EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: **Strain distribution in multi and mono layer of suspended graphene**	**Experiment number**: HC2202
Beamline: ID13	**Date of experiment**: from: 07-10-2015 to: 09-10-2015 from: 17-02-2015 to: 19-02-2015	**Date of report**: 21-04-2015
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Report:

The goal of this experiment was to investigate the strain and tilt distributions in the suspended graphene layers and over a multi-walled nanotube by means of x-rays. We performed forward x-ray diffraction measurements in transmission geometry. The sample consisted of 7 layers of graphene suspended over a hole in a silicon matrix (Figure 1a).

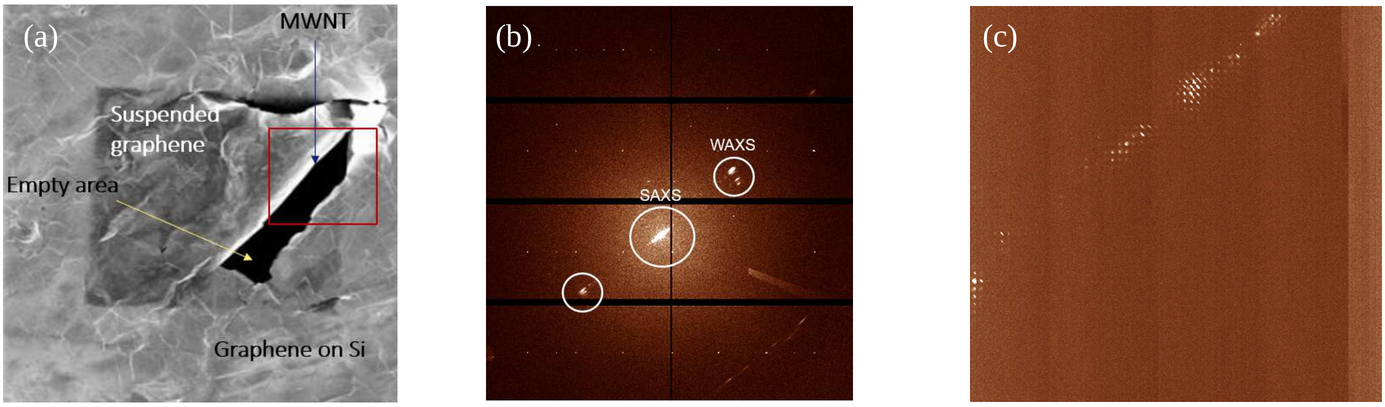
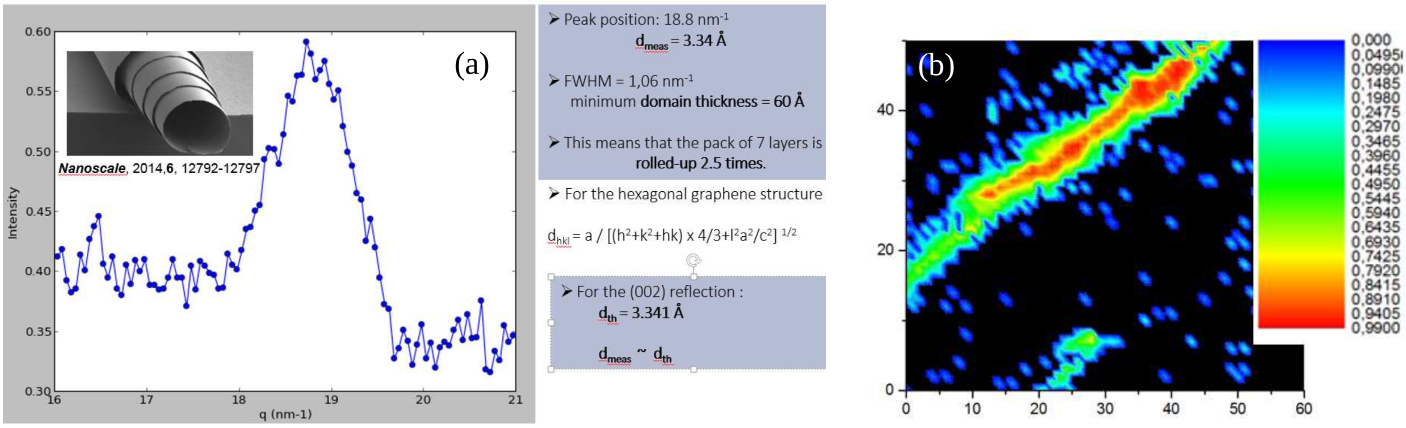


Figure 1: (a) SEM image of the suspended graphene. (b) Raw data detector image of the diffracted SAXS and WAXS signals. (c) Intensity map of the upper right WAXS region as a function of the beam position on the sample. It represents the red square in (a).

