ESRF	Experiment title: **Epitaxial Graphene on SiC: Effect of hydrogen absorption**	**Experiment number**: SI-2225
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Experimental Report:

The purpose of this proposal was the investigation of the structure of graphene on SiC, espacially the understanding of the hydrogen induced band structure changes of the graphene on the so-called Dirac cones required a high resolution GIXD study upon H deposition in real time.

Epitaxial growth of graphene on SiC is obtained by vacuum graphitization due to Si depletion of a SiC single crystal surface. This method has been shown to produce ultrathin carbon sheets and occasionally a graphene mono- or bi-layer. Most importantly, the first interfacial layer of carbon grown on SiC differs substantially from the ideal graphene structure. Recently, we have demonstrate a method of preparing graphene on 3C-SiC(001) that results in a significantly improved film quality and large area. The formation of a graphene monolayer has been characterized by LEEM, STM, XPS and magneto-transport techniques. Recently researchers have found that the interaction of hydrogen with graphene can dramatically affect its electronic

properties, rendering this nearly ideal two dimensional conductor insulating. Atomic hydrogen chemically binds to graphene in a pair configuration and when both sides are saturated the sp2 bonds of graphene are converted to sp3 bonded “graphane,” which is highly insulating.

The aim of this proposal was to study the structural ordering properties of hydrogen adsorbed on graphene using GIXD.

Sample#1 :

We first used a bulk 3C-SiC(001) sample. After alignment using bulk bragg peaks the surface diffraction has been explored in plane. It appears that the reciprocal space was polluted by many stacking faults making the identification of a top graphene layer fairly impossible (figure 1). Quantitative (11L)(22L)(20L)(-20L)(10L)rocking scan integrated crystal truncation rods were measured. Because of the poor quality of the surface in terms of extra features it is highly speculative to extract a reliable structural model from these data. The mosaic spread of the substrate was poor and in the range 0.5°.

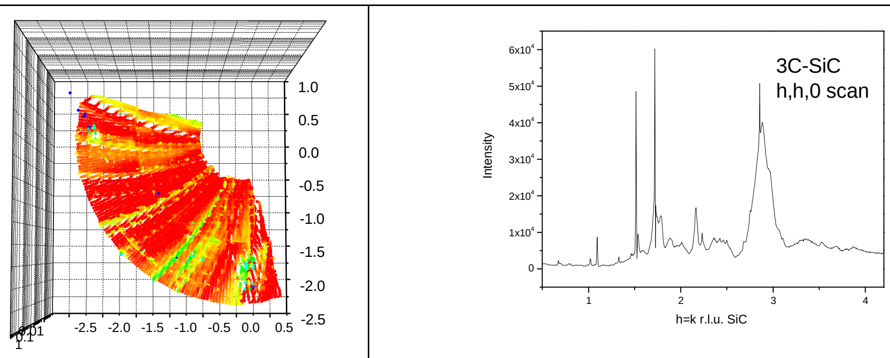

Figure 1: In plane diffraction as observed from a 3C-SiC (001) surface. (Left) In plane reciprocal space map. (right) typical (h,h,0) scan.

Sample #2

The sample second sample was of 6H-SiC(0001) type and could be successfully prepared using an in-situ annealing at 250°C for 30 minutes. For this surface a clear signal arising from the top graphene surface layer could be identified (see figure 2). The typical mosaic spread of the layer was in the 0.65° range. The (10L)(22L) and (20L) SiC crystal truncation rods were quantitatively measured as well as the (0.72, 0.72,L) (2.16,2.16,L) and (1.23,0,L) rods from the graphene layer.

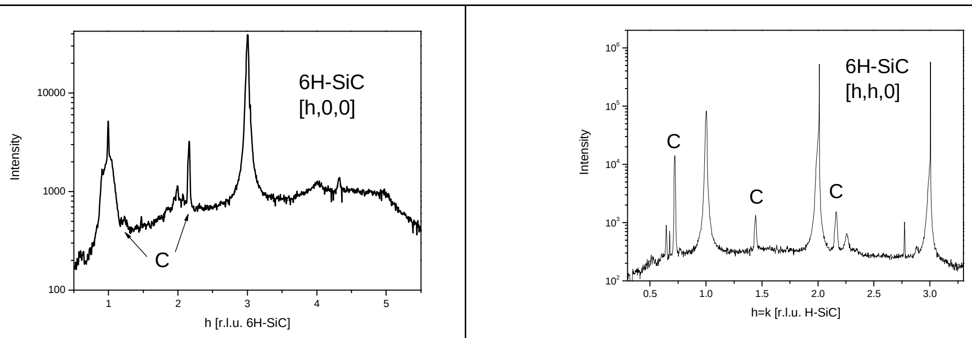

Figure 2: In plane scan through [h,0,0] and [h,h,0] direction of the reciprocal space of 6H-SiC(0001) after graphene growth.

We could also investigate the role of Au deposition. In the first case a deposition of about 5 ML of Au destroyed the graphene layer completely.

Sample #3

A third sample consisted of 4H-SiC(0001). The sample could be prepared in very good conditions and again a clear signal from the graphene layer could be observed and measured. On this sample first attempts of hydrogenation and annealing were performed successfully. The process was investigated using qualitative scans because of lack of time. The structural changes produced through hydrgenation proved to be reversible upon annealing.