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:** Crystal truncation rod scattering from graphene multilayers	**Experiment number**: SI2301
Beamline:	**Date of experiment**: from: 31. 8. 2011 to: 6. 9. 2011	**Date of report**:
Shifts: 18	**Local contact(s)**: Vincent Jacques	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Two-dimensional (2D) graphene sheets containing carbon atoms in a 2D honeycomb structure are nowadays intensively studied due to their unique electronic properties. In contrast to more complex 3D graphite, a linear gap-less energy dispersion of π-bands is found around the K points in the reciprocal space (so-called Dirac cone) [1-4].

The electron structure of the graphene multilayers on SiC substantially depends on the stacking of individual 2D hexagonal graphene arrays. The ABAB stacking of the graphene sheets in which the subsequent sheets are rotated by 60 degrees results in a three-dimensional lattice typical of bulk graphite. As clarified recently, the rotation of subsequent sheets by “magic” angle (30 ± 2.204) deg gives rise to a graphene multilayer with the same linear energy dispersion identical to a single graphene sheet. Therefore, a reliable determination of the rotation angles in multilayer stacks is of importance to predict their electronic properties.

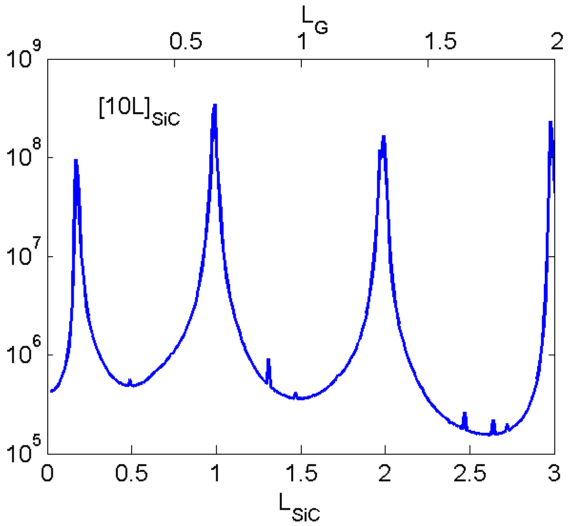

Fig. 1 10L crystal truncation rod showing the SiC diffraction maxima

The aim of he beamtime was to measure the rotation angles between idividual 2D graphene sheets. We investigated a series of graphene multilayer samples prepared by electron-beam annealing of SiC substrates (phase 4H) in UHV. The samples differ in the annealing time, i.e. probably in the thickness of the resulting graphane multilayer. We used a general non-coplanar scattering geometry at ID01 and measured the intensity distribution along various vertical crystal truncation rods perpendicular to the sheets. For the measurement we used a photon energy of 8keV, the primary beam was focused horizontally to enhance the measured intensity. The scattered radiation was detected by a 2D detector placed in the distance of about 1 m behind the sample.

For the measurement we used a hkl-mode with the incidence angle frozen to 0.3 deg (i.e. close to the critical angle of total external reflection).
As an example we present here the results of sample #782 (the longest annealing time, i.e. the thickest graphene multilayer – approx. 30 sheets). Figure 1 shows the mesured $[10L]_{SiC}$ crystal truncation rod (CTR). The curve exhibit usual diffraction peaks for integer indexes L_{SiC}, the maximum at $L_{SiC} \approx 0.2$ is the Yoneda surface peak.
It is well-known from the literature, that the graphene diffraction maxima are azimuthally rotated from the SiC peaks [5,6]. To find the azimuthal positions of the graphene maxima, we carried out a long azimuthal scan at L_G =1, which roughly corresponds to L_{SiC} = 1.5. Then we measured a series of vertical CTR scans crossing the graphene peaks found at various azimuthal directions (see Fig. 2 as an example).

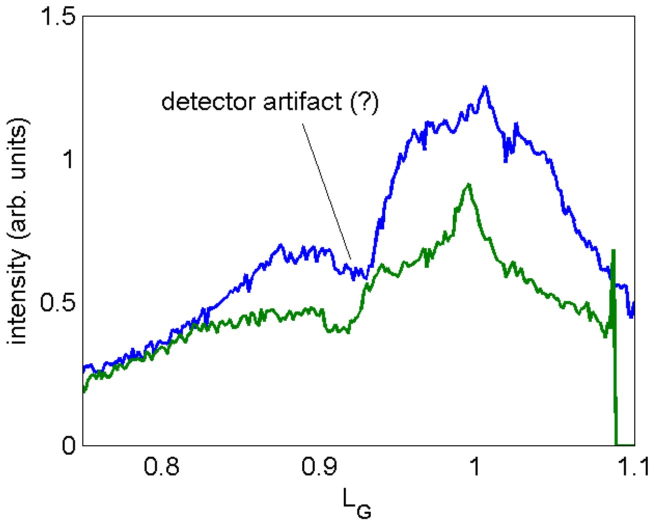

Fig. 2 Two L scans across the graphene maxima taken for different azimuths

Preliminary numerical simulations indicate that the L-width of the graphene diffraction maximum for a given azimuthal angle ϕ with respect to the azimuth of $[100]_{SiC}$ is inversely proportional to the amount of graphene bi-layers with the mutual rotation angle connected to ϕ [6]. A detailed analysis of the CTR scans will therefore bring information on the population of the rotation angles.

[1] K. S. Novoselov *et al.*, Science **306**, 666 (2004).
[2] K. S. Novoselov *et al.*, Nature (London) **438**, 197 (2005).
[3] C. Berger *et al.*, Science **312**, 1191 (2006).
[4] A. K. Geim and K. S. Novoselov, Nature Mater. **6**, 183 (2007).
[5] J. Hass, W. A. de Heer, and E. H. Conrad, J. Phys.: Condens. Matter **20**, 323202 (2008).
[6] M .Sprinkle et al., J. Phys. D: Appl. Phys. **43**, 374006 (2010).