ESRF	**Experiment title:** **In-situ X-ray study of Graphene formation on SiC(0001)**	**Experiment number**: SI2048
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Report:

Graphene, a single atomic layer of graphite, shows remarkable electronic properties, for one because of its truly two-dimensional structure. These properties, which include ballistic electronic transport on the micrometre scale and electric-field-controllable conductivity, are believed to hold promise for future applications in the so-called post-Moore's law era. Furthermore, the two-dimensional electron gas in graphene has become the playground for many theoretical and experimental studies. For these reasons, graphene has undoubtedly turned into a 'hot topic' in materials science over the past few years. Graphene can be obtained in different ways, for example by exfoliating flakes from larger graphite samples [1] or by decomposing small organic molecules at metal surfaces. Another way of obtaining graphene is by heating polished SiC(0001) crystals. This way of growth and optimization of graphene was initiated at the Georgia Institute of Technology [2]. One of the advantages of epitaxial graphene (EG) over free-standing samples is that of the substrate-induced opening of a band gap, which paves the way for real applications [3].

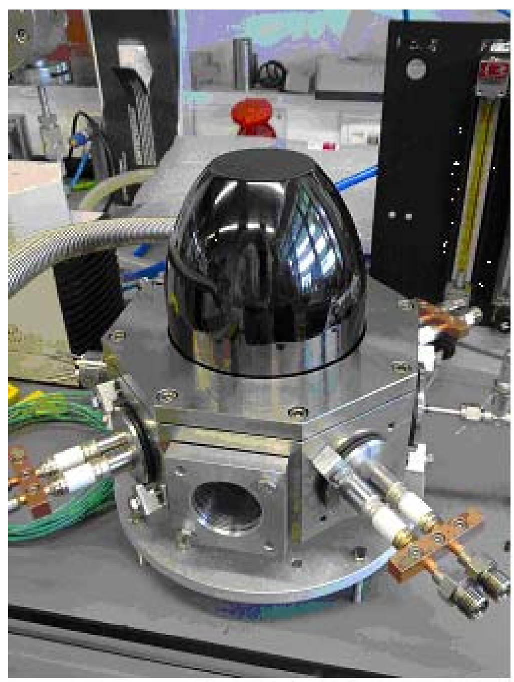

Figure 1. Graphene reactor for in-situ surface x-ray diffraction. The vitreous carbon dome is x-ray transparent.

The annealing, and subsequent Si evaporation, in ultra high vacuum of either of the two polar surfaces of SiC(0001) results in an epitaxial graphene layer [2]. In this way, graphene layers have been successfully grown on two different polymorphs of SiC substrates, namely 4H and 6H, which differ in the number of Si-C bilayers along the c-axis in the unit cell. Both structures can be either Si or C terminated. Remarkably, on the C-face of 4H-SiC a film of several 'graphene' layers can be grown that shows the same electronic properties as a single layer. The key to this behaviour is thought to be theorientational disorder in the stacking of the separate layers [4]. It seems likely that the fact that these layers are formed at a buried interface plays an important role in this stacking order. It is not clear at present how this process varies when using the different terminations of SiC.

For the purpose of studying the formation of Graphene on SiC substrates in-situ, a special sample chamber has been designed and constructed, see Fig 1. The temperatures that are obtained with this set-up range from room-temperature up to 2200K, as measured with a pyrometer. Although the set-up worked without any problems, it was not possible to grow graphene layers during the experiment. By the end of our beamtime we identified a small leak, which caused the oxygen partial pressure to be too high for successful graphene growth. Back in Nijmegen we isolated the leak and now the conditions in the graphene reactor meet the requirements for the proposed experiments.

Results

Several CTRs were measured from the SiC substrate at different temperatures. An already grown graphene sample was completely characterized. Several CTRs were measured from this sample. In Fig. 2 some typical measurements are shown. The specular reflectivity agrees qualitatively with earlier results [5].

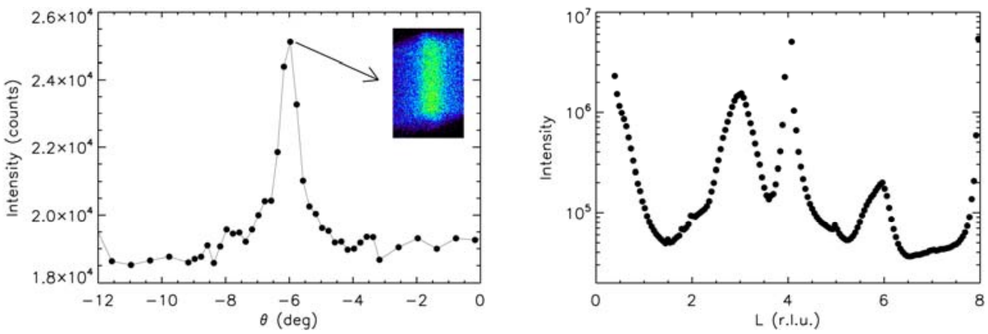

Figure 2. SXRD measurements from an approximately 4 layers thin Graphene film on a 4H-SiC(000-1) substrate. (left) Rocking scan at the Graphene (1,0,L) position, with L close the the Bragg condition. The inset shows the streaky CTR signal, that is measured with the Maxipix detector in the maximum of the scan. (right) Specular reflectivity, showing the Bragglike peaks around L=3 and L=6. The substrate Bragg reflections appear at L=4 and L=8.

Conclusions

The graphene reactor allows for in-situ SXRD experiments up to very high temperatures (~1800°C) and ambient pressures. To our knowledge, this is the first time that in-situ SXRD has been achieved at such high sample temperatures. Unfortunately, a leak that was present during the beamtime prevented us from growing graphene. Nevertheless, we have obtained extensive SXRD data from an already grown sample. These data are currently being analyzed.

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[3] S.Y. Zhou *et al.*, Nature Materials **6** (2007) 770

[4] J. Hass *et al.* Phys. Rev. Lett. **100** (2008) 125504

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