ESRF	**Experiment title:** Graphene on Al2O3: a surface x-ray diffraction study	**Experiment number**: HC-1966
Beamline: ID10B	**Date of experiment**: from: 19.06.2015 to: 23.06.2015	**Date of report**: 10.02.2016
Shifts: 12	**Local contact(s)**: Dr. Giovanni calogero Li destri nicosia	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Michael Hanke* Paul-Drude-Institut, Hausvogteiplatz 5-7, 10117 Berlin

Dipl.-Phys. Thilo Krause* Paul-Drude-Institut, Hausvogteiplatz 5-7, 10117 Berlin

Dr. Marcelo Lopes Paul-Drude-Institut, Hausvogteiplatz 5-7, 10117 Berlin

Dr. Timo Schumann Paul-Drude-Institut, Hausvogteiplatz 5-7, 10117 Berlin

Report:

The objective of the project was to study the in- and out-of-plane morphology of single- and few-layer graphene grown on different insulators (such as Al_2O_3(0001)) by molecular beam epitaxy. Especially important is to gain information about the epitaxial alignment of the graphene nanocrystals forming the layers in respect to each other and to the underlying substrate. The results obtained by grazing incidence diffraction (GID) will be fundamental for the elucidation of the nucleation and growth mechanisms of graphene layers prepared by MBE. The proposal is in direct relation to a previous beamtime (SI-2449), where we have investigated the structure of graphene layers on SiC [1].

Therefore we have focused on a set of six samples, grown at 950°C and 1150°C, respectively. At each temperature subsequent morphological steps have been established - as a function of growth time. From AFM data (not shown) we can distinguish three stages: (a) initial nucleation (with a non-closed layer of graphene), (b) a single and closed layer of graphene and eventually (c) multilayer graphene (for a TEM micrograph see fig.1 of the respective proposal).

For all the samples we have performed grazing incidence diffraction (GID) at an angle of incidence close to the critical one for total external reflection. To get an overview, in-plane maps have been recorded, see e.g. fig.1 of this report. Due to symmetry reasons it was sufficient to probe only an azimuthal area covering a range of slightly larger than 30°, however covering in radial direction at least the sapphire (11.0) and (30.0), which later serve as reference. Both graphene contributions,

G(10.0) and G(11.0), demonstrate that the graphene layer is 30° azimuthally off the sapphire substrate, i.e. Al_2O_3[11.0] || G[10.0] and thus Al_2O_3[10.0] || G[11.0]. This relation has been found for througout all growth stages and for different temperatures. Further on the graphene reflections show a pronounced angular spread of about ± 4° indicating a preferential, however, not perfect alignment with respect to the substrate. From the radial position, see detailed scans close to the graphene contribution, one can precisely estimate the lattice parameter of graphene, which is in this case 2.448(1) A. From the other data we could observe a clear trend towards larger lattice parameters for the three different growth stages changing its value from 2.442(1), over the given example 2.448(1) A towards 2.450(4) A.

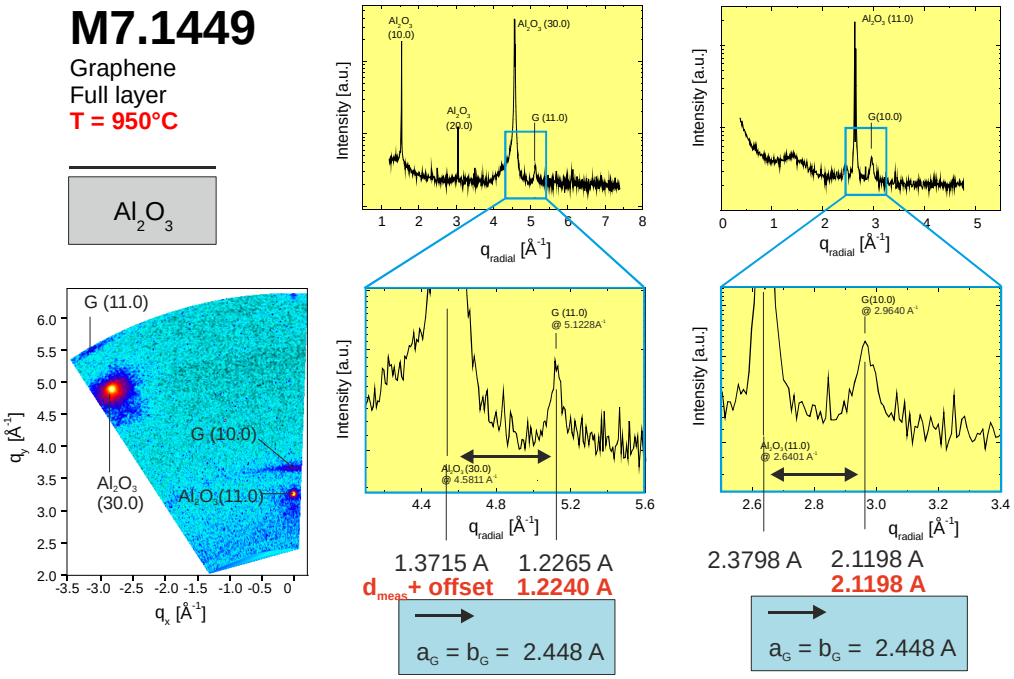

Fig.1: Schematic view of one of the investigated graphene samples carrying a single layer of graphene on a c-plane sapphire substrate. Grazing incidence diffraction in-plane map showing Al_2O_3 reflections, which may serve as reference and graphene contributions, G(10.0) and G(11.0). Radial scans with sufficiently high resolution along different in-plane direction yield a precise estimate of the graphene lattice parameter.

GID serves here as an excellent tool to gain precise experimental information of the lattice parameters, the relative uncertainty amounts to about 0.1%. This is, on the other hand, a parameter, which can be delivered by improved functional theory calculations, which provide the theoretical frame to describe the observed phenomenon. A publication based on these data is in preparation.

References:

[1] T.Schumann, M.Dubslaff, M.H.Oliveira, Jr., M.Hanke, J.M.J.Lopes, H.Riechert
The effect of the buffer layer coupling on the lattice parameter of epitaxial graphene on SiC(0001)
Phys. Rev. B **90**, 041403(R) (2014)