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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

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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.

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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.

ESRF	**Experiment title:** Caracterisation of GaP/Si Cornerstone for Intergrated Photonics on Silicon	**Experiment number**: 02-02-792
Beamline:	**Date of experiment**: from: 9/11/11 to: 14/11/11	**Date of report**: 14/02/12
Shifts:	**Local contact(s)**: Nathalie Boudet	*Received at ESRF:*
Names and affiliations of applicants (* indicates experimentalists): A. Létoublon*, Than T. Nguyen*, C. Cornet*, O. Durand*		

Report:

Background:

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, the structural properties of this interface are of primary importance and this is the main motivation of the present project.

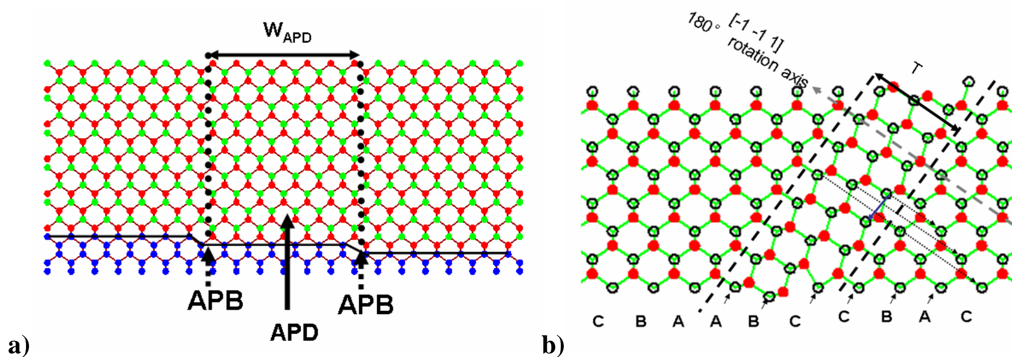


Fig. 1 Typical APD defect in GaP on a stepped Si surface with (1 1 0) oriented APB. b: typical microtwin (MT) in Zinc-Blend structure.

Several types of defects are observed on III-V growth on IV monolithic substrates: point defects, misfit dislocations, phase boundaries (APB) (fig. 1a), microtwin (MT) (fig. 1b) and stacking faults (SF). These defects are detrimental for optical properties and must be eliminated or at least

confined near the Si interface. Amongst them, APB and thus anti-phase domains (APD) are difficult to avoid since they are due to the intrinsic nature of the interface with two different crystal symmetries and polarities. In this type of material, APD have been characterized using mainly transmission electron microscopy (TEM) [1]. As shown for instance by Neuman et al. [2], X-ray diffraction is also a very efficient tools giving complementary information to TEM. Moreover, it is non destructive, it doesn't require sample preparation and is more statistically sensitive to defect density.

20nm GaP layers samples have been grown in our laboratory on 4° miscut samples, at the laboratory using a solid source MBE. The 4° miscut should favor the formation of Si bisteps in order to limit the formation of APD [1,3]. Growth conditions have been found for a low rate of plastic relaxation. Beyond this, different growth conditions are employed (growth temperature, post growth annealing, continuous MBE or MEE i.e. alternative deposition of Ga/P) in order to minimise the defect density and the roughness of the GaP surface. The present study focuses on MEE growth at for different growth temperatures.

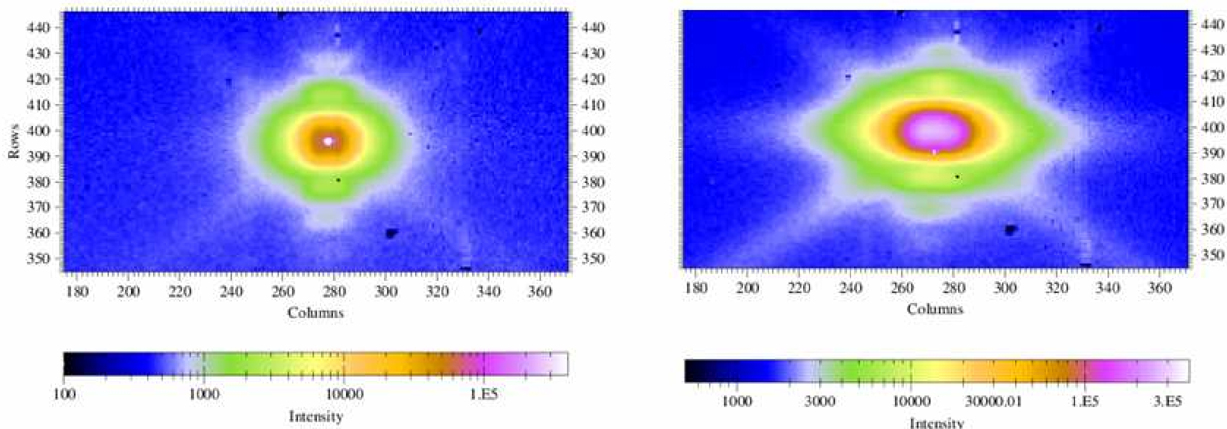
High resolution XRD on a laboratory setup has been employed for the GaP layer characterisation. High overall crystal perfection has been obtained since vertical X-Ray Diffraction scans through GaP(004) exhibit Laue fringes and are characteristic of a coherent epitaxial growth for the different samples. This has been also checked on the (224) reflection for which GaP peak is vertically aligned to the Si peak position. Howether transverse scans performed through (002), (004) and (006) exhibit a specific broadening of the weak reflections (002) and (006). This enhanced broadening is the signature of APD [2,4,5].

