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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

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-2490
Beamline: BM32	**Date of experiment**: (1) from: 2015/04/19 to: 2015/04/20 (2) from: 2015/05/07 to: 2015/05/09	**Date of report**: 2015/08/25
Shifts: 9	**Local contact(s)**: Jean-Sébastien MICHA	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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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 waffers 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.

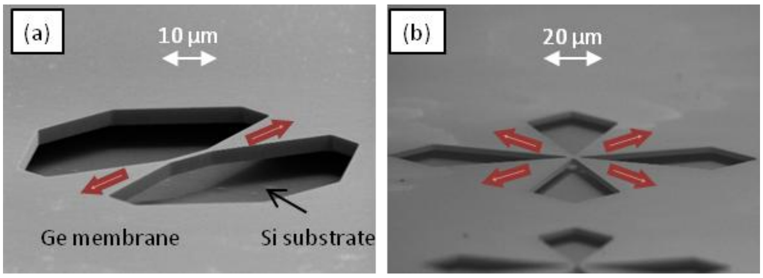

Figure 1. (a) Micro-bridge and (b) micro-cross sample patterned in a prestrained Ge layer. Changing the dimension of the arms, bridges or crosses allows one to tune the strain in the device.

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:

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. We also feared that the signal from the thin Ge membrane would be too weak compared to that of the Si substrate. However, we found it to be sufficiently strong in most cases for 1 to 10 s exposure. We tried using a ImageStar CCD with an antiblooming feature to allow for longer exposure but the reduction of the field-of-view compared to the usual MAR165 CCD was not worth the change. In the worst cases (thinnest membranes), we found that summing a few unsaturated images on the MAR165CCD was enough to raise the signal-to-noise ratio to acceptable levels. We also took advantage of the rainbow-filter that was already setup on the beamline to perform some measurements of the energy of the Bragg reflections.

Results:

Typical diffraction patterns are shown in Figure 2. The Si diffraction pattern was measured in a Ge-free region and subtracted from the measurements in the Ge membranes (Figure 2(a)). All peaks could then be fitted and indexed using the LaueTools software (Figure 2(b))..

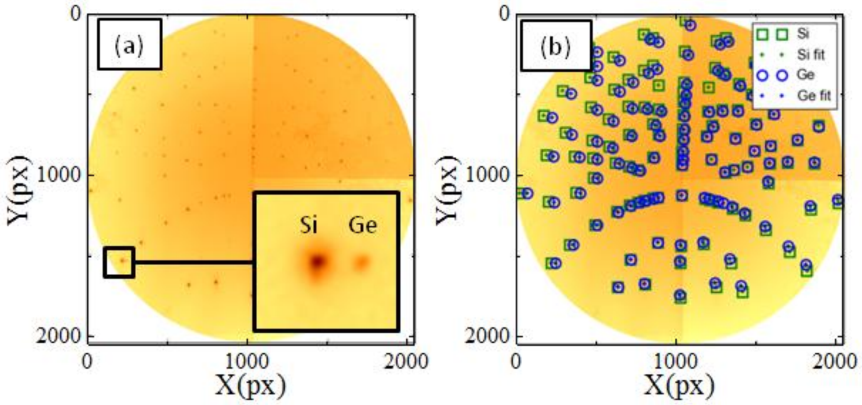

Figure 2. (a) Typical Laue diffraction pattern from a micro-bridge. (b) All peaks of the Ge membrane and of the Si substrate were indentified (empty symbols) and fitted for indexation (crosses) using LaueTools.

Such measurements were performed for each micro-device, either only in the center of the pattern or in a raster scan across it. An example of a series of point measurements in different devices is shown in Fig. 3. As the strain increases (as evidenced by an increasing Raman spectral shift indicated in the scale), the peak positions in the Laue patterns are shifted monotonously. The Laue patterns were then fitted and the deviatoric strain tensor corresponding to each pattern was calculated using LaueTools.

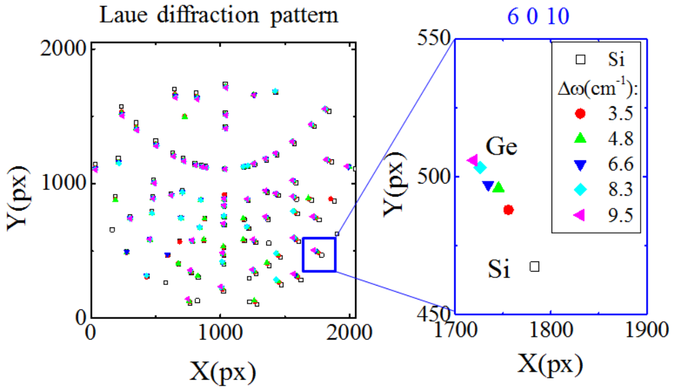

Figure 3. Laue patterns measured in the center of a series of micro-bridges, several reflection indices are indicated, as well as a close-up view of the 6 0 10 reflection. The caption indicates the Raman spectral shift that was measured in these samples prior to the beamtime. All axis are in MAR165 CCD pixels

