ESRF	**Experiment title:** Kinematic scattering in the nanodiffraction limit: Experimental testing on a model system (SOI ultra-thin films)	**Experiment number**: HC-5386
Beamline: BM32	**Date of experiment**: Oct 3rd to 5th, 2023	**Date of report**: 27 October 2023
Shifts: 11	**Local contact(s)**: Samuel Tardif and Jean-Sébastien Micha	

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Synopsis

This experiment was performed between October 03, 2023 to October 07, 2023 on beamline BM32 of ESRF. We characterized five silicon-on-insulator samples with in-plane and out-of plane radial scans and reflectivity measurements. While we were able to measure the in-plane Bragg peaks for both the SOI and the substrate, we were not able to detect the out-of-plane SOI layer peaks. This might be due to the very low intensity of these Si peaks and the inadequate mis-cut of the specimens used in the experiment. Based on our results we have designed a new sample set. We will manufacture these samples and repeat the experiment for a successful completion.

Samples

We obtained SOI samples, diced into 25 mm x 25 mm squares from a 300 mm diameter (001) wafer, with a nominal reported SOI thickness of 12 nm from the Global Foundries (GF) branch of OnSemi Corp. (Figure 1).

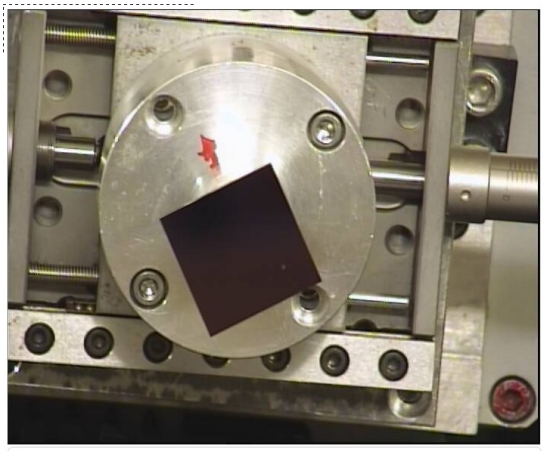

Figure 1: SOI sample mounted on the diffractometer. The chip sides are diced along <001> directions.

This thickness was tentative; an oxidation designed to thin the film ended up removing more film thickness than anticipated, and the remaining film thickness could not be characterized by GF. Our in-house reflectivity

measurements at Columbia and Grenoble (Figure 2) were not sensitive enough to detect the SOI, but showed the presence of the buried oxide layer, with a thickness of approximately 35∓0.5 nm. We initially thought that this was the Si layer, and tried to decrease the thickness by using an isotropic Hydrofluoric-Nitric-Acetic acid (HNA) etch. This etch has very high Si removal rates, around 1 -3 µm per minute (Figure 3). However, its SiO_2 removal rate is very slow (~10 Å per minute). After several etching studies, followed by in-house reflectivity measurements at the IM2NP (Aix-Marseille University), we noted that the reflectivity signal stabilized after one minute of etch, indicating that the (unresolved) SOI signal was completely removed in one minute or less.