New Experimental results:

This experiment has been carried out at an energy of 16keV allowing to reach (008) reflection (unreachable on our high resolution lab setup). We chose to use the large 2D detector XPAD 3 for faster data acquisition.

During this experiment, 6 samples grown under different conditions, have been deeply studied.

For all samples diffuse scattering around 4 (00*l*) reflections could be measured, using a large 2D X-ray counter. This allows a rapid evaluation of cristalline perfections through from cutlines extracted in snapshot images of the 2D detector such as shown fig. 2. Fig. 2 shows 2D images recorded on (002) GaP reflection for a sample grown at Tg=350℃. Images are taken for two different azimuth (ϕ=0 and 90°). For ϕ=90°, the column direction in the detector is parallel to the [110] Si direction. This anisotropy probably corresponds to the signature of a larger number of (110) than than (1 -10) APD. ((110) APD are bounded by boundaries lying along (110) crystal planes). For fig. 2a) the sharp centerd peak is the maximum of the GaP CTR (crystal truncation rod) which is almost but not fully tangent to the ewald sphere. Thickness fringes are also observed along the Rows direction, attesting of the high crystal perfection. For fig. 2b a slight misalignment of the crystal explain the off centered CTR maximum.



a) b)

Fig. 2 2D image using XPAD3 D2AM detector for (002) GaP reflection with for a): column axis of the camera parallel to [1 -1 0] of the Si crystal, and b: column parallel to [110]. The sample was fixed during 300seconds of exposure.

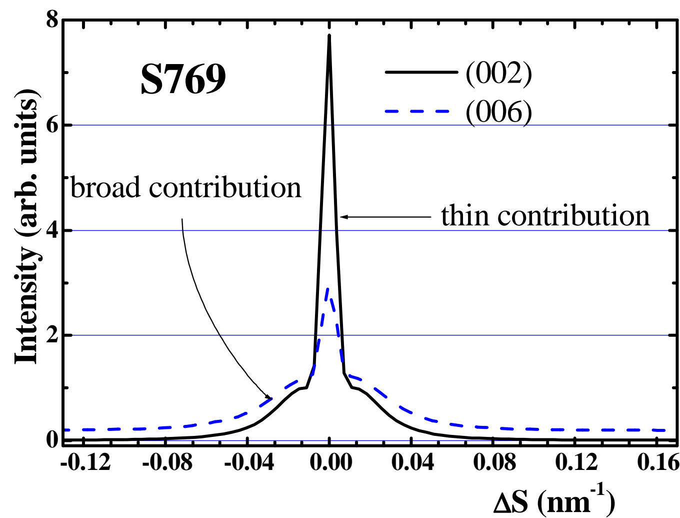
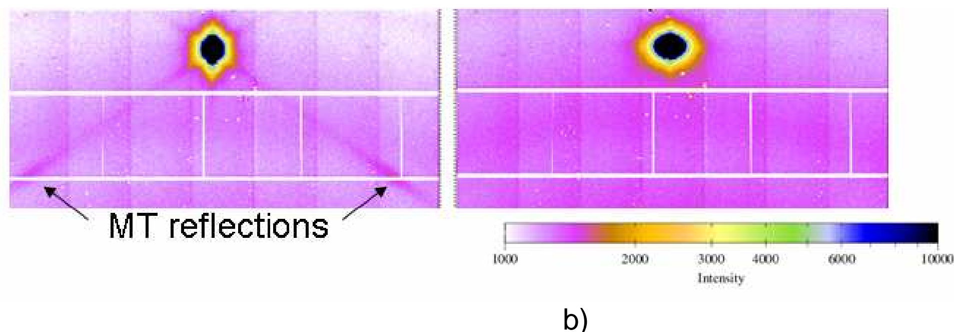


Fig. 3 Cutlines extracted from 2D detctor images for (002) and (006) weak reflections showing important broad contributions with very similar line profiles.



a) b)

Fig. 4 MT reflections are evidenced on a) for sample grown at 450℃ and are not present on b)for sampl e grown under the same conditions but at 580℃.

As shown fig. 4 MT reflections could be easely and rapidly identified (300sec exposure). It has been observed that MT reflection itensity decreases with growth temperature and could not be detected for Tg=580℃.

Perspectives and further experiments:

The full treatment of this first experiment will also allow 3D evaluation of the diffuse scattering around weak reflections and thus the extraction of scattering structure factors of the defects mainly for APD and MT when they contribute more than other defects. The substratction of the Si diffuse scattering will be also performed mainly around strong reflection where it partially burries the defect contribution.

The use of a new XPAD detector shouldl also allow a better background suppression (probably mainly due to Ga fluorescence).

The XRD characterisation of GaP defects should allow a better comprehension of the growth process and the mechanism of defect anihilation. This should help to improve the growth procedure of high structural perfection GaP layers that would serve as pseudo subtrates for subsequent growth of III-V active area with high optical performances.

References:

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[4] A. Létoublon et al. J. of Cryst. Growth. Journal of crystal growth **323** (2011) 409.

[5] W. Guo A. Bondi, C. Cornet, A. Létoublon et al. Appl. Surf. Science **258** (2012) 2808.

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