In order to recover the full strain tensor (i.e. the deviatoric strain + the hydrostatic strain), we assumed that the free surfaces of the Ge membranes were free from normal stress. Additionally, since the rainbow-filter setup was available, we were also able to perform some measurements of the Bragg peak energies. Once the deviatoric tensor and at least one peak energy are known, the full strain tensor can be computed. Our initial assumption was verified, as shown in Figure 4, where the uniaxial strain in the center of a series of micro-bridges evaluated using both approaches were similar. Having confirmed the validity of our assumption, strained Ge membranes were measured up to very large strain values (up to 4.8 % uniaxial), larger than the currently reported results. Thanks to such data, the conversion rule between Raman spectra shift and effective strain was revisited (article 1 in progress). Furthermore, we could extract strain maps from the micro-Laue diffraction measurements and compare them to FEM calculations, as shown in Figure 5 for a micro-cross similar to that shown in Fig.1(b). An excellent agreement is found between the simulations and the experimental measurements (article 2 in progress).

Conclusions and Outlook:

We obtained very statisfactory results from our beamtime: we could measure the deviatoric strain tensor in a wide range of samples and we have verified using the rainbow-filter technique that we could extract the full strain tensor from these measurements. We could also measure strain maps with micrometer resolution, which are consistent with the FEM simulations. Further work includes finishing processing all the raw data, comparing the results with other strain measurement techniques (*e.g.*

Raman spectroscopy, electronic microscopy direct imaging) and investigating in more details the slight asymetry observed between the strain along the [110] and the [-110] orientations (Fig. 5). Upcoming beamtime in the next run will allow us to complete our dataset. Measuring strain for high raman spectra shift is also envisioned as a function of the temperature.

Two articles are currently in preparation from these results. Our preleminary results were presented during 2 talks at the conferences listed below:

- S. Tardif, *et al.* “Strain mapping in Ge microdevices: a combined experimental study” **GDRi MECANO 2015** Grenoble, France
- A. Gassenq, *et al.* “Strain and optical characterization in strained germanium membranes”, **EMRS Spring Meeting 2015**, Lille, France

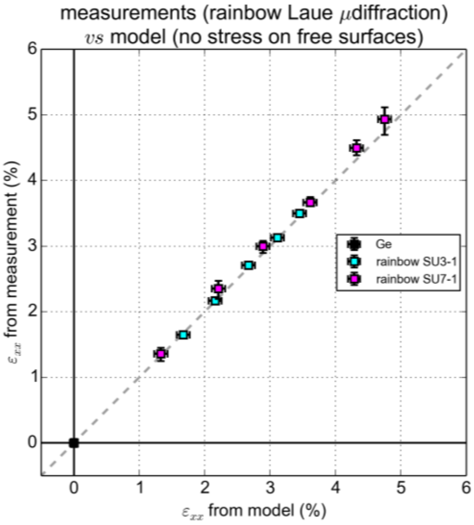

Figure 4. Uniaxial strain in the center of several different micro-bridges, as calculated from the deviatoric strain tensor and the “no normal stress” assumption, and as measured directly using the rainbow-filter technique (error bars were estimated from measurements of several reflections).

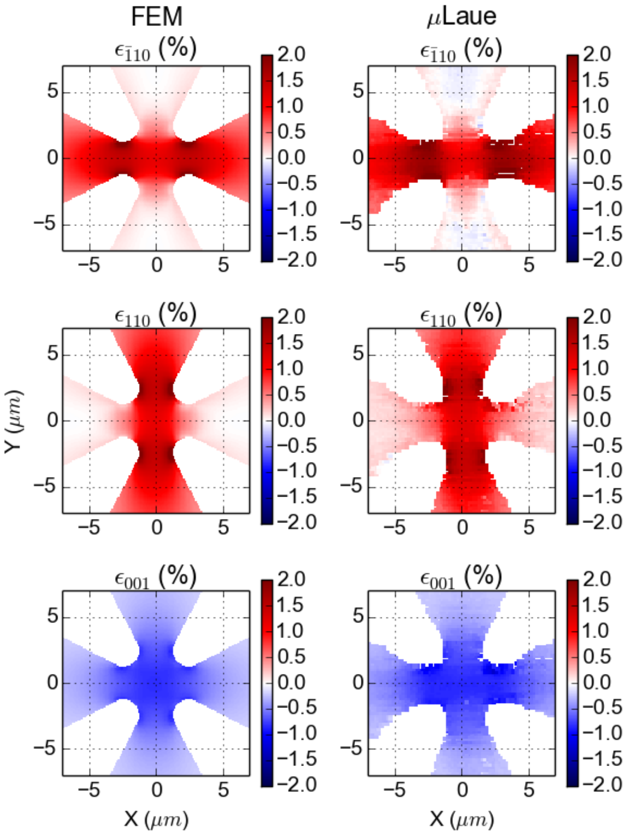

Figure 5. Maps of the different strain components in micro-cross from FEM calculations and µLaue measurements.