EFFECT OF RADIOFREQUENCY PLASMA TREATMENT ON MECHANICALLY EXFOLIATED GRAPHENE OXIDE PLATELETS

M. Cardinali^{a*}, L. Valentini^a and J. M. Kenny^{a,b}

 ^a Civil and Environmental Engineering Department, University of Perugia 05100 Terni-Italy
^b Institute of Polymer Science and Technology, (CSIC) 28006 Madrid-Spain
^{a*}Corresponding author: cardinali.marta@gmail.com

Graphene is the ideal candidates in advanced applications as electronics [1,2], hydrogen storage [3] and nano-biotechnology [4]. This great interest is motivated by their exceptional physical properties such as high charge carrier mobility up to $1,000,000 \text{ cm}^2/\text{Vs}$ [5-7]. Single layer graphene sheet was first obtained by mechanical exfoliation of graphite platelets [1]; this method is not suitable for large scale production of graphene, for this reason the solution route consisting of exfoliation of graphite by solvents and the stabilization of exfoliated graphene in liquid suspension have been addressed as the other common methods to produce graphene. Graphene flakes obtained by such chemical methods generally present reaggregation troubles due to their poor dispersion. One of the challenges besides engineering a transport band-gap in semi-metal graphene is the growth of large area graphene films of high quality. The easy processing and the versatile properties of graphene oxide (GO) make the reduction methods for such material attractive for restoring the charge carrier transport typically observed for ideal graphene. Here, we present a facile route to produce extended few layer flakes of graphene from graphene oxide platelets (GOPs). We propose the plasma treatment of GOPs. It was found how the application of a radio frequency Ar plasma assisted treatment exfoliates and reduces the initial bulk GO flakes without any use of further heating process or wet chemistry approaches. In this study we report the optimization of some operating parameters such as the argon flow rate, the substrate bias and the exposure time to obtain few layer reduced graphene oxide sheets.

Graphene oxide sheets produced by Hummers method were purchased from Cheaptubes. Water solution (1mg/1ml) of GO was prepared by tip sonication (750 W, 60% amplitude) for 1 h to yield a stable brown solution. After the sonication, the solution was transferred to a vial and it was centrifugated for 30 min at 600 rpm; this procedure was repeated two time. After that, the top of 12ml solution ($\approx 10\text{ml}$) was carefully extracted to remove residual aliquot of native dispersion in order to use a pristine one for the experimental process. The solution was deposited by drop cast method onto a silicon wafer substrate previously cleaned in an ultrasonic bath of acetone and ethanol and dried under nitrogen flow; after the drying the final product was large-area graphene oxide platelets. The film was then transferred on top of an another silicon substrate was subjected to plasma treatment in oxygen atmosphere to improve the wettability and the adhesion with the GOPs.

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After the transferring, the adhesive residual of the tape, were first removed in a bath of acetone for 2 min. and then in a bath of isopropanol for 1 min. The final deposit was dried under nitrogen flow. The plasma treatment of the samples was carried out in Ar plasma generated by radiofrequency plasma enhanced chemical vapor deposition source. The flow rate of the argon used for the deposition was 20 sccm. A 13.56 MHz plasma source with power fixed at 25W and 35W was used to create a plasma on top of the sample. Plasma treatments were carried out for 5 min., 20 min. and 40 min., respectively leaving the substrate temperature at 25° C.

The thickness of the prepared samples was investigated by atomic force microscopy (AFM). The infrared (IR) spectra of the deposited film were recorded in transmission mode between 250 cm⁻¹ and 3500 cm⁻¹. Raman spectroscopy of the prepared samples was carried out using a LabRam YvonJobin Raman spectrometer. The laser excitation of the HeNe laser was 633 nm and the power was kept at 2 mW. The spot size of the laser was about 12 μ m. Here we compare as figurative example the optical image of the extended GOPs transferred onto silicon substrate by mechanical exfoliation prior the plasma treatment and the plasma treated ones. The size of the untreated platelets is of several microns and the optical contrast allows a facile identification of the GOP edges with respect to the substrate (Fig. 1a). On the contrary the graphene oxide platelets plasma treated for a longer time (Fig. 1b) appear thinned having a low contrast. We are confident that this method will facilitate the preparation of graphene for applications in the field of transparent thin film using low cost native material and adopting an easy process at room temperature. In addition, the technique of plasma treatment, can open a new field in the production of graphene structures to be integrated in semiconductor devices [8].

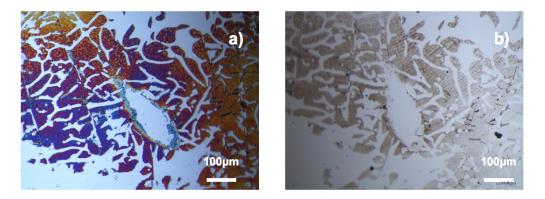


Figure 1: Optical microscopy images of (a) mechanically exfoliated GOPs and (b) plasma treated GOPs for 40 min.

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