Controlled synthesis and properties at the nano-scale of highly reduced graphene oxide (HRGO) obtained by Langmuir-Blodgett method.


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We present here the controlled obtaining of highly reduced graphene oxide (HRGO) thin films by using of Langmuir-Blodgett (LB) technique. To have a deep and definitive information about the chemical species in the samples and the spatial distribution of them, a multiple characterization on the different stages of the material (i.e. before and after each reduction treatment) was performed employing both spectroscopic and image-based analysis ones such as: SEM, AFM, grazing XRD, RAMAN, XPS and their space-solved versions: micro-RAMAN and nano-XPS. Finally, in order to correlate the chemical state, structure and defects present in the HRGO thin films, a set of current-voltage curves on macroscopic and microscopic distance between electrodes.

Graphene oxide was obtained by Hummers’ method [1] and deposited on Si(100) by LB technique. Similar samples could be obtained just by simply setting of the same parameters at the LB setup. Different thermal reduction treatments were performed subsequently at different accumulative steps at 300, 600 and 700 °C under UHV conditions. After each thermal treatment samples were characterized. Grazing XRD experiments were performed at the DRX2 beamline at the LNLS Synchrotron Laboratory (Campinas, Brazil), XPS and nano-XPS analysis were performed at the ANTARES beamline [2] at the SOLEIL Synchrotron Laboratory (Saint Aubin, France). The microscopic determinations of current-voltage curves were achieve using the assistance of a nanomanipulator and a probe station.

The thickness of the samples was analyzed by AFM and grazing XRD. It results thinner after thermal treatments, reaching between 1.3 and 1.8 nm after reduction treatments at 600 °C, which is very close to the expected value for very few layers HRGO [3]. After thermal treatments we also observe, from RAMAN experiments, the decrement of D band, associated with defective centers. Similar information was obtained by XPS, were different O-species were eliminated after each thermal treatment. From micro-RAMAN and nano-XPS we can establish the high spatial homogeneity of the thin-film after reduction treatments. In summary we can show a simple procedure to obtain, with high reproducibility, almost monolayers of HRGO with extreme chemical and structural homogeneity and conductive properties.
Figure: a) SEM image after thermal treatment at 600 ºC; b) high resolution XPS spectra of the C 1s peak of GO samples after different thermal treatments for reduction.

References