High-yield electrochemical exfoliation of graphene using high potentials

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With the aim to bring graphene from the lab to industrial application it is of great importance to find a suitable production method. While there are several methods, oftentimes the scalability, yield or graphene quality is not sufficient. Electrochemical approaches have shown the potential for scalability [1] and while anodic exfoliation suffers the problem of oxidation the reductive hydrogenation is fully reversible [2].

Here we present an up-scalable process [3] based on electrochemical hydrogenation of natural graphite flakes. Using boron-doped diamond as electrodes allows us to apply cell potentials as high as 60 V, since diamond is the only material able to withstand even such high voltages without degradation which would cause contamination of the graphene [4]. Under these conditions we were able to achieve a graphene yield of over 70 %.

The layer number of the flakes was determined by Raman spectroscopy. We developed an analysis of the 2D band symmetry. When using a symmetric profile the fit's coefficient of determination becomes a measure for the peak's symmetry. Calibrating with graphite and CVD monolayer graphene we were able to establish a benchmark by which to differentiate between multilayer graphene (<10 LG) and graphite (≥10 LG). The produced graphene flakes show a low D/G Raman band ratio after dehydrogenation by annealing of 0.17 on average, and an average flake size of about 30 µm² with some as large as 1000 µm².

References

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Figures

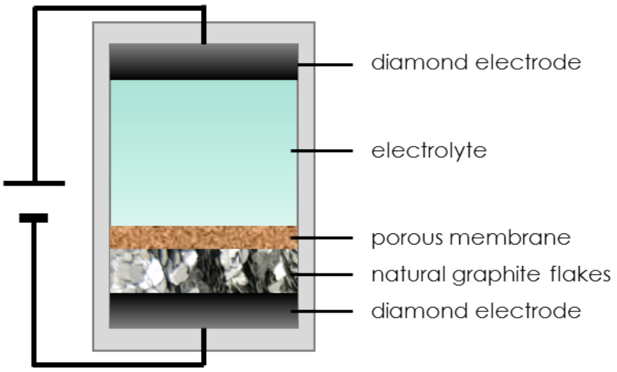

Figure 1: Schematic illustration of the reactor for electrochemical exfoliation.

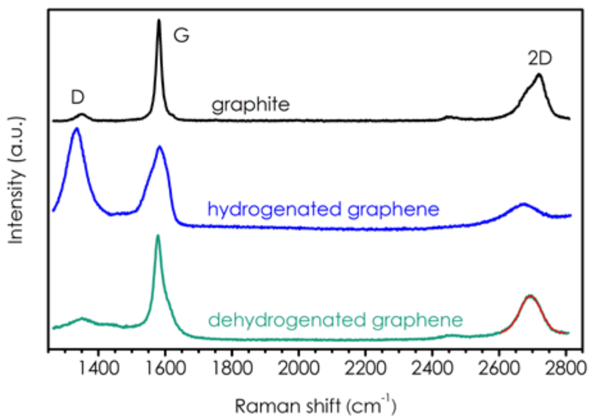

Figure 2: Raman spectra of the starting graphite, the hydrogenated graphene after the electrochemical process, and the dehydrogenated graphene with the symmetric Voigt profile fit of the 2D mode (red line).