



High-temperature multifunctional magnetoactive nickel graphene polyimide nanocomposites

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ABSTRACT

Composite Nickel graphene nanoparticles with hybrid magnetic and electrical properties were prepared. Nickel nanoparticles were tethered to the graphene through a carbon layers and were covered with an amorphous carbon layer to protect them from oxidation. Ni-graphene polyimide nanocomposites were prepared and exhibited magnetic characteristics and high electrical conductivity. The saturation magnetization of the polyimide nanocomposites increased with increasing magnetic nanoparticle content. First order reversal curve (FORC) magnetization showed a bimodal size distribution of the magnetic nanoparticles. Ultra-small-angle X-ray scattering (USAXS) of the nickel nanoparticles in Ni-graphene polyimide nanocomposites were estimated by a sphere model with bimodal size distribution. Nickel graphene nanoparticles were examined by high-resolution transmission electron microscopy (HR-TEM) where two size ranges of nickel were observed. Ni-graphene nanoparticles were well dispersed in the polyimide resin when examined by HR-TEM. Ni-graphene polyimide nanocomposites exhibited magnetic actuation when exposed to a static magnetic field.

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1. Introduction

Nanoparticles of silica [1], clay [2], carbon nanotube [3], POSS [4], titanium dioxide [5], gold [6], zinc oxide [7], silver [7], metallic magnetic nanoparticles [8], and graphene [9–14] have been examined extensively. Each nanoparticle has specific characteristics resulting from its atomic constituents, chemical state, and inherent physical characteristics due to structural organization of the atoms, defects, nanoscale size, shape, morphology, and aspect ratio. New generation of composite nanoparticles imparts characteristics of two or more nanoparticles where tailored design of composite nanoparticles could lead to new hybrid nanoparticles with multiple physical characteristics that supersede the characteristics of the individual nanoparticle constituents [1–14]. The multi-component composite nanoparticle provides the characteristics of each component, meanwhile new physical effects which result from the interactions between the two (or more) components could be achieved.

Graphene is a mono-atomic thin layer of carbon atoms connected with σ bonds and a shared π electron cloud forming a honeycomb structure of benzene rings [9–16]. Graphene has a charge carrier concentration up to 10^{13} cm^{-2} that is weakly temperature dependent [3–16]. The charge carrier travels with ballistic speed and behaves like massless Dirac fermions [9–16]. The thermal conductivity of single layer graphene has been measured in the range of 3000–5000 W/mK [9–16]. A graphene monolayer has high transparency along with exceptional electrical conductivity. Graphene's charge carrier mobility has been measured in the range of 3000 to 200,000 $\text{cm}^2/\text{V}\cdot\text{s}$ with a limiting theoretical value depending on defects in the crystalline lattice structure [9–16]. Graphitic film as monocrystalline graphene by mechanical delamination with superior electron transport and charge carrier mobility were first reported by Giem et al. [9–11]. Graphene nanosheets and stacks have been prepared by highly oxidative chemical and thermal processes [12,17–19]. These methods generally result in graphene oxide with high density of oxygenated functional groups such as hydroxyl, epoxide, carboxylic, and phenolics on the graphene surface which changes the carbon hybridization from sp^2 to sp^3 leading to a poor electron transport [12,17–19]. Graphene oxide

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can be reduced by chemical reduction methods such as hydrazine to generate graphene with lower oxygenated functional groups and higher inherent electrical and thermal conductivity [20].

In-situ grown titanium dioxide (TiO₂) graphene nanoparticles exhibited specific capacity more than double that of TiO₂ for lithium ion insertion [21]. Excellent charge carrier mobility of graphene ligand bounded iron oxide, titanium dioxide, or cadmium selenide were reported where the ligand bound nanoparticles act as charge reservoirs [21–23]. Ternary self-assembly of ordered metal oxide graphene has been reported where the hexagonal porous structure of graphene exhibited potential for energy storage [21]. SnO₂-graphene hybrids showed excellent charge–discharge properties for Li-ion batteries [21].

Nanoparticles have been incorporated into high performance polymer matrices such as polyimide to generate hybrid polyimide nanocomposites [24–28]. Light weight structural components for aeronautics and aerospace applications require multifunctional characteristics where enhancements of thermal, mechanical, and electrical properties can be achieved. In active polymer nanocomposites, addition of nanoparticles not only results in property enhancements but also induces a mechanical deformation when exposed to an external field. Active polymer nanocomposites responding to a range of external stimulus such as light [29], thermomechanical [30,31], electrical [32], and magnetic field [8,33] have been reported. Magnetic actuation due to the induced wireless remote mechanical deformation is of special interest for applications in the extreme environment of space [8,33]. Recently, large deformation of polyurethane magnetic nanocomposites containing iron manganese oxide super-paramagnetic nanoparticles in a magnetic field was reported [8]. Addition of graphene nanoparticles to the polymer resin matrix resulted in enhancements of physical and electrical properties [34], elastic modulus and mechanical properties [35,36], thermal properties [37], and light actuation [29]. Recently, we demonstrated that the addition of graphene nanosheets to a polycarbonate matrix results in significant enhancement of electrical conductivity [38]. A low percolation of 0.14 vol.% was obtained when graphene was mixed with PC using emulsion mixing [38].

