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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

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.

ESRF	**Experiment title:** Local strain measurements in highly strained suspended Ge microstructures using X-ray Laue microdiffraction	**Experiment number**: MA-2651
Beamline: BM32	**Date of experiment**: from: 2015/11/20 to: 2015/11/23	**Date of report**: 2016/02/25
Shifts: 9	**Local contact(s)**: Jean-Sébastien MICHA	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Samuel Tardif (CEA-Grenoble)
*Alban Gassenq (CEA-Grenoble)
*Kevin Guilloy (CEA-Grenoble)
*Esteban Marin (PSI)
*Thomas Zabel (PSI)

NOTE : This experiment was a continuation of experiment MA-2490

Report

Background:

Straining a germanium crystal is a way to control its electronic properties and under a large enough tensile strain, the indirect electronic bandgap may become direct, a prerequisite to make an efficient light emitter. In this experiment, we used pre-strained suspended Ge layers in which micro-structures (micro-bridges, micro-crosses) were patterned to locally concentrate the strain (Fig. 1). GeOI wafers made by SmartCut^{TM} technologies from CEA-LETI have allowed us to reach unprecedented strain values: the Raman shift values measured in our devices are higher than the current, published state of the art.

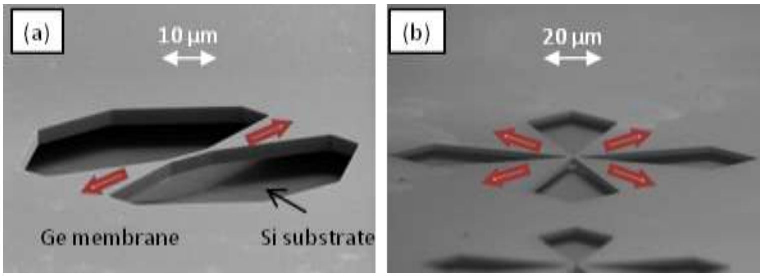

Figure 1. (a) Micro-bridge and (b) micro-cross sample patterned in a prestrained Ge layer. Changing the dimension of the arms, bridges or crosses allows one to tune the strain in the device.

Objectives:

The aim of this experiment was to measure the local strain in the microstructures. We wanted to compare direct strain measurements with other indirect techniques, such as micro-Raman spectroscopy (µRaman) and finite elements modeling (FEM) to validate our models and simulations. Since the typical size of the devices is on the order of a few microns, Laue microdiffraction is a well suited technique.

Experiments:

After our successful initial experiment (MA-2490), we performed the measurements in a new series of samples for which the processing steps had been further optimized [1,2]. In particular we investigated a new design of the micro-bridges as well as micro-bridges that were no longer suspended but bonded to the Si substrate [3]. Similarly to the previous measurements, we divided the available beamtime between real-space maps in selected devices (4 to 8 hours per map) and point measurements in the center of the devices (10 – 30 min per device). Preliminary alignment using the SPEC-interfaced optical microscope and fuorescence detector available at the beamline allowed a large number of samples to be measured. Beam quality (intensity, stability of the position on the sample) was very satisfactory. Since the Ge membrane were quite thin (less than 1 µm), we expected some difficulty in separating the diffraction patterns from the Ge membrane and from the Si substrate but since we used devices made from Germanium-On-Insulator, the slight misorientation between the two crystals introduced during the process was enough to clearly separate both contributions. As described in the proposal, we had prepared GeOI susbtrates for which the misorientation angle was larger, in order to be able to measure easily the small strains. However the wafers were destroyed in the fire that happened in the clean room of the LETI in April 2015. From our previous experience we also decided to use only the MarCCD detector at the standard position and not the ImageStar detector on a "side" position. We used the free space on the side of the setup to install a fluorescence detector mounted on translations. The fluorescence detector was used to measure the energy of at least one diffraction peak, so that we could access the full strain tensor.

Results:

Typical diffraction patterns were shown in the previous report (MA-2490). We could perform strain and tilt mapping in both micro-bridges and micro-crosses, thus enabling the calibration of the Raman-strain relation at higher strains [4]. The comparison with FEM calculations in typical devices are shown in Fig. 1 and an excellent agreement is found [5].

