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Surface preparation of carbonaceous films to increase wettability and integration with nanoparticles for electrochemical applications

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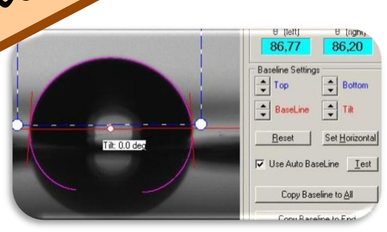
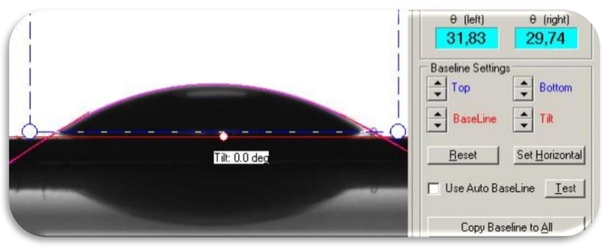
Abstract

The two most significant techniques for producing carbonaceous materials with exceptional conductivity for use in electrochemical devices are chemical vapor deposition and mechanical cleavage of graphite. The main advantages of using the CVD technique is represented by the ability to obtain materials that can be used on larger surfaces, with a uniformity superior to cleavage techniques. However, films of carbonaceous materials are hydrophobic, which makes it difficult to integrate nanoparticles. By adding nanoparticles (e.g., metallic, oxide), the electrochemical characteristics of carbonaceous materials can be improved, as a result of the increase in the specific surface of the electrode, catalytic effect, and enhanced electron transfer. In this paper, we present the methodology for modifying the wetting capacity of a graphene film grown on a copper substrate, which we transferred through a chemical process to the SiO2/Si substrate. To modify the wetting capacity of these materials, both plasma treatment using reactive ion etching equipment, and a chemical treatment in acid medium were carried out. For fundamental investigations of the graphene liquid interface, it is necessary to ensure that the graphene surface is free of any kind of residue. This is important not only to ensure the persistence of favorable properties, but also to ensure an ideal sp^2 carbon surface to allow suitable chemical interactions with NPs, precursors, and analytical molecules. The research has focused on the use of spectroscopy, SEM, and goniometry as techniques for structural, morphological, wetting capacity, and percolation analyses of carbonaceous materials. Their applicability in the electrochemical field was studied by cyclic voltammetry after the incorporation of nanoparticles.

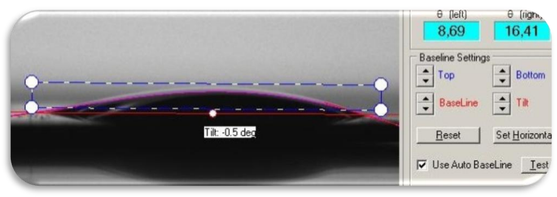
Results



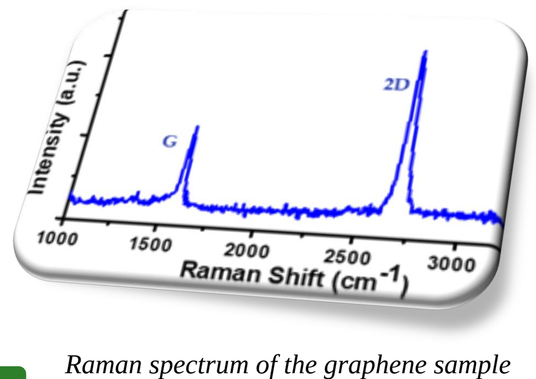
Water droplet distribution on the surface of the graphene sample



Distribution of water droplet on the surface of the graphene sample after treatment in acid medium

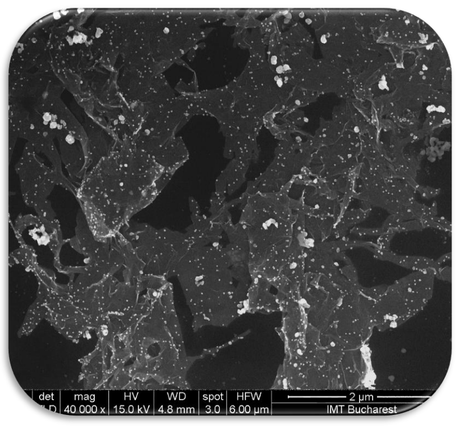


Water droplet distribution on the surface of the graphene sample after plasma treatment

