Imaging and spectroscopy of graphene oxide at atomic resolution

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Graphene oxide is a form of graphene that is modified by the addition of functional groups such as carboxylic groups, ketones and hydroxyl groups to its surface. The structure and distribution of the functional groups, which affect its chemical, electrical and mechanical properties depends on the synthesis method used. Of particular interest is the chemical composition and spatial distribution of these functional groups. Direct identification of the groups by high-resolution imaging is not possible at present owing to their similar composition to the graphene oxide substrate and thus low contrast, instead they can be made visible by bonding heavy atoms, such as Ba, to selected groups and imaging the distribution of the heavy atoms. High-resolution images of Ba-doped graphene oxide taken at 80kV in a Cs and Cc corrected transmission electron microscope (TEM) show the structure of the graphene oxide at atomic resolution with minimal electron beam damage. However, the atomic species cannot be identified directly from such images. High-resolution scanning transmission electron microscopy (STEM) at room temperature proved to be impossible due to contamination from migration of the functional groups on the graphene oxide surface. We were able to overcome contamination by imaging the graphene oxide above 400°C in a stable heating holder. Simultaneous STEM energy-loss spectrum images and high-angle annular dark-field images recorded at high temperature then allowed us to correlate the locations of the Ba atoms with features in the high-angle dark-field images at atomic resolution. Results will be presented showing how we were able to use STEM dark-field and bright-field images to determine the positions of the Ba atoms in high-resolution TEM images.