ESRF	**Experiment title:** Lattice parameter and strain in suspended graphene	**Experiment number**: HC-888
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Shifts: 18	**Local contact(s)**: Gilbert Chahine	*Received at ESRF:*

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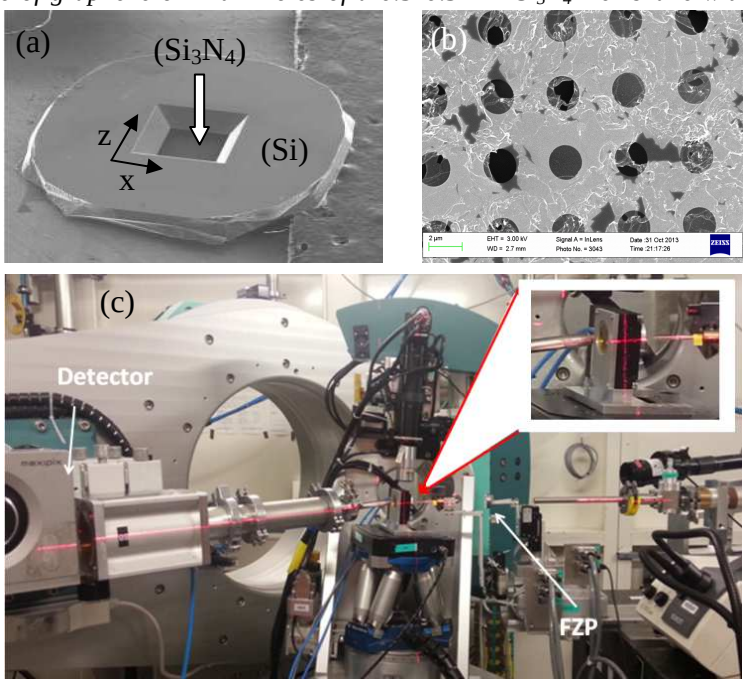
Report:

The aim of this experiment was to measure the lattice parameter of a suspended graphene by Xray micro-diffraction and to caracterize the strain distribution. To minimize the beam footprint, we decided to work under a transmission geometry.

Prepered by chemical vapor deposition, graphene presents several domains over microscopic scale. To suspend it, it was transferred, without any suppoting PMMA, on an amorphous Si_3N_4 membrane (see Figure 1a), with holes of 2µm (Figure 1b). To measure under a transmission geometry (Figure 1c), we developped a new motorized sample environment (inset Fig. 1**c**). Under vacuum, it hosts the Si_3N_4 membrane and avoid beam damage on graphene like the formation of carbone-dioxyde for instance. In addition, it provides the rotation of the sample arround the scattered x-ray beam, necessary for finding a well oriented domain.

Figure 1: *(a) Scanning Electron Microscopy (SEM) image of graphene on 2 um holes of a 0.5x0.5 mm Si_3N_4 membrane with a thickness of 50 nm and sarrounded by 200 um silicon. (b)) Newly developed chamber allowing measurements under vacuum and with the rotation arround the scattered xray beam direction.*



At the ID01 beamline, a sub micrometer beam size is achievable with the available x-ray optics. At 8.5 KeV, a tungsten Fresnel Zone Plates (FZP) focused the beam down to 300 nm (vertical) x 400 nm (horizontal). An order sorting aperture, mounted 2.5 cm upstream of the sample blocked the higher focusing orders of the FZP. The alignment of the sample was achieved by following two main steps. Firstly, the beam size was measured, after mounting the sample, by using implemented tungsten cross in the designed chamber. Thus we positioned the graphene at the correct focal distance, which corresponds to the center of rotation of the goniometer. Secondly, the (220) Si Bragg peak was measured at

the edges of the grid, not only to calibrate the angles but also to define a spatial reference for the graphene. At this reflection, we performed two linear scans across the membrane. These scans (Figure 2 a and b) illustrate the x-ray beam crossing the Si_3N_4 membrane and reveal the edges.

