Correlative Characterization of Graphene with the Linkage of SEM and KFM.

Y. Hashimoto¹, T. Yamaoka², S. Takeuchi¹, T. Sunaoshi¹, A. Miyaki¹, M. Sasajima³, A. Muto⁴, J. Yu⁴

¹. Application Development Dept., Hitachi High-Technologies Corporation, Hitachinaka, Japan
². Analytical Application Engineering Section Tokyo2, Hitachi High-Tech Science Corporation, Kawasaki, Japan
³. Electron Microscope Systems Design 1st Dept., Hitachi High-Technologies Corporation, Hitachinaka, Japan
⁴. Nanotechnology Systems Div., Hitachi High Technologies America, Inc., Clarksburg, USA

Graphene has been one of the most attractive and promising materials because of its unique material properties and potential applications. Its properties depend strongly on the number of layers, so the reliable examination method to determine its thickness has been required [1]. ULV (Ultra low voltage) SEM (scanning electron microscopy) is one of the possible methods for determining its thickness by clarifying layer-sensitive images of graphene. To explore further into the mechanism of the SEM contrast on graphene images, it has been explained that the difference of thickness causes the difference of surface potential that can affect the SE (secondary electron) signal intensity [2]. It has also reported that the effect of the thickness of graphene layers on its surface potential was detected by AFM-based technique KFM (Kelvin force microscopy) [3]. In this study, we developed the linkage system of SEM and AFM with compatible sample holder with correlation technology between SEM, AFM, and KFM image at the same area of interest to reveal the relationship between SEM contrast, its height, and surface potential.

Figure 1 shows the general view of Hitachi SU8230 FE-SEM and AFM5500M SPM. The beam deceleration function which achieves ULV imaging with high resolution capability was installed in the SU8230. Figure 2 shows the schematics of the linkage system of SEM and SPM. To get the linkage of SEM and SPM, three alignment positions need to be registered. Once this procedure is done, the stage can be moved to desired areas by “one-click” in both of SEM and AFM. Graphene on SiO₂ substrate which was grown by CVD (Chemical Vapor Deposition) technique was observed by SEM and SPM with the linkage system. The SEM image was taken at the landing voltage of 0.5 kV, and the AFM / KFM images were taken with the Rh coated conductive cantilever.

Figure 3 shows the correlative images of SEM, AFM/SEM overlay, and KFM/SEM overlay on graphene. The SEM image shows a darker patch structure which has 2 levels of darkness and also some darker line structures. The AFM image indicates that the difference of height between the patch structure and surrounding area was about 0.3 nm which is almost same as one layer thickness of graphene. KFM image indicates that there is up to 40 mV difference of surface potential between the patch structure and surrounding area. From these results, the authors are considering that the difference of surface potential which is caused by the difference of its thickness might provide the SEM contrasts. There is still room for discussion regarding the line structures because their surface potential were weaker than the patch structure while their SEM contrast were higher. We will disclose more details including SEM imaging at the landing voltages less than 0.1 kV in the session.
References:

Acknowledgement:
The authors wish to acknowledge Prof. David C Joy and Dr. Vighter Iberi of Oak Ridge National Laboratory, Dr. Walter E. Henderson, Dr. Eric Woods and Dr. Todd Walters of Georgia institute of Technology for their helpful discussion and sample preparation.

Figure 1. The general view of Hitachi (a)SU8230 FE-SEM, (b)AFM5500M SPM.

Figure 2. The schematic of the linkage system of SEM and SPM.

Figure 3. The correlative images of (a)SEM, (b)AFM/SEM overlay, (c)KFM/SEM overlay on graphene.