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| HZDR HELMHOLTZ ZENTRUM DRESDEN ROSSENDORF<br>ROBL-CRG | **Experiment title:**<br>In-situ XRD and XRR during graphene and Boron-Nitride growth | **Experiment number**:<br>**20-02-715** |
| **Beamline**:<br>BM 20 | **Date of experiment**:<br>from 20.06.2012 until 26.06.2012 | **Date of report**:<br>12.03.2013 |
| **Shifts:**<br>18 | **Local contact(s)**:<br>Dr. Carsten Baehtz (baehtz@esrf.fr) | *Received at ROBL:* |

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**Results**

Chemical vapour deposition (CVD) of graphene on Copper catalysts provides to date the most scaleable and controllable method for large-area, high quality graphene production [1]. While CVD can enabe integration of graphene into a variety of promising application areas, such as electronics, *in-situ* experimental verification of the suggested surface growth mechanism of graphene on Cu has remained largely elusive so far [2,3].

During this beamtime, we used in-situ X-ray diffractometry (XRD) during salient stages of CVD to follow the cyrstallographic state of the Cu catalyst *in-situ* under a variety of working reaction conditions. In general we find that as loaded Cu shows reflections corresponding to metallic fcc Cu. Upon heating/pre-treatment we observe an decrease of the reflection width in the fcc Cu, consistent with crystallisation and grain growth. FWHMs of the reflections approach the instrumental resolution of the XRD setup, implying the formation

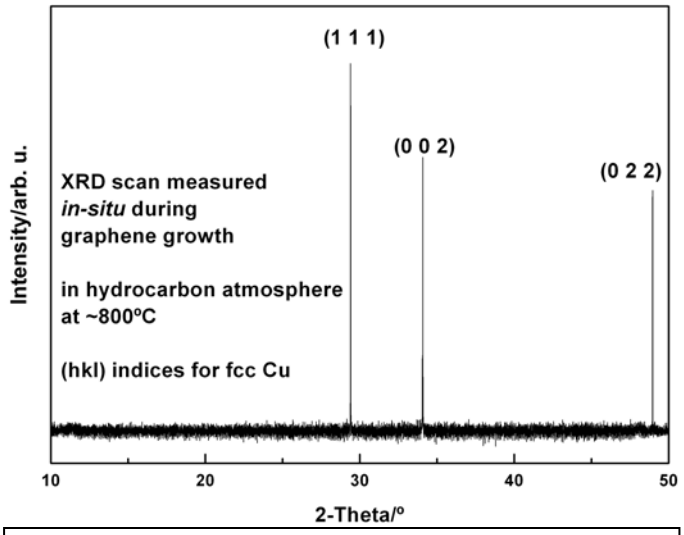


**Figure 1: In-situ XRD scan during graphene growth, evidencing metallic Cu as the active catalyst.**

of massive grains [1]. When we grow graphene by exposure of the Cu to hydrocarbon gases, we find that metallic fcc Cu remains as the only detectable phase (Figure 1). This *in-situ* XRD data corroborates complementary *in-situ* XPS experiments, confirming that for graphene growth metallic Cu is the active catalyst state on both the catalyst surface and in the catalyst bulk. Publication of the results is in progress.

## References

[1] Kidambi, Ducati et al. *J. Phys. Chem. C* **2012**, *116*, 22492
[2] Weatherup, Bayer, et al. *Nano Lett.* **2011**, *11*, 4154
[3] Weatherup, Bayer et al. *ChemPhysChem* **2012***, 13*, 2544