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IT-7-P-2887 In situ reduction of graphene oxide by Joule heating with TEM-STM system

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Graphene has attracted a great deal of interest from scientists due to its intrinsic mechanical, thermal and electrical properties [1], [2]. Graphene, one-atom-thick layer of carbon, is a semiconductor with zero band gap [3] and high intrinsic mobility [4]. The excellent properties of graphene [5] have driven the search for methods for its large-scale production.

Graphene can be prepared by various methods [6] including micromechanical cleavage, epitaxial growth, chemical vapour deposition, exfoliation using graphite intercalation compounds and oxidation-reduction methods [7], [8]. These methods render high-quality graphene flakes although its low productivity makes them unsuitable for large-scale applications. The alternative strategy is the chemical oxidation of graphite or different carbon materials followed by chemical or thermal annealing.

Although the chemical oxidation of graphite is considered one of the most attractive methods to obtain graphene because it is cheaply, scalable and versatile, it presents the disadvantage that the O-containing groups produced by chemical oxidation, which make graphene oxide (GO) non-conducting [9], cannot be completely removed by the thermal annealing reduction. Thus, the level of reduction of GO is directly related to the conductivity, which can increase several orders of magnitude through the reduction process [10], [11].

In this work, GO, produced using a slight modification of the Hummers oxidation method from natural graphite flakes [12], has been in situ reduced by Joule heating in a TEM with a STM holder. The reduction of GO has been measured qualitatively from the comparison of conductivity of the sample before and after the reduction, all in the same experiment. Besides, with this technique it is possible to control the reduction from the measure of the conductivity of the sample and also characterize the sample during the experiment (both through TEM observation and through I-V characteristic). Indeed, the results show how GO has been reduced by observing a decrease of the resistance of more than four orders of magnitude.

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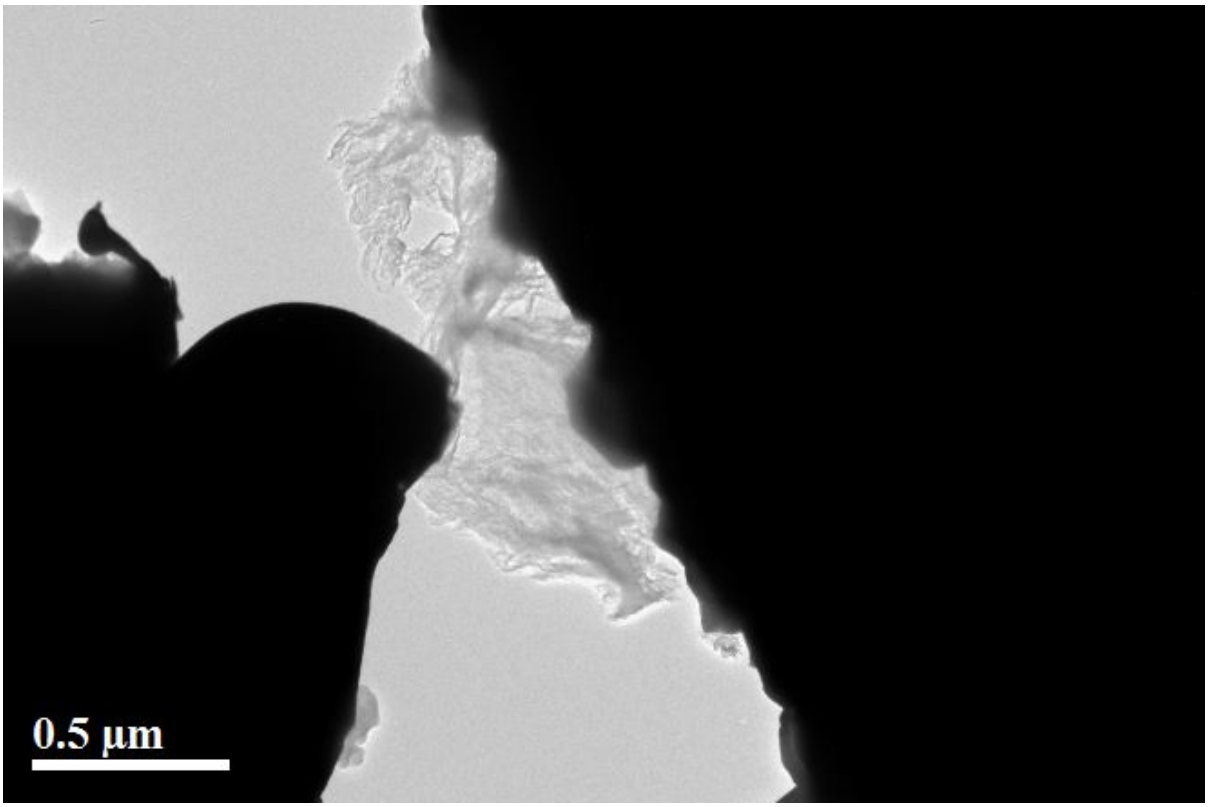

Fig. 1: TEM image of the tip contacting the GO during the experiment.