

Free-standing graphene: synthesis and functionalization using plasma-based methods

A. Dias^{1,2}, J. Berndt², E. Kovacevic², C. Pattyn², T. Strunskus³, J. Henriques¹, E. Tatarova¹

¹ Instituto de Plasmas e Fusão Nuclear, Instituto Superior Técnico, Universidade de Lisboa, Lisboa, Portugal

² GREMI UMR 7344, CNRS & Université d'Orléans, Orléans, France

³ Institute for Materials Science - Multicomponent Materials, Christian-Albrechts-Universität zu Kiel, Germany

In the present work, an atmospheric pressure microwave plasma-based method is used to synthesize free-standing graphene sheets (FSGs). The FSGs were then transferred to several types of substrates using different graphene suspensions. The results allowed to demonstrate the possibility to use graphene in future flexible nanodevices. Subsequently, the deposited graphene sheets were successfully N-functionalized and polymerized with aniline (ANI) using a capacitively coupled plasma at low pressure. The samples were characterized by transmission and scanning electron microscopy (TEM and SEM), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS), near edge X-ray absorption fine structure spectroscopy (NEXAFS) and by contact angle technique.

1. Introduction

A critical requirement for the mass production of graphene is the control of the synthesis processes. So far, conventional methods used for the synthesis of these 2D materials present several drawbacks most importantly the quite limited control on the assembly process. Moreover, N₂ functionalization of graphene is one of the key topics in materials research, since functionalized graphene finds extensive application in polymer science and technology due to its extraordinary electrochemical properties (eg. polymer-graphene nanocomposites).

Therefore, the aim of this work is to find a simple method for the subsequent deposition and treatment of free-standing graphene sheets.

2. Experimental

At first an atmospheric microwave plasma was used to synthesize FSGs [1-5]. To this end, a hydrocarbon precursor was injected into the microwave plasma environment, where decomposition processes take place. The main part of the solid carbon is gradually dragged into the outlet plasma stream, where the graphene sheets assemble and grow. A power of 2 kW was applied, injecting Ar as background gas and ethanol as precursor with a ratio of 10:1. The collected FSGs (see Fig.1) are deposited using a simple dispersion method, which enable the deposition on various substrates. Distilled water and methanol were used as solvents in this process. Subsequently, N₂ plasma treatment [6] was performed to turn graphene into a hydrophilic surface, enabling for example the adhesion of biomolecules. The N₂ plasma treatment consists on placing the samples in a remote zone of a capacitively coupled plasma (CCP) for different processing times. A RF power of 8 W was applied at

13.56 MHz, while maintaining 0.1 mbar pressure in the chamber.

The same experimental set up was also used for the deposition of thin films onto the graphene flakes. These experiments were performed with aniline as a precursor for the thin film synthesis.

The resulting nanostructures were characterized by SEM and TEM, Raman spectroscopy, XPS, NEXAFS and contact angle measurements.

Plasma characterization was also performed by mass spectrometry and optical emission spectroscopy.

References

- [1] E. Tatarova, A. Dias et al. Nova Science Publishers, ISBN: 978-1-63485-214-2 (2016).
- [2] E. Tatarova, N. Bundaleska et al. Plasma Sources Sci. Technol. 23, 063002 (2014).
- [3] E. Tatarova, J. Henriques, C.C. Luhrs, A. Dias, et al. Phys. Lett. 103, 134101 (2013).
- [4] E. Tatarova, A. Dias et al. J. Phys. D: Appl. Phys. 47, 385501 (2014).
- [5] A. Dias, N. Bundaleski et al. J. Henriques J. Phys. D: Appl. Phys. 49, 055307 (2016).
- [6] E. Kovacević, J. Berndt et al. J. Appl. Phys. 105, 104910 (2009).

Acknowledgements

Work partially funded by Portuguese FCT - Fundação para a Ciência e a Tecnologia, under project UID/FIS/50010/2013 and grant SFRH/BD/52413/2013 (PD-F APPLAuSE) and by French Regional Research Agency through the project APR Capt'Eau and ARD PIVOT.

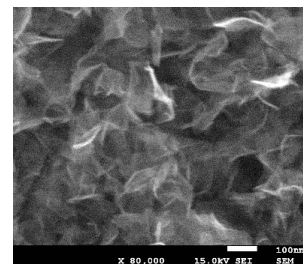


Fig 1 – SEM image of FSGs.