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Graphene based chemical sensor fabrication by means of Focused Ion Beam

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Abstract

Graphene is a material with surprising properties, that very recently aroused the attention of the scientific world for its potential applications in different fields from electronics to sensors. To date, many works have been already published dealt with graphene-based sensor in which the preparation of the devices involves the use of some lithographic method. It is known, however, that typical nano-lithographic processes leave an uncontrolled residue on graphene. In this work we present a graphene based chemiresistor, where the electrode structure is realized by Ion Beam Induced Deposition technique; the current-voltage characteristics of such a device will be investigated in different environments.

Keywords: graphene; gas sensors; focused ion beam

1. Introduction

Graphene, a single atomic layer of graphite sheet, has aroused a considerable interest after the achievement of its synthesis and the experimental observation of Dirac charge carrier properties in graphene-based devices [1]. Comprehensive treatments of graphene properties can be found in recent reviews [2-7].

It has been reported that, similar to other solid-state sensors, the absorption onto the surface of a graphene sensor of individual gas molecules, acting as donors or acceptors, leads to a detectable change in its electrical resistance [8]. In addition, graphene is an exceptional low-noise material and the signal-to-noise ratio can be optmized to a level sufficient for detecting changes in a local concentration by less than one electron charge at room temperature, making the graphene an excellent candidate for chemical detectors [9].

The realization of a conductometric device, however, frequently requires a series of nano-lithographic steps that are known to leave an uncontrolled residue on graphene [10] whose impact on device transport and vapor sensing properties has not been fully explored, whereas it has been ascertained that the key for producing sensors with high selectivity is the use of clean graphene. One of the most promising technique that can overcome the obvious

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complexity related to nanodevice fabrication, is Focused Ion Beam (FIB) that represents an already well established technique in microelectronics and microfabrication for maskless patterning, that allows the deposition of nanoelectrodes in precise and controlled sites.

Here we report on the preparation of graphene-based devices where FIB is used for nanoelectrode patterning, realizing platinum electrical contacts. The current-voltage characteristics of such a device will be investigated in different environments.

2. Experimental

Graphene was prepared starting from commercially available HOPG of grades ZYB (www.ntmdt.ru), by means of the 'scotch tape' exfoliation method and transferred onto Si substrates where 300nm of SiO_2 were previously evaporated by e-beam deposition. To select only the few layers graphene flakes, we used a combination of optical and atomic force microscopy. A validation of the graphene structure of the material (single-, bi- and few layers) was made through Raman spectroscopy.

The samples were then placed into the vacuum chamber of Dual Beam apparatus (FEI QUANTA 200 3D). The system integrates a high resolution FIB obtained from a Ga+ liquid metal source and a Scanning Electron Microscopy (SEM) with tungsten filament electron source. Once graphene fragments were located by SEM, to complete the chemiresistor architecture, platinum nanoelectrodes (500 μm long, 200nm wide, 300 nm thick) were deposited on it by means of FIB using 30 kV as accelerating voltage and 10 pA as emission current.

Measurements in controlled environment were performed using a Gas Sensors Characterization System (GSCS) better described elsewhere [11].

3. Results and discussion

In fig. 1 a) and b) the optical micrograph and the AFM image of the same few layers flake are reported.

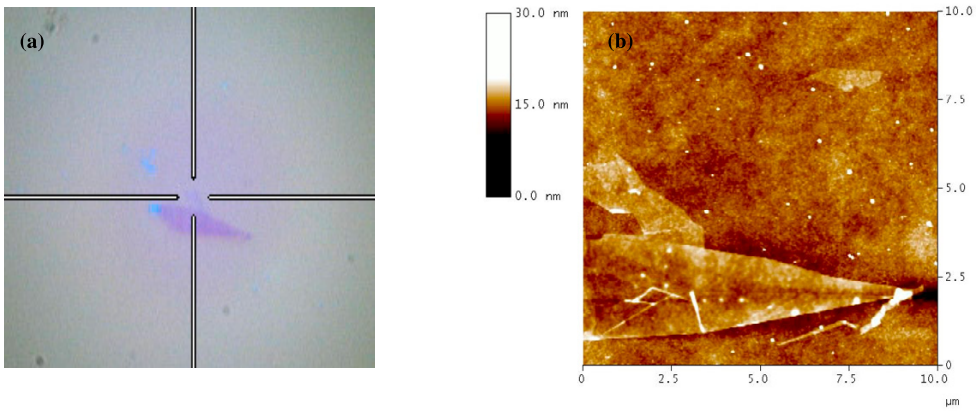


Fig. 1: (a) optical micrograph of a few layers graphene flake; the area highlighted in the center of the cross is a single layer flake: (b) AFM image of the same flake.

