RECONSTRUCTION OF GRAPHENE LAYERS UNDER ELECTRIC CURRENT

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Abstract: Due to its different hybridization, carbon atoms are very important both for technological application and for research aspects presenting great perspectives for nanotechnology products. In the last few years different carbon structures have been discovered including: Fulerens, nanotubes, nanorribons and graphene single layer. For that reason, the Materials Metrology Division of the National Institute of Metrology of Brazil is making efforts in the study and development of carbon reference materials also including basic research in new carbon materials. The main purpose of this work is to study a recent discovered carbon nanorribon where we are building suspended devices I order to understand the reconstruction of graphene layer.

Key words: graphene, dual beam, carbon nanoribbon, Raman spectroscopy.

1. INTRODUCTION

The quest for perfect graphene edges have kept many scientists captivated since its firsts observations. Based on the results of Joule heating of graphitic nanoribbons inside a TEM, where sharp zigzag and armchair edges were visualized [1], we were motivated to construct a device to induce Joule heating of nanoribbons in a configuration such that the sample could be studied outside the electron transmission microscope. Such device could allow the metrology of the material pre- and post- experiment. In this work we will expose our different approaches to build such device and the results of the electric transport experiments will be presented.

2. MOTIVATION

CVD grown graphitic nanoribbons consist of many graphene layers stacked in AB configuration, their typical dimensions range from 50 to 300 nm in width, 20 nm in thickness and tens of micrometers in length. Joule heating experiments were conducted on this sample and healing of defects and sharp edge transformations were observed during electric current transport (see Fig. 1).

We searched to reproduce these results in such a configuration that the Joule heated sample could be recovered and characterized. We proposed to mount a carbon nanoribbon making contact on both ends of a conductive micro-wire placed itself on the void space of a TEM grid. Ideally this TEM grid would be insulator so that current would only pass through the wire. This could allow TEM and SEM observation of the material pre- and post-experiment.

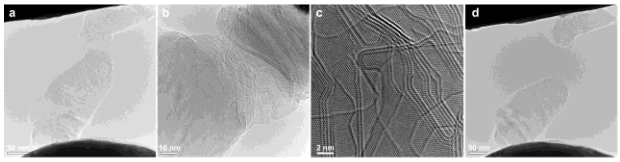

Fig. 1. Joule heating experiment inside a transmission electron microscope. a) Sample before passing current, b) sample after ~15 min of transport, c) detail on the structure of b), d) end of the experiment when the nanoribbon fractured from the middle

3. METHODOLOGY

To build and test our devise the experiment is divided in five steps. First using conventional transmission electron microscopy sample preparation we proceed to the fabrication of insulating grids using 3 mm (ϕ) discs. After opening a small hole in the disk, a 30 µm (ϕ) gold wire is placed and attached to the glass disk using a silver ink. In the third step a transversal cut on the wire using the FIB is performed. Using an Omniprobe nanomanipulator and Pt deposition to make contacts, the nanoribbons is placed across the wire bridging both ends. Finally, a bias voltage is applied in the gold wire and current through the device is measured.

4. RESULTS AND DISCUTIONS

We fabricated grids from several insulator materials. We first tried depositing silica layers on top of Cu grids but the silica easily broke and lost contact with the grid. We also tried cork, quartz and glass, our results showed that glass grids were the less fragile. We manually placed a gold wire through the grid and fixed it with silver ink. Placing the grid inside a Nova Nanolab FEI microscope, we cut on the wire using the FIB, we tried two different configurations, shown in Fig. 2 a) and b), the second one resulting more resistant to manipulation. We placed nanoribbons across the Au wire bridging both ends, using an Omniprobe nanomanipulator inside the microscope and we fixed them by depositing platinum (see Fig 2)

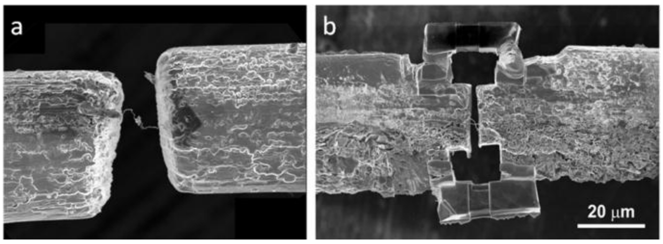

Fig. 2. Gold micro wire (~30 μm), a) transversal cut made with the FIB, and b) modified configuration of the cut with glass splints at each side of the wire to strengthen the device

Once the sample was prepared, using gold wires we connected the grid to two quartz plates covered with ITO, which were further connected to the electrodes of a digital multimeter. In order to avoid oxidation during Joule heating, we placed the device inside a chamber with constant Ar flux. The resulting IV curves for one experiment are presented in Figure 3.

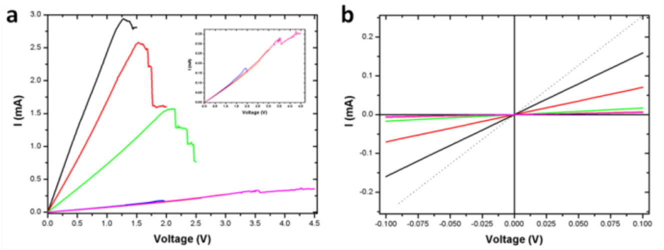

Fig. 3. IV curves of the electronic transport through the nanoribbon in Fig. 4a. We linearly applied voltage to our device and measured its response in current, plot a) displays the results of different experiments on the same sample up to 4.5 V. After each experiment we performed a short measurement from -0.1 V to 0.1 to evaluate changes in the resistivity of the material due to structural transformations induced by Joule heating. In plot b) we present such measurements, where the colors correspond to each experiment in a) (dotted line represent initial IV curve).

The initial morphology of such sample is shown in Figure 4a and b and the rupture of the nanoribbon after electric transport is shown in Figure 4c. Contamination of the sample during device preparation is evident in Figure 4b and d, where the contrast shows nanoparticles on the surface of the nanoribbon.

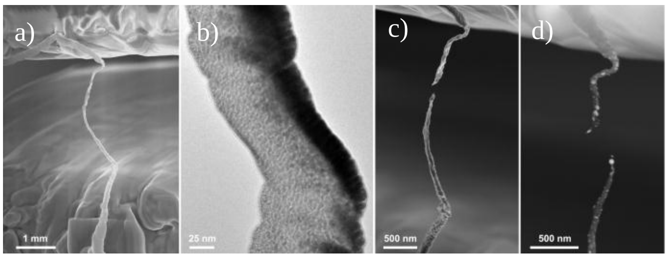

Fig. 4. Electron microscopy micrographs: a) SEM of nanoribbon making contact on both ends of the Au wire, b) TEM image of the nanoribbon, where metallic nanoparticles covering the structure are visible, c) the same nanoribbon in a) after passing current and causing rupture from the middle, away from the contacts, d) SEM image in vCD mode image of the nanoribbon, the contrast allows us to identify metallic nanoparticles in the surface of the nanoribbon due to Pt contamination during sample preparation

5. CONCLUSION

We have successfully constructed a TEM-electronic transport device in which the electron microscopy observation of a single nanoribbon and the electronic transport through it was possible. Our IV curves of the transport experiment show an interesting behavior that needs to be further investigated. We proved that the temperatures achieved by Joule heating are high enough to break the nanoribbon structure from the middle, away from the contacts that serve as heat sinks. Further challenges and obstacles to overcome remain in this experiment towards the quest of sharp edges, for example: avoid platinum contamination of the nanoribbon during sample preparation, and Raman spectroscopy measurements pre- and post-electronic transport.

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