Scanning Electron Microscopy Analysis at Low Voltage in Carbon Nanotubes Doped with Heteroatoms

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Carbon nanostructures have been developed as supporting material of active metals, and recently as doped carbonaceous materials using heteroatoms creating metal-free electrocatalysts for fuel cells, batteries and sensors. However, some features remaining as unknown need to be assessed regarding their effects in the electrochemical devices. On the other hand, recent development of new detectors for electron microscopes have allowed to better characterize the properties of new nanostructures, especially of graphene and doped carbon nanotubes. In this work, the high resolution scanning electron microscopy (HRSEM) study on carbon nanotubes doped with Si, S and N (CNT-H) is investigated and correlated with other characterization techniques. The CNT-H were synthesized using a modified chemical vapor deposition method using ferrocene, toluene and different organic reactants to dope them with sulfur, nitrogen and silicon, respectively. Pristine carbon nanotubes were also synthesized for comparison. CNT-H were evaluated by high resolution scanning electron microscopy using not only the conventional secondary and backscattering electrons detectors but also a new electron energy loss spectroscopy (EELS) detector. Chemical microanalysis is performed using an X-ray energy dispersive spectroscopy (EDS) annular detector permitting to achieve high X-ray counts rates even at low primary energy. Transmission electron microscopy and X-ray photoelectron spectroscopy (XPS) were also used to correlate the results with HRSEM. Electron microscopy results show that morphologies were influenced by the dopants. CNT-H with sulfur show a horn-like nanostructure with two different types of layers, the outer layer being more amorphous than the inner one. Meanwhile, CNT-H with nitrogen show the typical bamboo-like structure. However, micrographs of CNT-H with silicon show the same morphology that the pristine carbon nanotube. EDS elemental mapping results show the presence of the doping elements along the nanostructures which is in agreement with the chemical states detected by XPS.

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