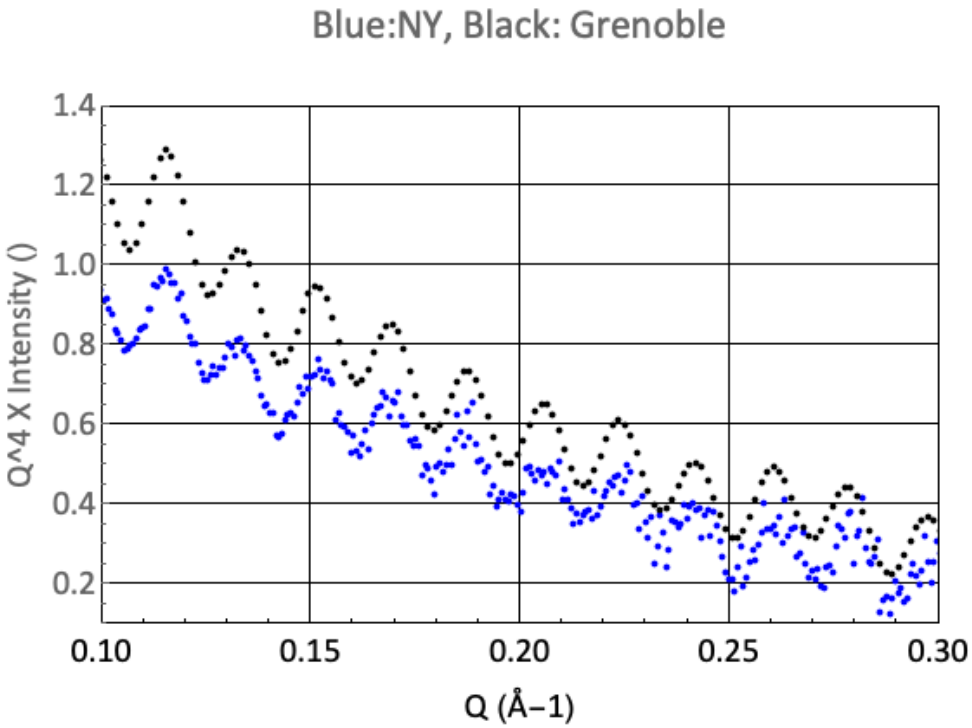

Figure 2: Reflectivity curves measured at Columbia University and LMGP-Grenoble on laboratory instruments. The SOI silicon layer is not visible. The fringes originate from the buried oxide layer interfaces.

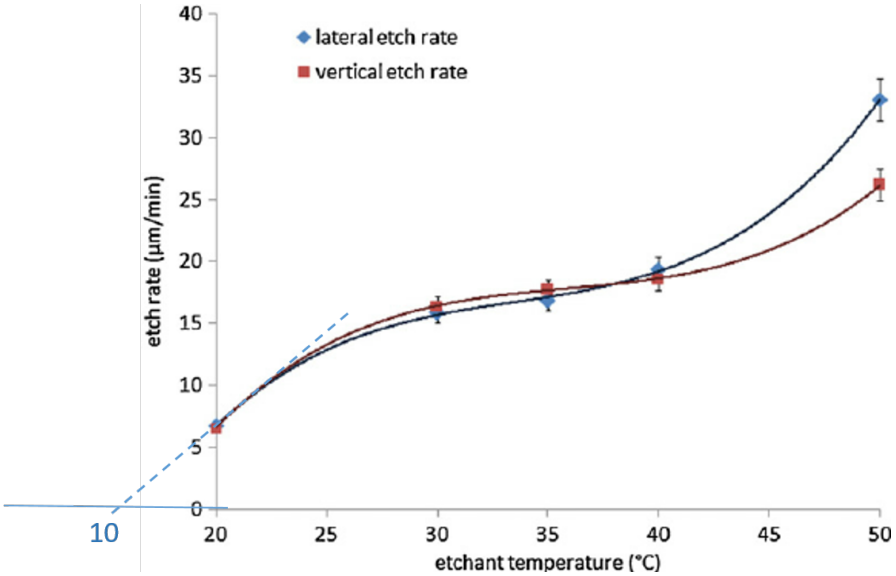

Figure 3: Plot of etch rates versus temperature for the 2:7:1 (HF:HNO3:CH3COOH) solution (fromHamzah, et.al., J. Micromech. Microeng. 22 (2012) 095017)

Consequently, we chilled the HNA etching solution using an ice-water bath, and prepared several samples (Table 1). The SOI thickness of these samples were determined by X-ray reflectivity experiments at the beamline (Figure 4).

Table 1: Etching conditions and structural parameters for the sample set Center-C8. The samples C1-C8 were etched for the times listed in a cooled bath. Only the as-received center sample could be partially characterized with diffraction. The remaining data will be filled in at a future experiment.

Property\Sample→	**Center**	**C1**	**C3**	**C5**	**C8**
Etch time (sec.)	As-received	1-1.5	1.5-2	2.5-3	0.5-1
SOI: (220) a_{220} (Å)	5.4157				
SOI: (220) $\beta°$					
Sbstr. a_{220} (Å)	5.4358				
Sbstr. (220) $\beta°$					
Sbstr. a_{004} (Å)	5.4315				
Sbstr. (400) $\beta°$					

