



Experiment title: High resolution coherent imaging of strained SiGe-On-Insulator nano-structures for electronics

Experiment number:  
MA-3323

Beamline:  
ID01

Date of experiment:  
from: 20 June 2017 (08:00) to: 26 June 2017 (08:00)

Date of report:  
12 September 2017

Shifts:  
18

Local contact(s):  
Steven LEAKE

*Received at ESRF:*

Names and affiliations of applicants (\* indicates experimentalists):

- Vincent FAVRE-NICOLIN (ESRF) \*
- Joel EYMERY (CEA) \*
- Remy BERTHELON (CEA/ST)
- Gaétan GIRARD (ESRF) \*
- Francois ANDRIEU (CEA)

## Report:

### Objective

Strained semi-conductor nanostructures are an essential component for existing and future electronic devices. The electronic properties of semiconductor nanostructures are strongly influenced by their strain state –a fact which is exploited particularly in micro-electronics, as the mobility of charge carrier can be enhanced by a factor up to  $\times 2,5$  in the case of strained silicon-on-insulator [1]. As the size of functional nano-structures is reduced, the ability to map strain at the nanoscale has become essential.

The material of choice for p-MOSFET channels is SiGe, obtained by condensation, which is currently developed by ST Microelectronics in collaboration with CEA LETI [2] – this method produces a layer with a good crystallinity with a high degree of control of the strain and composition. This quality of the SiGe layers obtained by condensation was recently asserted using TEM and X-ray diffraction [3], but a more quantitative study of individual nano-structures is necessary to understand the effect of etching to the border relaxation, and to assert possible interdiffusion process during the condensation process.

The principle goal of this experiment is to perform a quantitative study of 2D and 3D strain of individual, strained SiGe nano-structures (lines and squares, average 25% Ge composition) on silicon oxide (SiGeOI), with a different crystalline orientation to avoid overlapping diffraction, developed by LETI and ST-Microelectronics for pMOSFET devices. The three samples observed were synthesized at STMicroElectronics using a mask which produces SiGe lines and square or rectangular SiGe islands of lateral size 250, 500 nm and 2, 5  $\mu\text{m}$ , with a spacing between devices typically equal to the object sizes. The thickness of the SiGe objects was either 20 nm or 13 nm.

### Experimental method

We used a monochromatic, KB-focused beam of size 220x100 nm (vertical x horizontal) getting a flux of few  $10E9$  ph/s at 8keV. We used a microscope to get clear location of the samples' zones of interest, and then removed it to go to (004) and (113) Bragg reflection. The profile of the beam along with its phase distribution were retrieved thanks to spiral-scans on a Siemens star via Ptychographic analysis (see Fig 4).

The following samples were studied :

- 20 nm-thick strained SiGe-on-insulator with Nitride on top. For this sample:
  - First we observed squareislands of 250nm x 250nm with a 500nm spacer between islands (so-called zone E6 on the mask) at the (004) reflection and collected data from : ID01's k-map with few scanning points and a small FOV to get a general shape information (figure 1), time-scan to evaluate the beam damage, spiral-scans (256 pts, 10s/pt) at several eta angles (11) to perform 3D Ptychography and eta-scans to achieve 3D CDI. Afterwards, we moved to the (113) reflection and collected energy-scans (11 energies) and eta-spiral-scans (512 pts, 10s/pt). (figure 2,3), in order to collect both 3D Coherent Diffraction Imaging scans, as well as Bragg Ptychography data.
  - Then we observed squares of 500nm x 500nm spacer 500nm (D2) at the (113) reflection and also performed the 3D energy-scans and eta-spiral Ptychography scans.
  - After considering that the exposure time of eta-spiral-scans was too long (with respect to both beam damage and collection time), we decided to reduce it to a maximum of 4s/pt. Squares of 2 $\mu$ m x 2 $\mu$ m spacer 2 $\mu$ m (D4) at the (113) reflection could then be scanned with energy-scans and eta-spiral-scans (256 pts, 2s/pt).
  - Finally we performed spiral-scan and eta-scan on 130nm-wide lines spaced by 130nm (A4).
- 20 nm-thick strained SiGe-on-insulator without Nitride on top. Here we looked at A4, A6 (130nm-width lines spacer 500 nm), D2, D4 E6 and C1 (500nm-width lines spacer 500nm) zones and around the (113) reflection. Eta-spiral-scans (256 or 512pts, 2 or 4s/pt, according to the size of the object of interest) were performed on all these zones, plus energy-scans on squares patterns.
- 13 nm-thick strained SiGe-on-insulator. For this sample we performed eta-spiral-scans on A4, A6, C1, D2 and D4. (figure 3)

Note that all spiral-scans have steps of 0.2  $\mu$ m and that we also measured Ptychography scans on a Siemens stars at several energies in order to retrieve the right probe for each energy and thus be consistent with energy-scans. (figure 4) Preliminary analysis indicates that there is very little variation between the focused X-ray beam at the different energies around 8keV.

