observed on S3, as shown on Fig.1 b): for azimuth 1, the spot of greater intensity with diffus vertically elongated, and the QF is measured to be 49.3 ± 1.3 ; for azimuth 2, the spot represents a round shape, with a QF much smaller (6.8 ± 0.3). These results opened our mind to study more deeply the coverage of Ga on an extended series. As the absolute quantification measurements of MT have already been developed and aided the quasi-elimination of MT during the growth procedure[7], the future work will be centered on the quantification of volume fractions of the APDs in the quasi-perfect layers without MT and with very low density of APD.

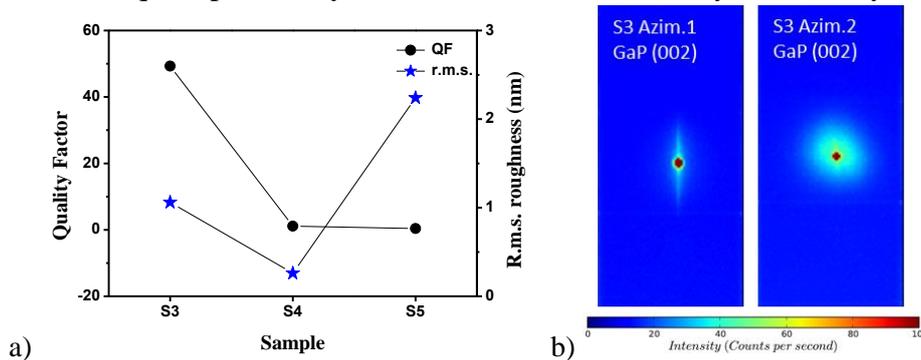


Fig.1 a) Quality factor and r.m.s. roughness for samples of Group 2;
b) 2D image centered on GaP (002) of S3 on two azimuths

References:

- [1] T. Nguyen Thanh, C. Robert, E. Giudicelli, A. Létoublon, et al. *J. Crystal Growth* **378** (2013) 25-28
- [2] T. Nguyen Thanh, C. Robert, A. Létoublon, C. Cornet, et al., *Thin Solid Films* **541** (2013) 36
- [3] T. Nguyen Thanh, J. Stodolna, Y. Ping Wang, S. Almosni, et al, submitted to *Thin Solid Films*
- [4] T. Quinci, J. Kuyyalil, T. Nguyen Thanh, Y. Ping Wang, et al. *J. Crystal Growth* **280** (2013) 157-162
- [5] O. Durand, A. Létoublon, D.J. Rogers, F.H. Teherani, *Thin Solid Films* **519** (2011) 6369
- [6] A. Létoublon, W. Guo, C. Cornet, A. Boule, et al. *J. Crystal Growth* **323** (2011) 409
- [7] Y. Ping Wang, A. Létoublon, T. Nguyen Thanh, M. Bahri, et al. *J. Appl. Cryst.* **45** (2015) 702-709

Justification and comments about the use of beam time (5 lines max.):

12 shifts combined with the use of an efficient 2D detector allowed us to study 5 samples grown under various conditions. The use of synchrotron is here very important for detection of thin film (5 nm) and very low density nanodeflects.

Publication(s):

- 1 paper submitted to *Thin Solid Films* [3], 3 paper published from the previous experiments [1,2,4], and several conference contributions