Extremely high sensitive graphene sensor for NO_2 detection at room temperature

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Abstract

Graphene is nowadays more and more the fulcrum of the research in physics and materials science for both fundamental research and applications, thanks to its unique and supreme properties. Being a two-dimensional fabric and a surface without bulk, graphene has properties that make it sensitive to the environment and different gases. The sensing field has been showing particular interest due to the high sensitivity and flexibility of the material, the possibility to integrate graphene with other material and in portable devices.

Herein we report on extremely high sensitive devices based on graphene derived by a liquid-phase exfoliation process. The employment of this technique, besides having a low environmental impact, is very powerful: (a) it does not require sophisticated equipments with high operating and production costs; (b) it provides a high yield in terms of few-layers graphene; (c) it represents a potentially scalable production of large quantities. The ongoing challenge is to obtain larger flakes of higher quality.

To this aim, we prepared a colloidal suspension of graphene by dispersing 2.5 mg/mL of graphite powder (Sigma-Aldrich) in N-methyl-pyrrolidone (NMP) and sonicating in a low power bath (~16 W) for about 168h. The unexfoliated graphite flakes were removed by centrifugation at 1000 rpm for 45 minutes and the top half of the surnatant was collected. The so-obtained colloidal suspension was characterized by Dynamic Light Scattering technique with a Zetasizer Nano (Malvern Instruments), that supplies information on the mean size of the dispersed flakes (~140nm). Raman analysis (Figure 1) was performed on graphene films prepared by drop-casting few microliters of the graphene solution on the top of oxidized silicon wafers. Analogously, chemiresistive devices were fabricated by drop-casting onto Al_2O_3 transducers with interdigitated Au contacts. Devices were mounted in a Gas Sensor Characterization System (Kenosistec) and tested towards 350 ppb of NO_2 for 10 min in wet nitrogen with a flow of 500sccm at T=25^{0}C and relative humidity of 50%. The normalized conductance, reported in Figure 2a, shows the remarkable response of 27% with a signal-to-noise ratio (SNR) equal to. To the best of our knowledge this is the best performance reported in literature [1, 2].

The findings reported herein were compared to those obtained in a previous work where a much lower sonication time was used to exfoliate graphite (3h instead of 168h) [3]. As reported in several works by Coleman and coworkers [4, 5], a prolonged sonication time enriches the few layer content, that is the most sensitive fraction of the resultant films. The improvement of the graphene flakes concentration results in an overall improvement of the sensing performances with a conductance response (Figure 2a) up to 5-6 times higher than that shown in Figure 2b.

At the aim of understanding the actual interaction mechanism operating on the multilayer system presented above, further investigations are ongoing to compare its performances with those of a sensor device based on a single layer of graphene realized by CVD. Preliminary results seem to confirm the key role of the material fabrication process into the device chemical response.

References

[1] G. Lu, S .Park, K. Yu, R. S. Ruoff, L. E. Ocola, D. Rosenmann, and J.Chen, ACS Nano, **5** (2011) 1154-1164

[2] A. Serra, A. Buccolieri, E. Filippo, D. Manno, Sensors and Actuators B, **161** (2012) 359-365

[3] M. L. Miglietta, E. Massera, S. Romano, T. Polichetti, I. Nasti, F. Ricciardella, G. Fattoruso, G. Di Francia, Procedia Engineering, **25** (2011) 1145-1148

[4] J. Colemann, Acc Chem Res., **46** (2013) 14-22

[5] U. Khan, A. O'Neill, M. Lotya, S. De, J. N. Coleman, Small, **6** (2010) 864

Figures

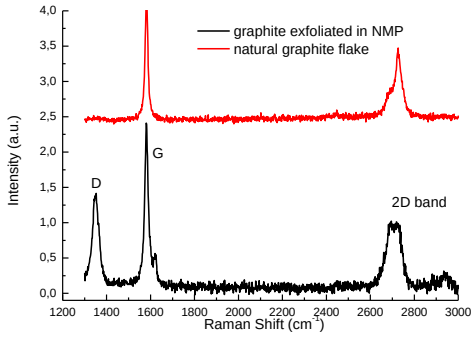

Figure 1: Raman analysis of graphite exfoliated in NMP compared to the spectrum of pristine material

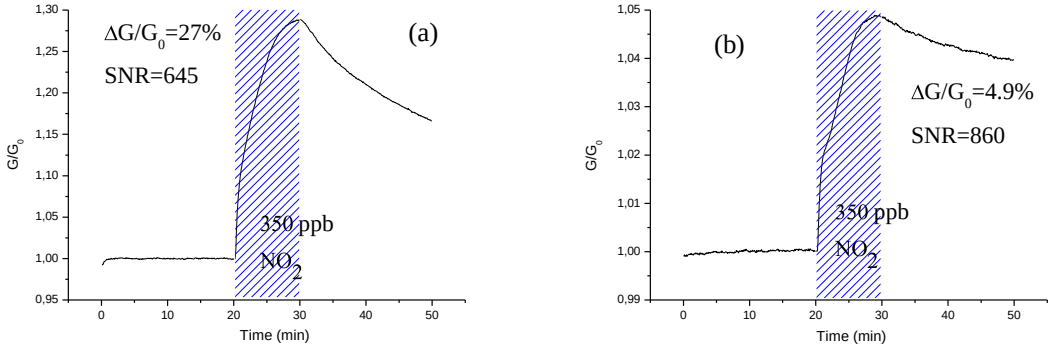

Figure 2: Conductance response upon exposure to 350ppb of NO_2 for 10 min in wet environment at 25°C related to sensing materials exfoliated by bath sonication for: a) 168 h; b) 3 h.