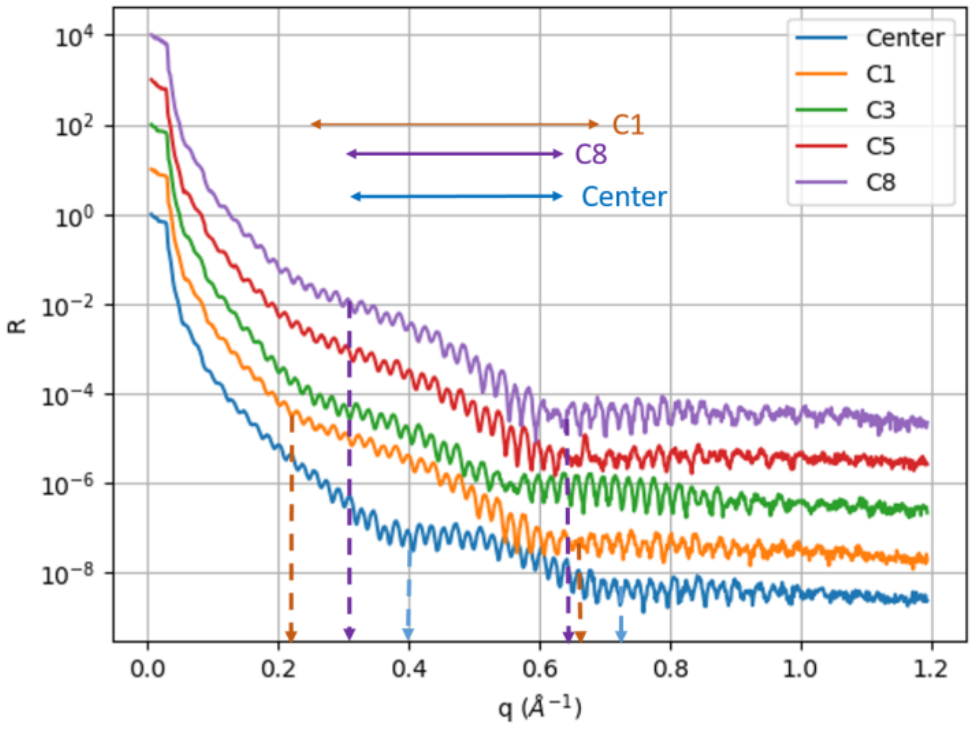

Figure 4: XRR scans (measured at beamline BM32) for the as-received sample (Center) and samples C1, C3, C5 and C8. For these scans the incident beam energy = 27 keV. The as-received film is much thinner than expected. Low-temperature etching is able to decrease the thickness w/o completely removing the film. (measurements performed by Dr Samuel Tardif).

This figure shows that the as-received SOI film was very thin (around 15 Å), and low-temperature etching successfully decreased this film thickness while somewhat increasing the surface roughness as observed from the increasing diffuse scattering, broadening and increasing asymmetry of the in-plane reflections from the SOI (Figure 5).

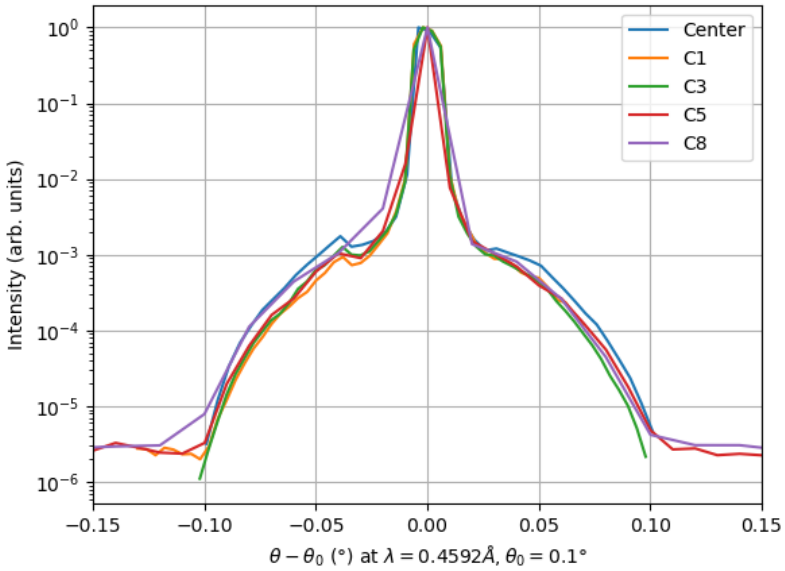

Figure 4: Rocking curves measured at 2theta = 0.2° for the as received sample (Center) and samples C1, C3, C5 and C8. For these scans the incident beam energy = 27 keV.Etching seems to increase sample roughness. (measurement by Dr. Samuel Tardif).