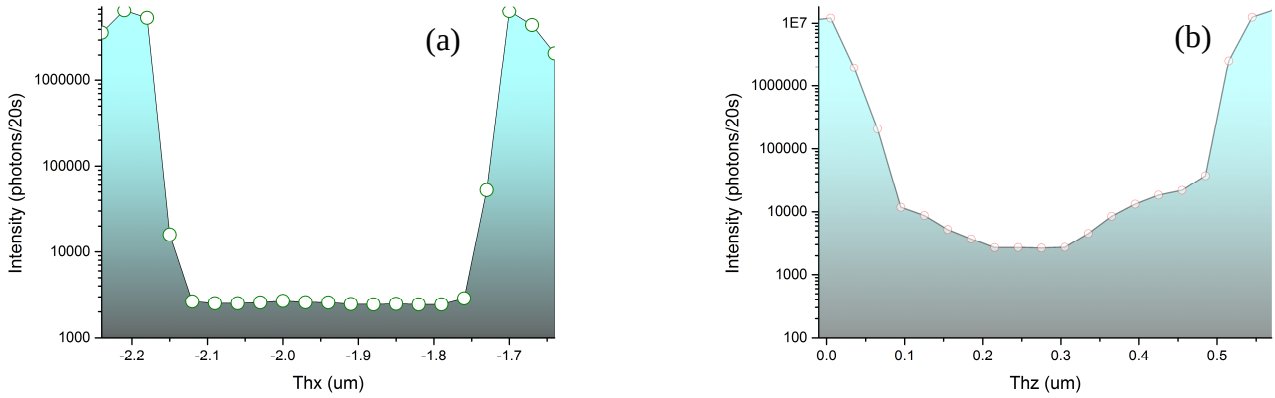


Figure 2: *(a) and (b) are linear scans along x and z of the (220) Si Bragg peak respectively.*

It is known that suspended graphene wets its support on the holes sidewalls, over a few 10 nm (*Z. Han & C. Schwarz, unpublished results)*. This means that the suspended graphene may not be flat enough to detect a diffraction signal. This makes the alignment for a specific crystallographic orientation more challenging.

In Figure 3.a, a graphene layer, having the proper orientation, appears as a spot with higher intensity at the bottom of the 2D mesh. The corresponding detector image (Figure 3.b) shows a main peak. The second peak may be due to a bending in the graphene sheet or a multilayer graphene.

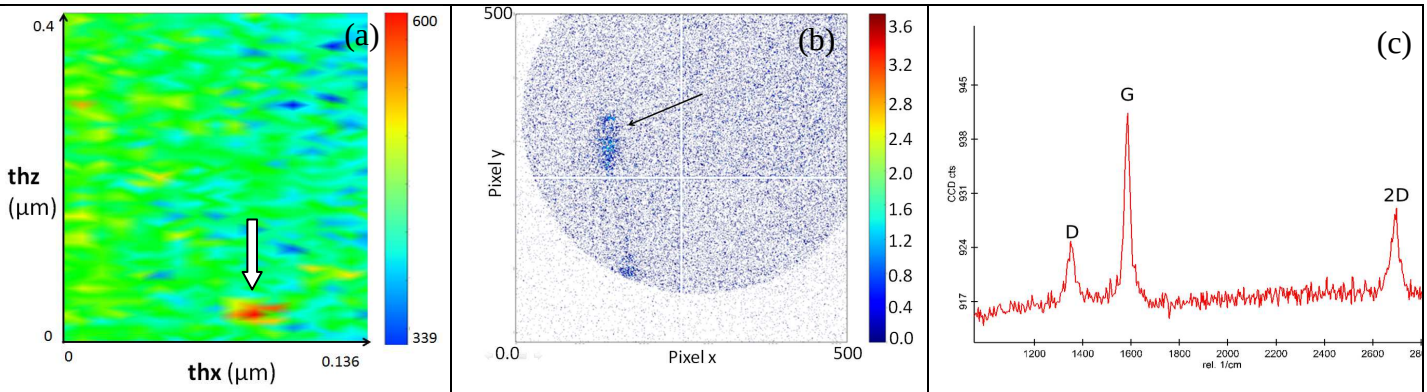


Figure 3: *(a) 2D mesh of the membrane at the graphene (220) Bragg reflection. (b) Maxipix image of the scattering signal of the graphene layer. (c) Graphene Raman spectrum measured, after the ID01 experiment, over the covered holes. The G peak is more intense than the D shows that x-rays didn't induce structural defects and that the graphene quality was relatively preserved.*

The detected background was relatively high and might be caused by the low vacuum in the fly tube between the sample and the detector and also from the kapton foils used for vacuum isolation. Nevertheless, we managed to measure a diffraction signal with an exposure time up to 20s per point. At this position on the sample, Raman measurement shows that is still preserved. Whereas SEM images confirmed that the region measured by x-rays correspond to a suspended graphene covering the complete hole.

Moreover, a complete angular scan was recorded around that region in order to study the strain spatial distribution over one hole. These data will be analyzed by using the X-SOCS software developed at ID01 to retrieve strain distribution.

In conclusion, we succeeded to locate and to measure a free standing graphene lattice parameter with X-ray diffraction in transmission geometry. Next experiments should consider some modifications on the sealing of the chamber and also on the quality of the vacuum to overcome background and graphene reactivity issues.