Experimental report : SI2193 beamtime on BM32.

Graphene is the name given to a monoatomic layer of carbon atoms arranged in a honeycomb structure. A strong effort is being focused worldwide on understanding the interaction between graphene and a metal. This is on the one hand in view of optimizing the growth of graphene on metals, which appeared in the last three years as a promising route towards mass production of graphene [1]; on the other hand this is for fundamental questions pertaining to the manipulation of graphene's electronic band structure and/or it's interaction with magnetic objects (thin films, NPs, atoms). Graphene/Ir(111), which preparation was optimized by us in the last few years [2], now appears the prototypical system for addressing such fundamental questions using surface science methods [3]. Noteworthy, the graphene and Ir(111) surfaces exhibit a lattice of coincidence sites (CSL) usually referred as a moiré, which may serve as a very efficient template for the self-organized growth of a variety of clusters [4].

The goal of the proposal was to: -1- optimize the preparation of the Ir(111) surface with characterization with X-rays, RHEED and AES; -2- optimize the growth of graphene on it according to a well established procedure, and fully characterize the structure and morphology of the grapheme layer; -3- Study the first stages of the growth of Pt on GEM. This was performed in the INS/BM32 UHV chamber, which is equipped with surface science tools for surface preparations, gas injection (Ethene) and molecular beam epitaxy (MBE) sources. Surface X-ray diffraction (SXRD), Grazing Incidence X-ray Diffraction and Scattering (GIXD & GIXS) and Grazing Incidence Small Angle X-ray Scattering (GISAXS) were combined *in situ* in UHV during growth:

-SXRD/GIXD: in-plane measurement of the Ir(111) and graphene over Ir(111) were performed together with first measurements of graphene rods

-GISAXS [5] was performed on the Gr film.

The experimental conditions in the INS instrument leading to high quality graphene on Ir(111), noticeably low mosaicity of Ir(111) and graphene, and large, atomically smooth Ir(111) terraces, have been determined using RHEED and surface X-ray diffraction (SXRD). For this repeated cycles of Ar^+ sputtering and annealing (1500 °C) of Ir(111) are needed.

As an illustration of the wealth of information which can be obtained from surface X-ray diffraction/scattering we briefly discuss some of the results which were obtained during the S1(2193) beam time. We show that a weak interaction with the metallic substrate imposes partial matching, deformations, in-plane rotations and shearing even in high quality samples. We find indications for incommensurate phases, which do not exclude commensurate ones, and finally evidence that the distribution of phases can be tuned thanks to the temperature of the sample.

In-plane cuts of reciprocical space yeald to graphene lattice parameter determination, at room temperature it corresponds to $a_{||}$ = 2.4470±0.0005 Å, the value of the in-plane projection of the lattice parameter of the graphene. Considering the geometric corrections (nanorippling) the measured lattice parameter is about 0.3% smaller thant that of isolated graphene. A superstructure peak is found just between the K^2_{Ir} and the K^2_{Gr} corresponding to a 24.80±0.20 nm superperiodicity (Figure B). The four order measurements of graphene in-plane contribution peaks shows linear width increase in the radial direction while it remains constant in the azimutal direction. This can only be understood as the consequence of modulations of in-plane projection of graphene's lattice parameter. Scattered intensity along graphene rod shows a broad signal (Figure A), which can reflects weak modulation of the out-of plane graphene superstructure nanorripling. Such defects were never reported so far for graphene/metals, and are of prime importance for future electronic characterizations of

graphene layers produced from metal surfaces. The epitaxial matching of graphene on Ir(111) can be tuned by varying the temperature, an in-plane projection of the lattice parameter, $a_{||}$ = 2.455±0.001 Å, is evidenced at 700±60 °C. This is noticeably different from the room temperature value (+0.34 %), but close (-0.1 %) to the expected lattice parameter for isolated graphene at this temperature, a_{800} = 2.4581 Å (*10*). The superperiodicity slightly decrease (24.53±0.19 Å), radial scans width increase as a function of the peak order but to a lesser extend than at room temperature, this mean that various epitaxial matchings are present in the graphene film at this temperature.

GISAXS images of bare graphene have been performed (figure C), the superstructure signature is observed after several hours of acquisition, but the low brilliance of the bending magnets doesn't allow us to performed quantitative measurements.

Given that the first experiments yielded a wealth of unexpected results already with the simplest system [graphene/Ir(111)], we are confident that the system is suitable for a thorough and fine structural investigation with X-ray probes, nevertheless high flux from insertion devices is mandatory to reach quantitative measurements of graphene GISAXS maps and surface Rod's to get a complete sketch of it's structure. We wish to underline the fact that the field is highly competitive, other research groups currently investigating quite similar systems, if not the same ones, at other synchrotron sources (ANKA, Swiss Light Source, Bessy, Elettra, and ESRF).

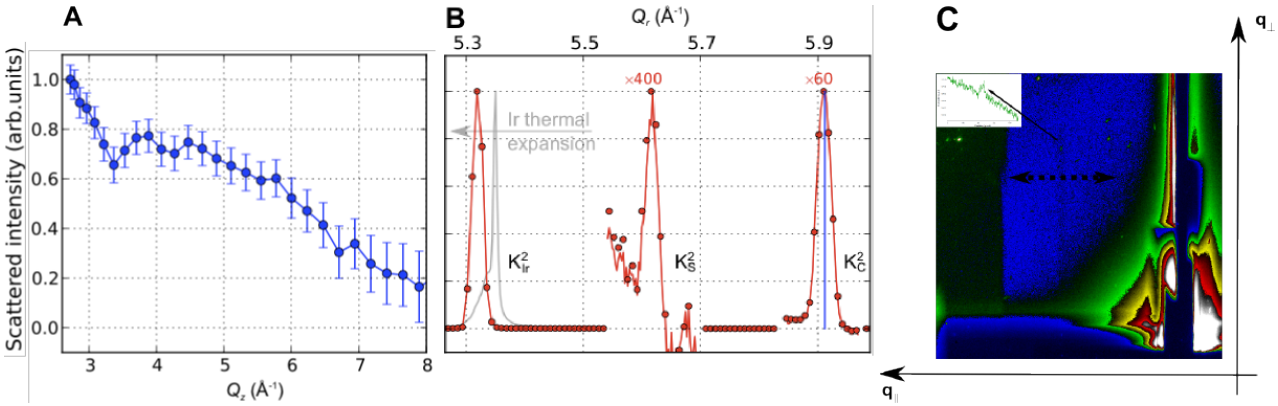

figure 1: A. scan of the scattered intensity along the out-of-plane scattering vector for a graphene rod; B. Radial scan close to the second order Brillouin zone; C. GISAXS map in the [1100]C direction for bare graphene/Ir(111).

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