Diffraction Measurements ESRF-BM32

To verify the presence of the SOI layer we conducted in-plane scans of the 220 reflection in the grazing-incidence geometry. Figure 6-a shows these in-plane peaks from both the SOI layer and the substrate. The relative intensities of these peaks could be adjusted (6-b), as expected, by adjusting the grazing-incidence angle; shallower incidence close to the critical angle could completely eliminate the substrate peak. Careful radial scans of the SOI and substrate peaks were used to obtain the in-plane Si lattice parameters for these layers. While we could locate the out-of-plane 004 peak of the substrate easily, we could not locate the peak for the film despite one day of reciprocal space mapping of the as-received substrate (Fig. 7); thus we could not fulfill the primary goal of our experiment, a careful measurement of the peak shape and position of the out-of plane SOI reflections as a function of thickness. This might be due to the very strong signal from the substrate washing out the signal from the SOI. The horizontal diffraction geometry we used to scan for the out-of-plane reflections might have also contributed to the problem due to the large beam divergence associated with this geometry. Unfortunately, we did not have time to do extensive reciprocal space mapping since we ran out of beam-time; it took far longer to align the sample than we anticipated, and we were able to start acquiring real data only on the final night.

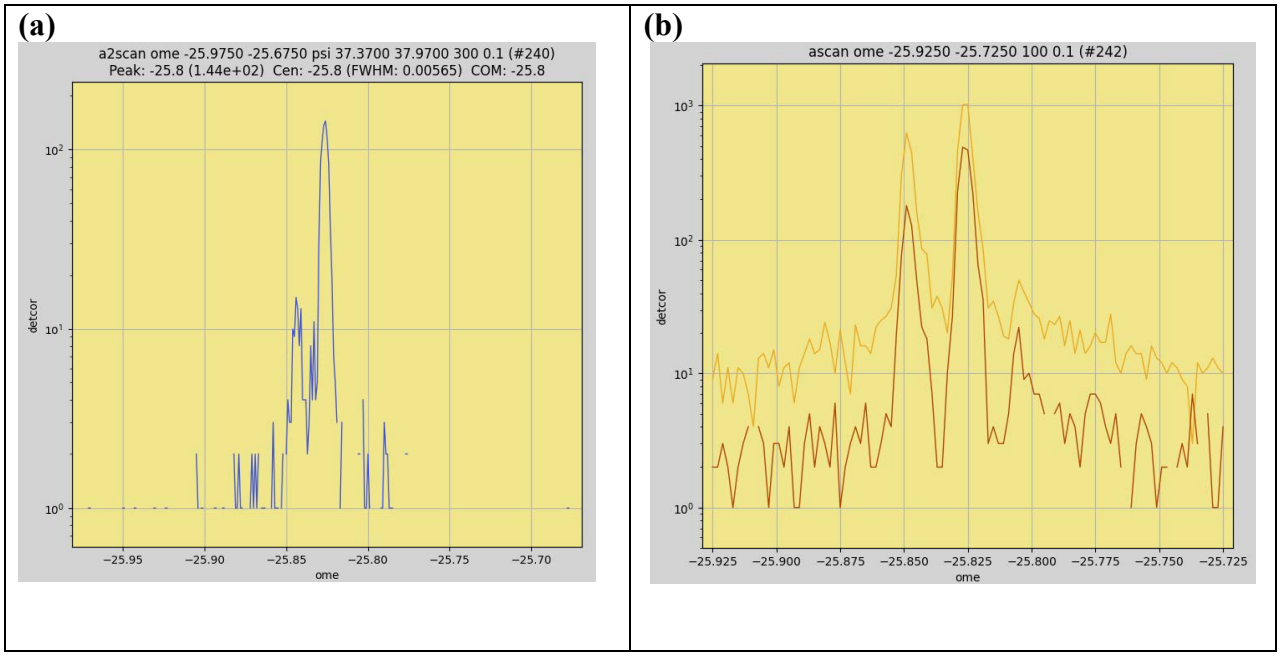

*Figure 5 Radial scans performed at grazing incidence geometry of the in-plane 220 Si reflection with 10 keV beam energy. Both SOI and substrate peaks are visible. Decreasing the incidence angle increases the intensity of the SOI peak (**b**).*