## Preliminary results

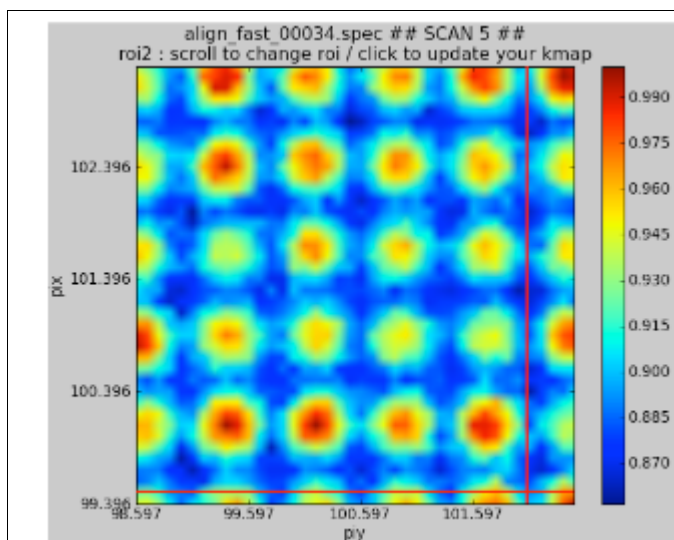


Figure 1: K-map on an area of  $2 \times 2 \mu\text{m}^2$  with step size of 100 nm. We can recognize the shape of several  $250 \times 250 \text{ nm}^2$  squares spaced by 500 nm. This also allows us to estimate the speed of radiation damage for the beam on the centre squares.

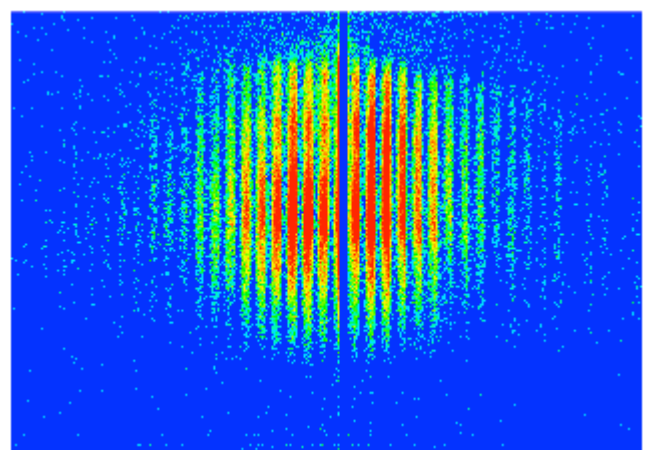


Figure 2: Diffraction pattern of 130nm-width lines spaced by 130nm. Here the lines are perfectly aligned with the equatorial plane thanks to some tilt in phi. The period can be estimated from the fringes seen on the detector: 8 pixel period corresponds indeed to a 260nm period in real space.

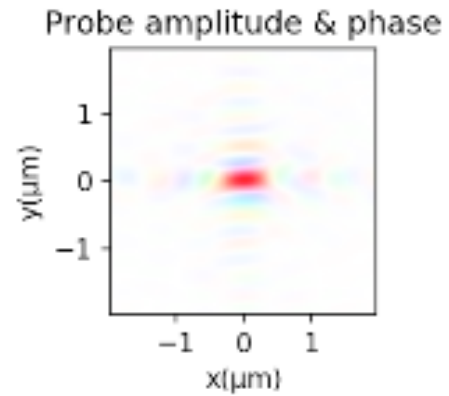
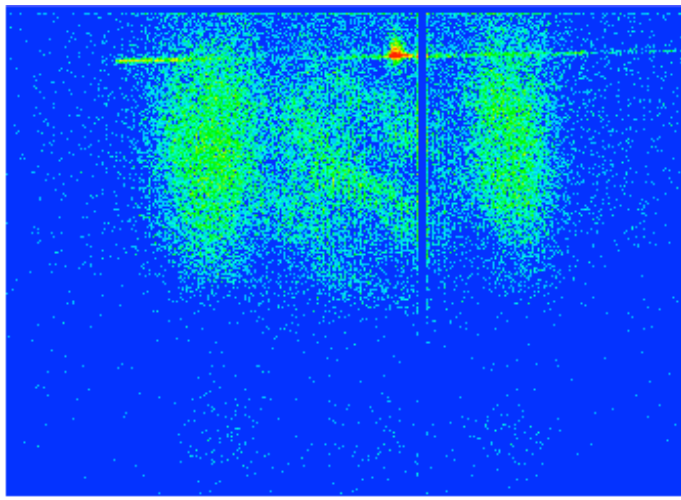


Figure 3: Diffraction pattern on E6 (single  $250 \times 250 \text{ nm}^2$  islands) at the (113) reflection. Here we see that we adjusted the del angle of the detector in order to get the silicon peak (in red, maximum of diffracted intensity) as far as possible from the SiGe diffused intensity. Several shapes are visible on this image, which may lead to a good 3D CDI reconstruction even if we have to be careful of the Si diffused line of intensity (horizontal green line) and of the fact that there are few diffracted photons on each pixel.

Figure 4: Probe amplitude and phase pictured on the same graph, showing that our probe was very symmetric and well-defined, with FWHM less than 300nm in horizontal and 100nm in vertical.

Image obtained via the PyNX software developed at the ESRF by V. Favre-Nicolin.

## Conclusion

This experiment was very successful as we collected data for numerous analysis, whether of 3D CDI or 3D and Back-projection Ptychography. However this type of coherent experiment requires long collection times and very robust algorithms for phase retrieval. The algorithms for data analysis are still being perfected, notably to account for the low count (Poisson noise), and should be fully adapted this autumn 2017. The analysis is still ongoing, and results should lead to an article early 2018.

[1] S. Baudot, S., F. Andrieu, F. Rieutord, and J. Eymery, J. Appl. Phys. 105 (2009),114302

[2] Berthelon et al., 2016 Joint International EUROSIOI Workshop and International Conference on Ultimate Integration on Silicon (EUROSIOI-ULIS). IEEE, 2016.5.

[3] David et al., J. Phys. Chem. C (2016), in press, DOI: 10.1021/acs.jpcc.6b06037