In the optical image two zones with different shades of color (light and dark violet) are clearly visible, attributable to different thickness layers. The AFM analysis on both the zones shows that the flake is not completely flat, and some areas are cracked and folded back. However the thickness of the clear zone varies between 10 and 14

Å whereas the dark one has a thickness mean value of ~30 Å. It has been reported that the single layer thickness is usually up to ~10 Å, rather different compared to the theoretical value reported in the literature for the single layer (4 Å). This can be attributed to the ever–present "dead layer", of absorbed water, which remains captured between graphene and SiO_2 [1]. From the thickness of the "dead layer", we can conclude that the clearer zone contain 1 or 2 layers while the dark area is made of 5 layers maximum of graphene.

This is further confimed by the Raman analysis (see fig. 2) which illustrates the spectra acquired on both the zones. The solid curve which refers to the thinner part of the flake is a typical Raman profile of single graphene layer with a sharp 2D peak (~2700 cm^{-1}) markedly more intense than the G peak (~1580 cm^{-1}). The Raman spectrum of the thicker part of the flake is reported as dotted line: the 2D band is typical of the graphene multilayer with less

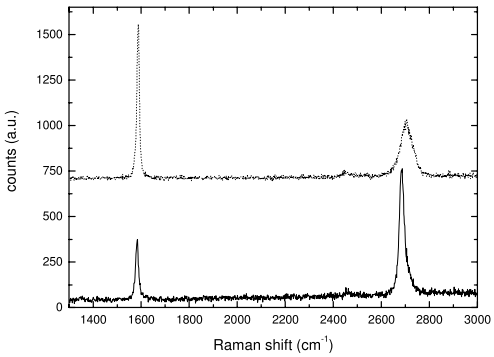


Fig. 2: Raman spectra of a few layers graphene samples

than 5 layers [12].

Platinum nanoelectrodes, shown in fig.3, are deposited onto the few layers graphene flakes with the FIB equipment using 30 kV as accelerating voltage and 10 pA as emission current.

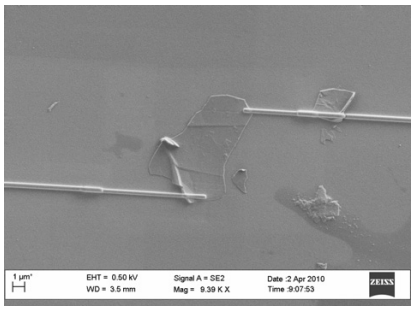
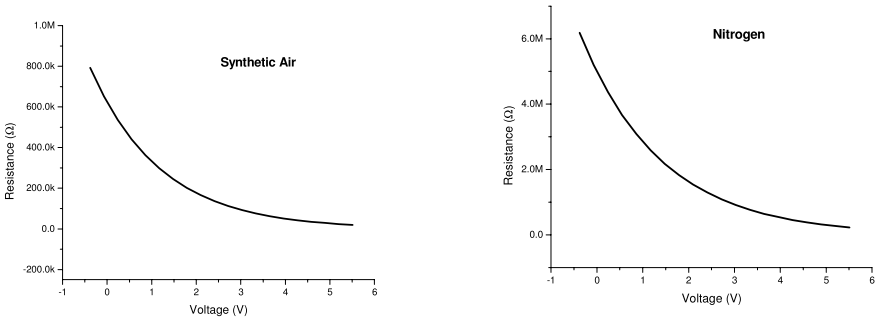


Fig. 3: A typical SEM image of a graphene sample mounted onto the FIB stage; the electrodes are realized in Pt by means of Ion Beam Induced Deposition technique.

So prepared devices have been electrically characterized. The preliminary tests, carried out in ambient air, have highlighted a great variability of the electrical performances. Hence, further characterizations have been

accomplished in controlled environment. In fig. 4 (a) and (b) are reported some preliminary results of the graphene based chemiresistors in nitrogen and synthetic air.



(a) (b)

Fig. 4: Curve fitting of the experimental data of the resistance vs. applied voltage on graphene based chemiresistor in: (a) synthetic air, (b) nitrogen

The devices are biased at DC voltage; each voltage value is applied for 1 min. The resistance is recorded every 10 seconds. The voltage sequence begins from 0.1V to 5V and return to 0.1V. As shown in the figures the O_2 contained in the synthetic air reduces the device resistance, confirming that the graphene based devices are extremely sensitive to the gases composition. These results are very promising for further investigation on other analytes.

Acknowledgements

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