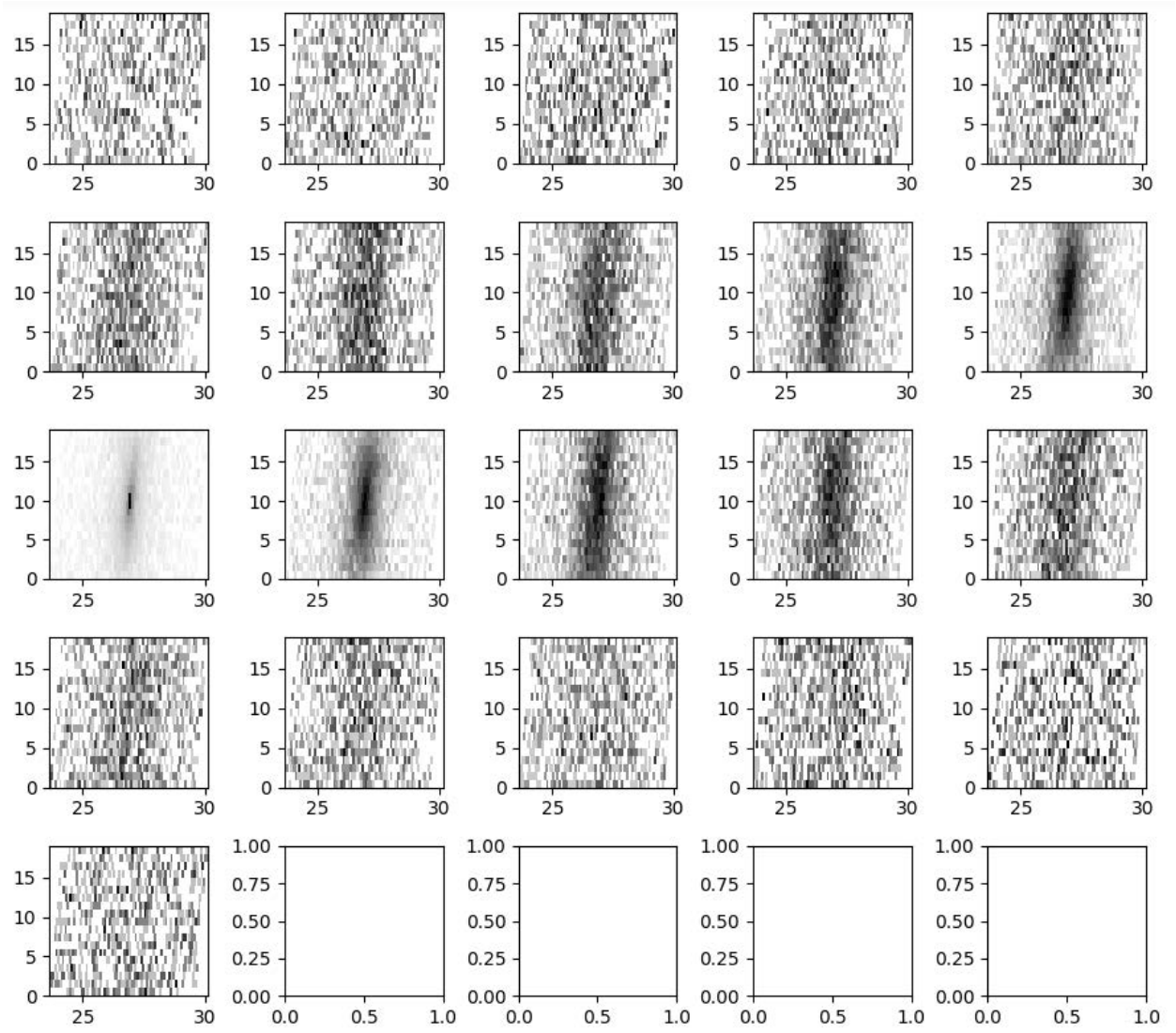

Figure 6: Out-of plane reciprocal space mesh of the "Center" sample. While the 004 substrate peak is clearly visible, we were not able to locate the SOI peak.

Future Work

Our results show that we need a set of SOI samples on a substrate other than Si or, if this is not possible, to obtain samples with large mis-cut angles, preferably greater than 0.75°. Our current results illustrate some issues which need to be resolved:

1-The large difference between in-plane substrate and film lattice parameters, corresponding to ~0.35% strain.

2-The large difference between the in-plane and out-of-plane lattice parameters of the Si substrate.

3-Spurious intensity peaks which have very strong dependence on theta but no dependence on 2-theta. We hypothesize that they might originate from the fluorescence of particles (Fe?) located on the sample edges and possibly related to the dicing process.

Most importantly we need to check and verify the dependence of critical diffraction parameters on film thickness in the nanometer range. Our theoretical analysis shows that peak shape (peak asymmetry, FWHM), peak intensity (integrated and maximum) and peak position depend very strongly on the number of atomic layers participating in diffraction at this level, and the influence of such changes on the structural parameters determined from analysis of experimental data from nano-sized samples should be quantified.

Acknowledgements

We thank Dr. Steffen Kaldor of OnSemi Corp. for his generous help with the SOI samples. We also thank beamline scientists of BM32, Dr. Samuel Tardif and Jean-Sébastien Micha, for their help during alignment and data acquisition. Their assistance was invaluable.