Due to the limited time, only a small region was scanned with a focused x-ray beam (150x150 nm^2). This area was selected as it contained suspended graphene, a hole for intensity calibration and normalization and graphene laying on Si. In this configuration, the Eiger detector was set at 156 cm from the sample. The wide angle of view and the high signal-to-noise ratio allowed the detection of SAXS and WAXS signals simultaneously (Figure 1b). From our two consecutive beamtimes on ID13, we measured and demonstrated the stability of 7 suspended graphene layers under high exposure times of x-rays (8s at 14.5 KeV). The recorded data allowed the visualization of a rolled-up graphene forming a multi-walled carbon nanotube (MWCNT) (Figure 1a-c).



- Peak position: 18.8 nm^{-1}
  $d_{meas} = 3.34$ Å
- FWHM = 1,06 nm^{-1}
  minimum **domain thickness = 60 Å**
- This means that the pack of 7 layers is **rolled-up 2.5 times.**
- For the hexagonal graphene structure

$d_{hkl} = a / [(h^2+k^2+hk) \times 4/3+l^2a^2/c^2]^{1/2}$

- For the (002) reflection :
  $d_{th} = 3.341$ Å

  $d_{meas} \sim d_{th}$

Figure 2: (a) line profile along a WAXS signal and calculation of the number of walls in the rolled-up graphene. (b) Orientation distribution of the MWCNT along the rolled-up region.

A fast quantitative analysis showed the possibility of extracting the size, walls number of the MWCNT (Figure 2a) and the orientation of its long axis (Figure 2b). Moreover, from the same measurement, we intercepted the crystal truncation rods (CTRs) of the graphene sheet. Beside the good match between the measured signal and the SEM images, the six-fold symmetry (Figure 3a) of the measured intensity stands for a clear proof that graphene was the material contributing to this diffraction pattern. To our knowledge, this is the first time that suspended graphene is measured by x-rays in a forward transmission geometry. This symmetry appeared at numerous positions corresponding to graphene domains perpendicular to the beam direction (Figure 3b)

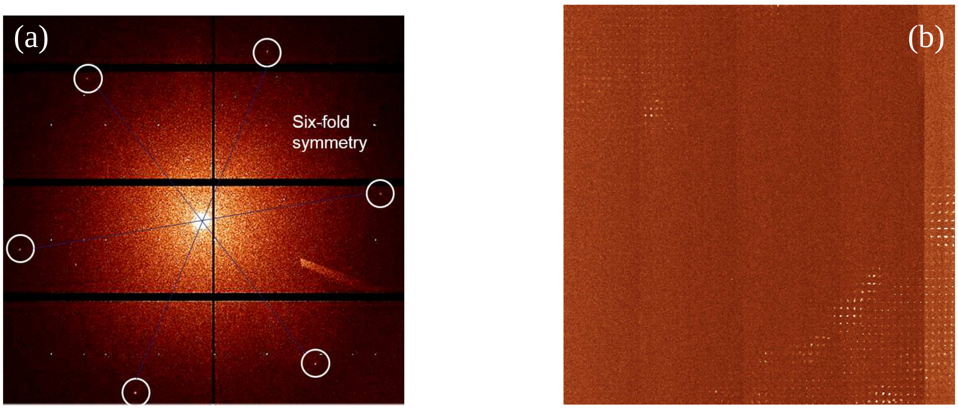


Figure 3: (a) six-fold symmetry of graphene CTRs. (b) Intensity 2D map of the measured sample. The white dots represent graphene perpendicular to the incident x-ray beam.

We managed thus to determine the graphene lattice parameter value from the CTRs. This information is crucial to test the existing theories predicting substantially different values of the lattice parameter. Moreover, we will determine for few positions the Grüneisen parameters of graphene by using a complementary a spatially-resolved Raman spectroscopy for a better understanding of graphene unique electronic transport and optomechanic properties.