Graphene oxide sheets chemically cross-linked by polyallylamine

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Methods

GO was synthesized from natural graphite (SP-1, Bay Carbon, MI) by the modified Hummers method.[1] Colloidal dispersions of individual graphene oxide sheets in purified water (18.2 MΩ resistance, 30 mg GO/10 mL water) were prepared with the aid of ultrasound (Fisher Scientific FS60 ultrasonic cleaning bath) in 20-mL batches. Adding polyallylamine (PAA) (Sigma-Aldrich, 20 wt% aqueous suspension of PAA with M_w = 17,000, 0.2 ml for 30 mg of GO, GO/PAA = 3.75 in weight) to the aqueous suspension of the graphene oxides immediately generated agglomerates. Additional ultra-sonication for 6 h under diluted condition (1 mg GO/5 mL water) afforded the homogeneous colloidal suspension of the PAA-modified graphene oxide sheets (Figure 1b). Both unmodified and PAA-modified graphene oxide paper was made by filtration of the resulting colloid through an Anodisc® membrane filter (47 mm in diameter, 0.2-μm pore size, Whatman, Middlesex, UK). To wash

the paper samples, purified water (3 x 10 mL) was passed through the wet paper prior to air drying and peeling from the filter. The air-dried paper samples were cut by blade and put into pocket of aluminium mesh and then soaked into purified water (3 x 50 mL) to further rinse.

Static mechanical uniaxial in-plane tensile tests were conducted with a dynamic mechanical analyzer (2980 DMA, TA Instruments, New Castle, DE). The samples were mounted using film tension clamps with a clamp compliance of ≈ 0.2 μm/N. The sample width was measured using standard calipers (Mitutoyo Co., Japan). The length between the clamps was measured by the DMA instrument, and the sample thickness was obtained from SEM imaging of the fracture edge. Normal tensile tests were initially conducted for 4 h at 35 ^{o}C in controlled force mode with a preload of 0.01 N and force was loaded with a force ramp rate of 0.05 N/min. XPS measurements of 'paper' samples were performed using an Omicron ESCA Probe (Omicron Nanotechnology, Taunusstein, Germany) with a monochromated Al K_{α} radiation ($h\nu$ = 1486.6 eV). Scanning electron microscope (SEM) images were taken using a Nova 600 NanoSEM (FEI Co, USA). FT-IR spectra (KBr pellets) of ground powder samples of papers were obtained using a Nexus 870 spectrometer (Thermo Nicolet, USA) with a tabletop optics module. Atlantic Microlab, Inc. (www.atlanticmicrolab.com) did the elemental analysis of paper samples.

Reference

(1) Park, S.; An, J.; Piner, R. D.; Jung, I.; Yang, D.; velamakanni, A.; Nguyen, S. T.; Ruoff, R. S. *Chem. Mater.* **2008**, *20*, 6592-6594.