JEOL FE-SEM – Innovative Design

Imaging and Analyzing Graphene Layers

SMART – POWERFUL – FLEXIBLE

Graphene is a crystalline form of carbon defined as a hexagonal arrangement of carbon atoms in a one-atom thick planar sheet. Graphene has outstanding properties (mainly mechanical strength, optical transparency and excellent electrical and heat conductivity) that make it an attractive material for electronics applications. Traditionally, graphene structures have been imaged with aberration-corrected TEM, AFM, or STM.

Graphene layers, especially if deposited on a solid bulk substrate, can also be imaged and analyzed using low kV FE-SEM. Fig. 1 shows a FE-SEM image of graphene oxide flakes taken at 50V. The images were taken with a through-the-lens detector, which is highly sensitive to surface specific information at low kVs (especially while imaging in conjunction with specimen bias), where the graphene layers are limited to few atomic layers on the specimen surface.

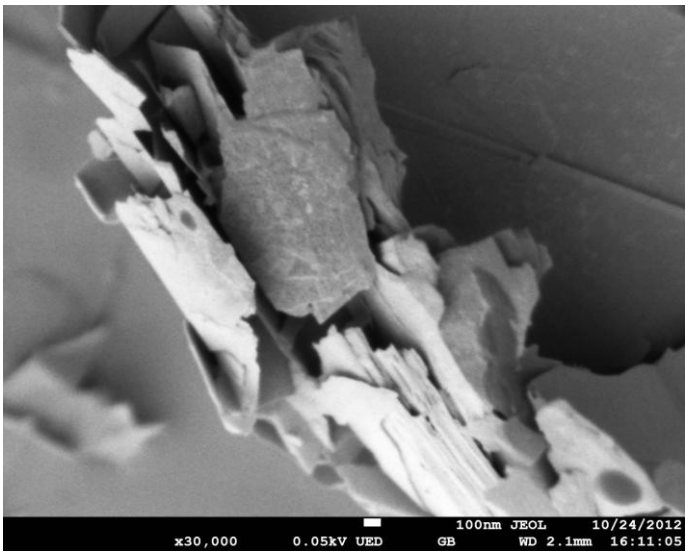

Fig. 1 Graphene oxide flakes imaged at 50V with through-the-lens detector.

The use of SEM also facilitates imaging and EDS analysis of graphene layers with respect to the substrate; thus, providing a more direct analysis of graphene in its application-based environment as opposed to TEM where only individual suspended sheets of graphene can be imaged. Fig. 2 shows an example of EDS maps of graphene layers on a Ni substrate, obtained at 1kV and 5kV. By performing EDS analysis at low accelerating voltage, the beam/specimen interaction volume is significantly reduced, so that discrete maps of graphene and Ni components can be resolved. In order to minimize any significant buildup of carbon contamination on the sample surface that could obscure characterization, the mapping time

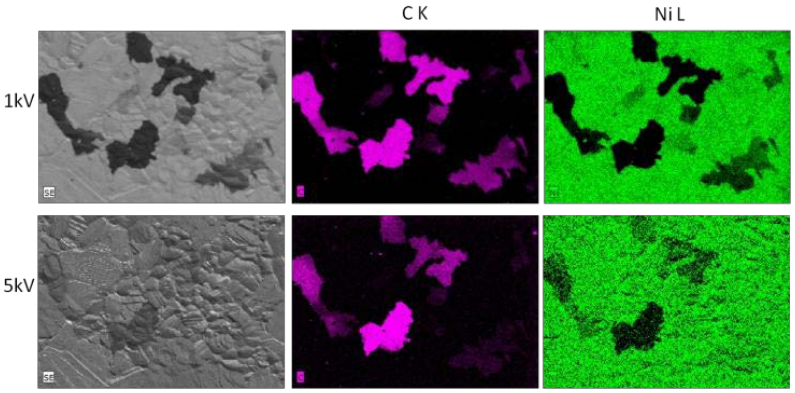

Fig. 2 Ultra-low kV EDS mapping of graphene film on Ni substrate. Top set of data acquired at 1kV using a probe current of 32nA; bottom set acquired at 5kV using a probe current of 4nA.

was less than 5 min. The high beam current setting, combined with large area detector (100 mm^2) used for this analysis, ensured adequate data collection for compositional interpretation even with a very short acquisition time at low kV.

Given its excellent properties, graphene can also be incorporated into composite materials as an additive to improve electrical, thermal and mechanical properties and improved resistance to moisture adsorption. An example of such a composite material can be achieved via incorporation of graphite into PETI (phenylethynyl terminated imide) resin, which results in delamination of graphite in to individual graphene sheets. Such nano-composites have the potential of being utilized for use in structural components of advanced high-speed aircraft, weapon systems, and space vehicles. It is important to understand the overall distribution, size of the individual clusters and the overall morphology of the graphite/graphene in the polymer matrix. Fig. 3 shows an example of the graphite/graphene dispersion in PETI298. The image clearly shows the delamination of graphite into individual graphene sheets.

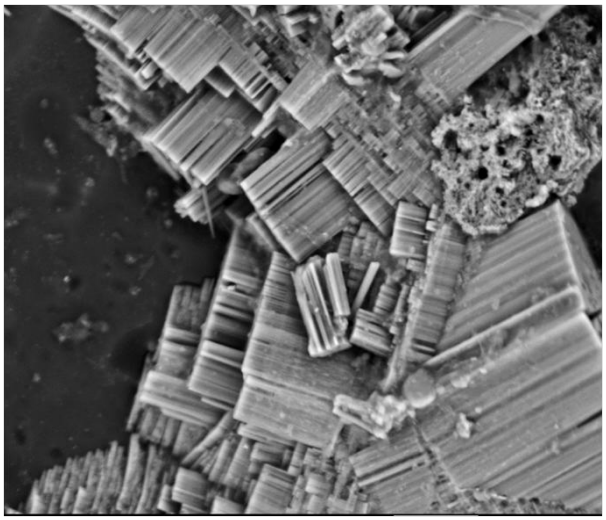

Fig. 3 BSE image of graphite/graphene dispersion in PETI298 acquired at 3kV (sample courtesy of Prof. D. Veazie, Southern Polytechnic State University).

The current lineup of JEOL FE-SEMs offers a cutting edge Through-The-Lens (TTL) electron column design that minimizes effects of chromatic and spherical aberrations on the ultimate probe size and resolution. This novel electron column design also features a TTL detector with an energy filter. This combined with precise control of the landing energy of the primary electron beam (beam deceleration via Gentle Beam function), has created new opportunities for specimen observation. In particular, these new design improvements have advanced significantly the ability to image nanomaterials with previously unattainable nanometer scale resolution (7Å) at landing voltages as low as 10V.