Conductivity Contrast in SEM Images of Hydrogenated Graphene Grown on SiC

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Graphene consists of a planar single sheet of sp^2 -bonded carbon atoms arranged in a two-dimensional (2D) honeycomb lattice. One of methods of obtaining epitaxial graphene is growth on SiC(0001) by CVD (Chemical Vapour Deposition) [1]. In this growth mode the first carbon layer it is not a graphene one, it is attached to Si atoms of the substrate by sp^3 bonds and it is called the buffer layer. The conversion of the buffer layer into graphene may be obtained by hydrogenation process. Hydrogen molecules introduced between the buffer layer and the SiC substrate, break most of the sp^3 Si – C bonds and the buffer layer converts into graphene lattice [2].

Graphene grown by CVD technique on 4H-SiC(0001) substrates and hydrogenated at a high temperatures were subjected to SEM investigation with low energy (~0.2 keV) electrons using Auriga CrossBeam Workstation (Carl Zeiss) equipped with In-lens SE (true SE1) detector and Energy selective Backscattered electrons (EsB, low-loss BSE) detector, both positioned on the optical axis of the Gemini (TM) column. The main aim of the work was to determine the contrast origin of the specified areas of the graphene intercalated by H₂. The results obtained by low-kV SEM were supported by LC-AFM (Local Conductivity AFM) technique. The presence of graphene has been confirmed by Raman spectroscopy measurements.

Figure 1 presents the SEM images obtained in the In-Lens (a) and EsB (b) detectors in parallel. Both images show characteristic dark parts in the middle of the terraces, surrounded by brighter zones. The bright lines visible in the images are thermally driven cracks in the graphene (due to the high temperature during hydrogenation process) uncovering the SiC substrate. Assuming that the contrast in the SE image (In-lens) is mostly originating from the conductivity differences, and the image of low-loss BSEs is based on the compositional contrast, the reason of the unquestionable contrast of carbon layers may be explained as follows.

It has been shown that the graphene buffer layer grown of SiC(0001) is not an uniform one and the area close to step edges are well saturated with carbon atoms [3]. On the other hand, terraces are not so well saturated with carbon atoms and characterize with a larger concentration of defects [3]. The hydrogenation of well saturated regions, close to step edges, will result in breaking Si – C sp³ bonds between buffer layer and substrate, and formation of Si – H bonds and the p-type conductive graphene layer. However, the defected graphene terrace regions, in addition to formation of Si – H bonds underneath of graphene layer, may have defects of a donor character, which will compensate p-type conductive the by the Si – H bonds. Therefore, the central regions of terraces may have less conductive character and to be depleted of charge carriers. These subtle phase differences are clearly visible in the EsB images obtained in a very low energy regime, where the yield of low-loss BSEs is strongly influenced by the nature of molecular bonds, thus the presence of defects in graphene may create differences in scattering coefficient. The results have been confirmed by AFM measurements in

LC-AFM mode. Figure 2b presents AFM image showing local conductivity map of the same sample, with a high-conductivity regions of graphene along step edges and a low-conductivity on terraces.

References:

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Figure 1. SEM images of CVD-grown graphene intercalated with H₂: a) SE image, b) low-loss BSE image.



Figure 2. AFM and LC-AFM images: a) topography b) map of local conductivity (dark regions are connected with a low and the bright ones with a high conductivity).