EUROPEAN SYNCHROTRON RADIATION FACILITY

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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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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: Structure of metal clusters on graphene**	**Experiment number**:
Beamline: ID32	**Date of experiment**: 19.11.2010 to 1.12.2010 and 24.3.2011 to 2.4.2011	**Date of report**: 27.4.2011
Shifts: 30	**Local contact(s)**: Jörg Zegenhagen	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Epitaxial growth on transition metal surfaces is a promising method for graphene preparation. Especially on Ir(111) we have previoulsy proven a high degree of structural quality of graphene [Coraux2008, NDiaye2008], while its electronic structure is largely identical to the one of free graphene [Pletikosic2009]. In a previous beamtime at ESRF, we were able to precisely measure the distance between the graphene sheet and the metal substrate [Busse2011] using x-ray standing waves (XSW). Together with extensive DFT calculations we could thereby show that graphene on Ir(111) is only weakly bound to the substrate through Van-der-Waals-forces. The experimental data also showed that graphene on Ir is significantly buckled, and that furthermore the amplitude of this buckling depends on the graphene morphology (small isloated flakes are less buckled than a percolated film). We tentatively attributed this to different stress states of the film. One objective of the current experiment was to harden this assumption. Another goal of the experiment was to investigate the changes in carbon-carbon and carbon-metal bonds upon growth of metal cluster lattices on the graphene [NDiaye2006], especially to test the proposed rehybridisation model of cluster binding [Feibelman2008].

The experiment was conducted in two separate runs. In the first run we intended to use a new electron-beam heater for sample heating, as rather high temperatures are needed to clean Ir(111) and subsequently grow well ordered graphene onto it. However, we were faced with serious problems concerning this heater (arcing, shortcuts) so finally we had to rely on a standard heater not capable of reaching the high temperatures needed for in-situ graphene preparation. Consequently, we performed a set of measurements on ex-situ prepared graphene. Here the quality of the film was lower as compared to our first beamtime, where we studied in-situ grown graphene, resulting in a larger spread of the observed carbon heights above the substrate. Still we performed XSW measurements using the (111), (222), (333), and (444) reflection. As this experimental run was also our first use of the new HAXPES-system (new chamber, new photoelectron spectrometer), we gained useful information for the second run.

In the second run we could use an improved e-beam heater capable of reaching the necessary temperatures. We also used a newly acquired single crystal with much better crystal quality. On this substrate we prepared well ordered, fully closed graphene layers in situ according to a refined recipe [vanGastel2010] and checked the film quality using LEED. We performed an XSW experiment using both (111) and (222) reflexes. A first analysis yielded a bond distance which coincides within errors with the one determined earlier. However, the

buckling seems to be much larger in line with our espectations. Furhter data evaluation of this experiment (which was conducted only recently) is under way.

On the closed graphene film we deposited 0.1 ML of Pt to prepare a cluster lattice (using a calibrated source supplied by ourselves). We analysed XSW of the (111) and (222) reflex and observed a broadening of the carbon height distribution, but a rather narrow distribution of Pt heights. Also here further evaluation is necessary.

Finally, we prepared rather small flakes of graphene to extend the range of studied morphologies all the way from small flakes to a closed layer. Also this sample was studied using XSW of the (111) and (222) reflection. The data analysis has still to be performed.

Technically, we faced problems using the new Kappa-maipulator which got stuck several times during the measurements. This reduced the ammount of usable shifts.

In summary, the second round can be considered as a success, also due to the problems identified and solved after the first run, which therefore served as an important trial. Graphene can now routinely be prepared in situ at ID32. Full evaluation of the data is still necessary and under way. Prelimary evaluation points to an increased buckling of graphene for closed films and a change in the bonding pattern after depositon of metal cluster.

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