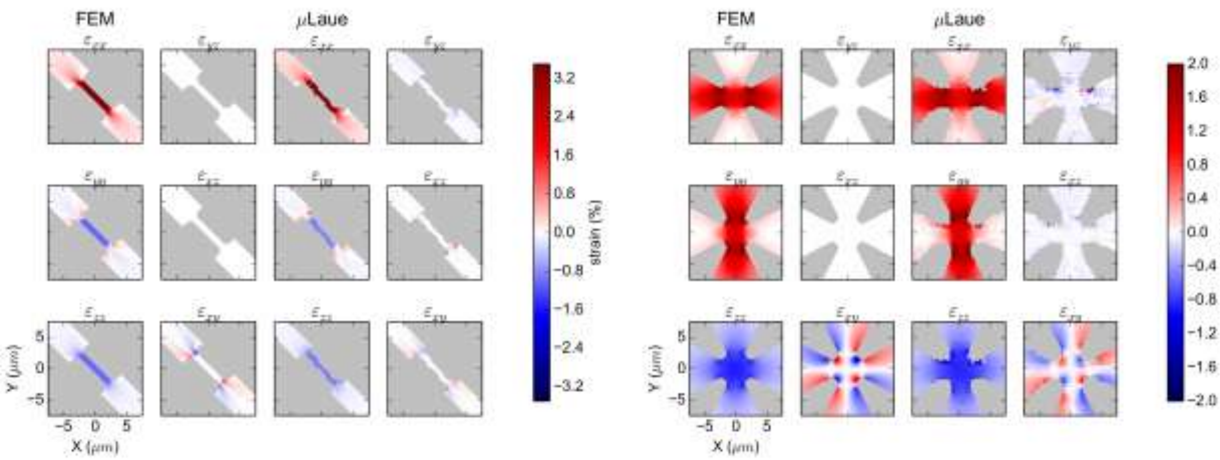

Figure 1. Maps of the strain tensor components, as calculated using FEM and measured using Laue micro-diffraction

We also used a new EIGER 1M detector, available at the endstation INS2 on the beamline. This detector was mounted in place of the usual MarCCD165 detector. The EIGER 1M detector covers only 25% of the area of the MarCCD detector but the pixelsize is comparable (80 µm) and since it is a pixel detector, there is no noise and the readout rate is much faster (10 Hz vs 0.2 Hz for the MarCCD). As result we could perform much faster Laue mapping. A typical Laue pattern from both the MarCCD and the EIGER are shown in Figure 2. The analysis of the EIGER measurements is underway.

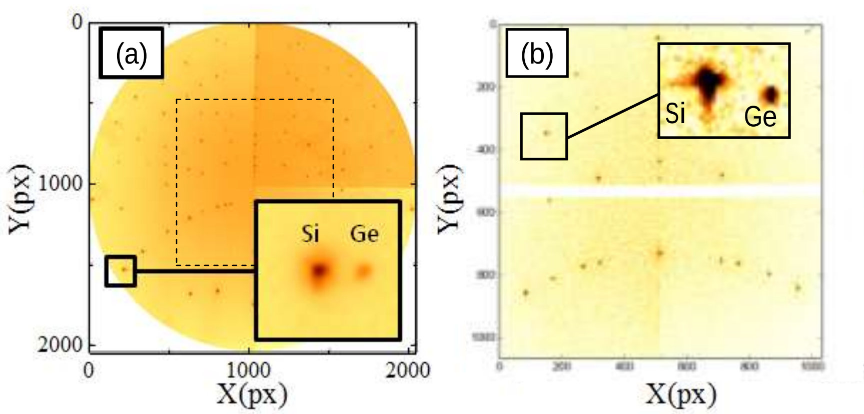

Figure 2. Typical Laue diffraction patterns from a micro-bridge showing both the Si substrate and the Ge bridge measured using (a) the usual MarCCD165 and (b) the new EIGER 1M detector. The dashed line in (a) indicates roughly the coverage of the smaller detector shown in (b)

Additionally, we took advantage of the new xiatek fluorescence silicon drift detector (SDD) on the experimental setup on the beamline to record simultaneously the energy spectrum. The detector was mounted on translations on the side of the sample to allow 2D scanning. This way we could position the detector on a diffraction spot and record its energy at the same time as we measured the Laue pattern on the 2D detector. Typical results are shown in Fig. 3. First we simulated the diffraction pattern on the side of the sample (Fig. 3a) and then we scanned the SDD in that area to find and identify the position of the diffraction spot (Fig.3b). Then we could measure the energy of the reflection (Fig. 3c). Note that the fluorescence of the Ge, as well as the reflexions from the Si crystal can be used as internal calibration of the energy. The analysis of the data is underway.