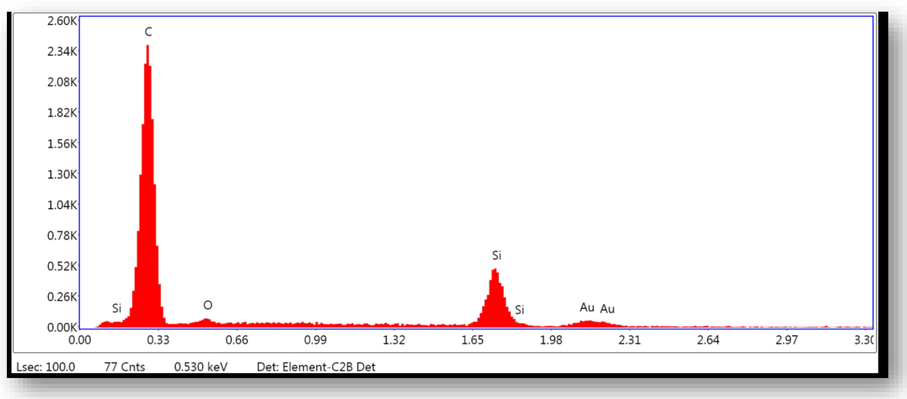


Raman spectrum of the graphene sample after plasma treatment

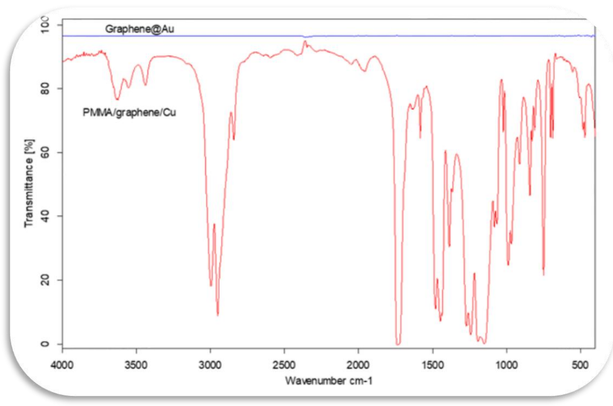
- Measurements performed before and after treatment in plasma and acidic medium (H_2SO_4-HNO_3) show a sharp decrease in the contact angle, suggesting a hydrophilic character exists.
- The Raman spectrum is characterized by G and 2D peaks confirming the graphitic nature of the film.



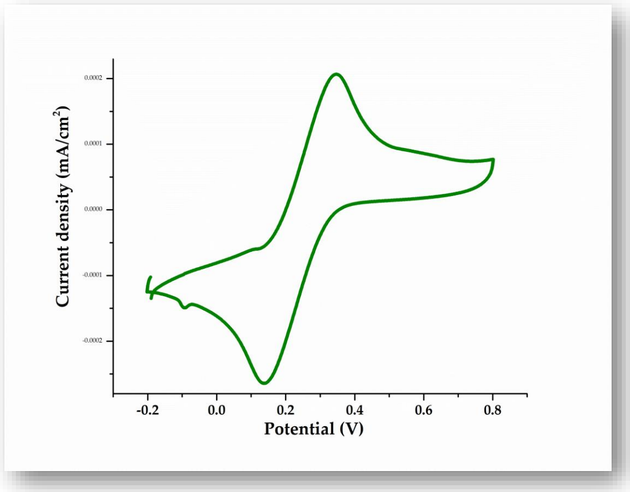
SEM images for a representative sample of monolayer graphene decorated with gold nanoparticles



EDX spectrum for a representative sample of graphene decorated with gold nanoparticles



FTIR spectra for the graphene sample before transfer onto the SiO_2 substrate, after modification of wetting capacity and decorated with gold nanoparticles



The CV of curve one cycles was taken from −300 mV to 900 mV vs. Ag/AgCl with a scan rate of 100 mV/s of graphene@Au sample in 2.5 mM $[Fe(CN)_6]^{3-/4-}$ in PBS (pH 7.4) electrolyte.

- Before transfer, the FTIR spectrum for the graphene sample is characterized by absorption bands that can be attributed to the PMMA polymer used as a sacrificial layer in the graphene transfer.
- For the modified carbon material sample on which Au NPs were anchored, only low intensity absorption bands are observed, which can be associated with the adsorption of water and carbon dioxide molecules from the air. Graphene or Au NPs do not show any absorption bands in the IR.
- From the SEM image, an average size of the Au particles of about 20 nm was estimated, without affecting the initial morphology of the carbon materials.
- EDX spectroscopy confirms the presence of Au atoms on graphene transferred onto SiO2/Si substrate and modified.
- Well-defined redox peaks showed an electro-catalytic activity of graphene films decorated with gold nanoparticles.

Acknowledgements

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