Morphology and Structure Analysis of Graphene by Low Voltage TEM

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Graphene, a single atomic monolayer of $sp^2$-bonded hexagonal carbon with extraordinary mechanical, electronic, and optical properties, has become a subject of great interest in materials science. Since its first isolation in 2004, graphene has been researched by many research groups in the fields of physics, chemistry, and material. And it is expected that graphene will be applied in various industry [1]. To analyze properties of graphene, optical and electrical analyses are well used [2, 3]. For observation of graphene morphology, electron microscopy, atomic force microscopy, and scanning tunneling microscopy, etc. have been used. But it is very difficult to observe graphene because of its ultra-thin thickness.

Transmission electron microscopy (TEM) is a microscopy technique in which a beam of electrons is transmitted through an ultra-thin specimen, interacting with the specimen as it passes through. Therefore thinness of graphene and the low atomic number of carbon make graphene almost transparent to the electron beam. And the slight beam interaction with the hexagonal carbon monolayer generates a well-defined signal that can be easily subtracted from resulting images and diffraction patterns. Therefore TEM technique is getting a lot of attention as an attractive method to analyze both morphology and structural properties of graphene. However, the damages by electron beam energy make it difficult to observe graphene because conventional TEM is operated at an accelerating voltage up to 200 kV. For successful TEM analysis of graphene, it needs to observe at the low acceleration voltage under 100 kV which can minimize the damages [4].

In this study, we analyzed graphene using low voltage TEM at 80 kV. The graphene specimens were prepared by a direct transfer method. The graphene films were grown on Cu foils by chemical vapor deposition. Then holey TEM grid was placed on top of graphene on the Cu foil and isopropanol was dropped to bond the graphene film and TEM grid. After baking at 120°C, etching away the Cu foil was achieved by floating the sample on a solution of ammonium persulfate. The graphene specimens were observed by using a JEM-ARM200F microscope operated at 80 kV. To analyze structural properties such as grain boundary and orientation, dark-field TEM (DF-TEM), high-resolution TEM and electron diffraction analyses were performed.

Figure 1(a) shows diffraction pattern taken from a region which reveals that the graphene layers have two different orientations. Figures 1(b)-(c) show DF-TEM images obtained each diffraction spots in (a) and
(d) shows color-coding them produces. It is indicated that each diffraction spots contains each grains and confirmation of grain boundary is possible by DF-TEM measurement. Figures 2 (a)-(c) show a bright field TEM image, the corresponding diffraction pattern and its intensity profile, respectively. The intensity ratio shows that the graphene is single layer [5]. Also we performed diffraction simulation for the graphene structure and the simulation results matched experimental ones.

These results demonstrate that low voltage TEM technique is useful method to analyze the morphology and structure of graphene.

References:

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Figure 1. (a) Electron diffraction pattern of graphene. (b) and (c) Corresponding dark-field images. (d) False-color dark-field image overlaid with emanating dark-field images from color-coding diffraction beam in (a).

Figure 2. (a) TEM image of graphene. (b) Corresponding electron diffraction pattern of (a), (c) Intensity profile of -12-10 to 2-110 for the diffraction patterns indicated in (b).