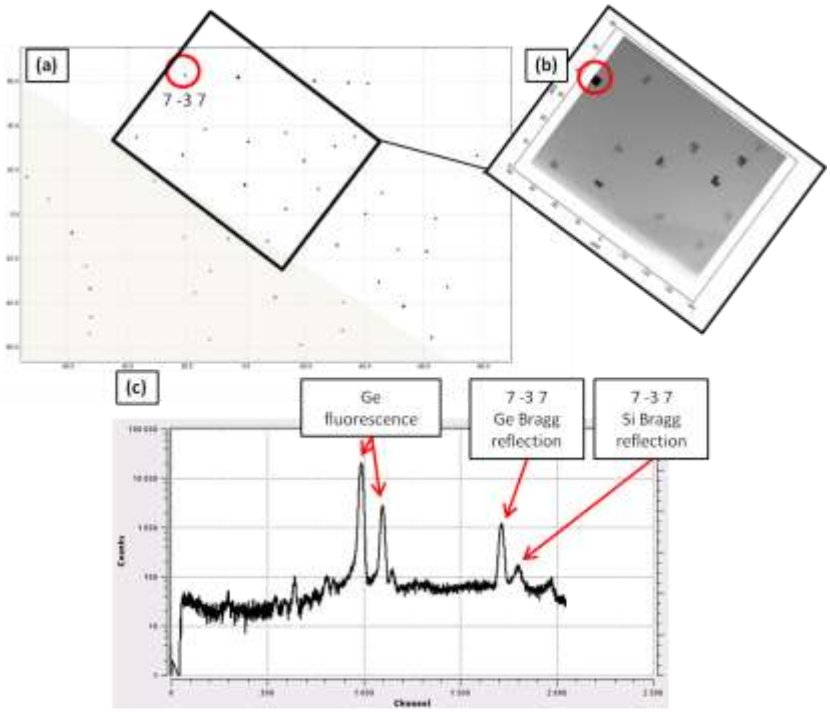

Figure 3. (a) Simulated Laue pattern on the side of the sample. The gray area is below the sample horizon. (b) Corresponding scan of the SDD detector (spectrum integral) at the same location. (c) Energy spectrum measured at the position of the 7 -3 7 Bragg reflection, indicating both the Ge and Si Bragg peaks (the detection solid angle of the SDD is large enough to measure both peaks at the same detector position).

Conclusions and Outlook:

We obtained very statisfactory results from our beamtime: we could complete our dataset from our previous experiment (MA-2490) and measure the deviatoric strain tensor in a wide range of samples. We could measure strain maps with micrometer resolution, which are in excellent agreement with the FEM simulations. We were also able to perform measurements of the energy of the Bragg reflection using an energy-resolved detector. The use of a new pixel detector (EIGER 1M) was also possible and sped up considerably the measurements. The analysis of the data and the comparison with the MarCCD measurements will provide useful information on how such fast pixel detectors can change the paradigm of Laue micro-diffraction at BM32. Further works also include finishing processing all the raw data and comparing the results with other strain measurement techniques (*e.g.* Raman spectroscopy, electronic microscopy direct imaging).

Our results were or will be presented in the communications indicated hereafter. Three additional articles are currently in preparation and will be submitted shortly.:

Publications:

- [1] V. Reboud et al., *Proceedings of SPIE* **9367**, 936714 (2015)
- [2] A. Gassenq *et al., Applied Physics Letters* **107**, 19 (2015)
- [3] V. Reboud *et al., Proc. SPIE 9752, (2016)*
- [4] A. Gassenq *et al., submitted to nanoletter (2015)*
- [5] S. Tardif, *et al., to be submitted (2016)*

International conferences:

- A. Gassenq *et al.*, "*Strain characterization by Synchrotron based Laue micro-diffraction and Raman spectroscopy in highly strained Ge micro-bridges fabricated in GeOI wafer for photonic applications*", **SPIE Photonics West 2016**, San Francisco, USA (Feb. 13th -18th, 2016)

- K. Guilloy *et al.*, "*Strain Dependence of the Direct Bandgap in Highly Strained Germanium Micro-Membranes*", **MRS Spring meeting 2016,** Phoenix, USA (March 28th – April 1st, 2016)

- A. Gassenq *et al.*, "*Biaxial and Uniaxial Strained Cavities in Suspended Ge Layers Using Strain Redistribution in 200 mm GeOI Wafers for Laser Applications*", **MRS Spring meeting 2016,** Phoenix, USA (March 28th – April 1st, 2016)

- S. Tardif, *et al.* "*Mapping the Full Strain Tensor and Lattice Tilts in Ge Microstructures for Photonics Applications*", **MRS Spring meeting 2016,** Phoenix, USA (March 28th – April 1st, 2016)

- A. Gassenq, *et al.* "*Raman frequency shifts versus strain measurements in highly strained Ge along <100>, <110> and <001> directions for laser applications*", **SPIE Photonics Europe 2016**, Brussels, Belgium [9891-22] (April 4th – 7th, 2016)

- K. Guilloy, *et al.* "*Nonlinear strain dependences in highly strained germanium micromembranes for on-chip light source applications*", **SPIE Photonics Europe 2016**, Brussels, Belgium [9891-33] (April 4th – 7th, 2016)

- K. Guilloy *et al.*, "*Nonlinear direct band gap and Raman shift strain dependence in germanium under high uniaxial stress*", **E-MRS Spring meeting 2016,** Lille, France (May 2nd – 6th, 2016)