GRAPHENE PREPARATION USING HIGH INTENSITY ULTRASOUND

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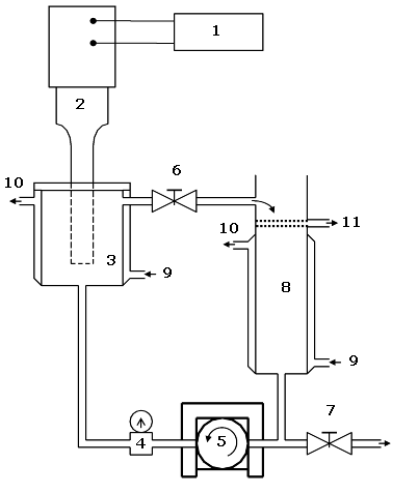
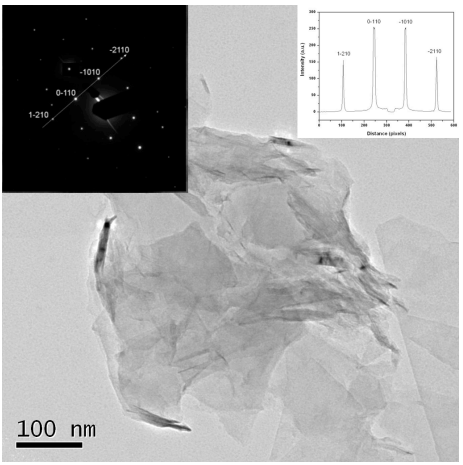
Mechanical exfoliation, called 'Scotch tape method' [1] was the first method used for preparation of single-layer graphene from natural graphite. Oxidation pathway to graphene preparation starts by graphite oxidation to graphite oxide or graphene oxide using strong oxidants in the environment of strong concentrated acids. The graphite oxide was first prepared by B.C. Brodie [2] in 1859 by reaction of graphite with a mixture of potassium chlorate $KClO_3$ and fuming nitric acid HNO_3. In 1898, Staudenmaier [3] improved this method by using concentrated sulfuric acid as well as fuming nitric acid and adding the chlorate in multiple aliquots over the course of the reaction; Hofmann [4] used concentrated nitric acid, concentrated sulfuric acid and $KClO_3$. The best-known Hummers methods [5] uses a mixture of sulfuric acid, sodium nitrate $NaNO_3$ and potassium permanganate $KMnO_4$. The exact composition of graphene oxide is defined by the various functional groups attached to graphene planes, mainly hydroxyl, ether and carbonyl groups. They are eliminated by strongly reducing agents, such as hydrazine hydrate or sodium borohydride, which convert graphene oxide to more or less reduced graphene oxide.

Liquid exfoliation [6] uses the effect of low-intensity ultrasound and suitable solvents. The layered material is sonicated for tens of hours in ultrasonic bath and individual nano-sheets are separated by centrifugation.

A new and efficient method to produce a large quantity of high quality and non-oxidized graphene flakes from powdered natural graphite by using a high-intensity cavitation field in a pressurized ultrasonic reactor is demonstrated [7]. The delamination (exfoliation) of natural graphite in the liquid phase depends on the physical effects of ultrasound, which break down the 3D graphite structure into a 2D graphene structure. The prepared graphene is of high purity and without defects because no strongly oxidizing chemicals are used and no toxic products are formed. Another advantage of this method is a dramatic reduction of preparation time.

Figure 1. The ultrasonic device for graphite exfoliation: 1. ultrasonic generator, 2. ultrasonic horn, 3. ultrasonic high pressure reactor, 4. pressure gauge, 5. pressure pump, 6. pressure valve, 7. drain valve, 8. compensatory vessel, 9. cooling liquid input, 10. cooling liquid output, 11. delaminated product output (left). HRTEM images of ultrasound exfoliated graphite in ethylene glycol. Inset SAED patterns (right).





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