Magnetic nanoparticles are metal/metal oxide nanoparticles with magnetic properties depending on the chemical composition, size, and aspect ratio [39,40]. Below a critical diameter, they are super-paramagnetic where their magnetic moment has a fast response to a magnetic field with no hysteresis [39,40]. Nickel nanoparticles have a critical diameter of 55 nm [40], and also contain free electron contributing to its excellent electrical charge transport with slow oxidation rate.

In-situ synthesis of graphene metal composite nanoparticles by thermal reduction has been reported [41–43]. Adsorption of metallic nanoparticles such as Au, Pt, and Pd on the graphene oxide generating metallic graphene nanocomposites for catalytic process use has been studied [41]. Magnetite on the graphene oxide with several surface particle densities has been prepared [42]. Iron oxide (Fe₃O₄) graphene composite particle exhibited magnetic characteristics [43]. Graphene iron oxide nanoparticles are magnetic [43], however, their electrical and thermal transport properties would not be comparable to graphene due to the oxide state of the iron oxide. Nickel has unique properties of high oxidative stability, meanwhile possessing magnetic properties. Its alloy with iron and molybdenum exhibits magnetic shielding properties. Tethering nickel nanoparticles on the graphene oxide surface not only generate magnetic characteristics, but also maintain the electrical conductivity of the graphene nanosheets. These nickel metallic nanoparticles contain free electrons which contribute to the transport properties of the graphene. Ni-graphene hybrid nanoparticles have superior properties compared to individual graphene or magnetic nanoparticles. There is no report of the hybrid graphene nickel nanoparticles with both magnetic and

electrical properties and their polymer nanocomposites to the best of our knowledge.

Here, we report preparation of hybrid nickel tethered graphene composite nanoparticles, their polyimide nanocomposites, and their morphology and properties. These composite nanoparticles have both magnetic characteristics of nickel and high strength and excellent electrical and thermal conductivity of graphene. Their magnetic characteristics and morphology were examined by first order reversal (FORC) curve, USAX and HR-TEM. Ni-graphene polyimide exhibited high electrical conductivity and actuation performance.

2. Experimental section

2.1. Raw materials

Bisphenol A dianhydride (BPADA), 4,4'-Bis(4-aminophenoxy) biphenyl (BAPP), nickel acetyl acetonate were purchased from Polyscience Inc., Chriskev, and J.T. Baker Chemical Co., respectively. Methanol, chloroform, N-methyl-2-pyrrolidone (NMP), and acetone were obtained from Sigma and used as received. Oxygenated graphene and reduced graphene were generously donated by Vorbeck Materials Corp.

2.2. Synthesis of Ni-Graphene

A dispersion of highly oxygenated graphene (0.1145 g) in NMP (50 cc) was prepared by mixing and sonication. A second solution of Ni(acac)₂ (4 mmol, 1.0277 g) in NMP (20 cc) was prepared. The graphene NMP dispersion was heated to 190 °C and stirred under nitrogen atmosphere while the Ni(acac)₂ solution was added dropwise during a hour period. The resultant was centrifuged, and washed with acetone several times. Then, the sample was dried in vacuum oven at 70 °C for 40 min. Synthesis of Polyimide: Polycondensation reaction of flexible polyimide based on BPADA and BAPP was performed using *in-situ* thermal imidization in anhydrous NMP under dry nitrogen flow based on a recent published procedure [28].

2.3. Nanocomposite preparations

Graphene (Ni-graphene) was dispersed in chloroform (0.1 mg/cm³) by sonication for 1 h, followed by another 1 h sonication after mixing with a 20 wt% solution of polyimide in chloroform. An ice bath was used during sonication to maintain the dispersion temperature 25–30 °C during the first hour of sonication. After completion of 2 h sonication, the Ni-graphene/polyimide/chloroform dispersions were mixed at room temperature with a magnet under flow of nitrogen for 1 h until a viscous dispersion was obtained. A minimum solvent amount in dispersion was removed during solvent casting to lock the nanoparticle in well dispersed form. Ni-graphene polyimide nanocomposite films were prepared by solution casting and removing the residue of the solvent in a vacuum oven with stepwise increase of temperature from 60 °C to 170 °C. Thermal gravimetric analysis confirmed complete solvent removal for all samples. Graphene (Ni-graphene) polyimide nanocomposites in the range of 0.0022–3.67 vol.% were tested and examined.

3. Results and discussions

3.1. Ni-graphene nanoparticle

3.1.1. Synthesis of Ni-graphene composite nanoparticle

Highly wrinkled exfoliated graphene and graphene stacks with extremely large aspect ratio and large surface area has been

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