



Mechanical and electrical properties of carbon fiber composites with incorporation of graphene nanoplatelets at the fiber–matrix interphase



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ARTICLE INFO

Article history:

Received 16 July 2014

Received in revised form 30 September 2014

Accepted 2 October 2014

Available online 14 October 2014

Keywords:

A. Carbon fiber

A. Nano-structures

B. Mechanical properties

E. Autoclave

ABSTRACT

In this study, carbon fibers (CFs) were coated with graphene nanoplatelets (GnP), using a robust and continuous coating process. CFs were directly immersed in a stable GnP suspension and the coating conditions were optimized in order to obtain a high density of homogeneously and well-dispersed GnP. GnP coated CFs/epoxy composites were manufactured by a prepreg and lay-up method, and the mechanical properties and electrical conductivity of the composites were assessed. The GnP coated CFs/epoxy composites showed 52%, 7%, and 19% of increase in comparison with non-coated CFs/epoxy composites, for 90° flexural strength, 0° flexural strength and interlaminar shear strength, respectively. Meanwhile, incorporating GnP in the CF/epoxy interphase significantly improved the electrical conductivity through the thickness direction by creating a conductive path between the fibers.

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1. Introduction

Due to their high specific strength and stiffness, fiber reinforced polymer composites have been increasingly in applications for which light weight is essential, like in the aerospace or aeronautics industries. Carbon fibers (CFs) attract high interest because of their superior strength and modulus, low density and reasonable cost [1,2]. However, the use of conventional CFs composites may be hindered by the potential weak off-axis strength properties and poor crack resistance and delamination [3–6]. Furthermore, CF reinforced polymer composites still do not meet particular demands for applications in specific harsh environments that require both high electrical conductivity and high toughness.

Recently, fiber reinforced polymer composites incorporating nanoparticles have triggered considerable interest due to the potential extensive applications that conventional fiber reinforced polymer composites cannot offer [7–9]. In comparison with other nanoparticles, Graphene nanoplatelets (GnP), particles made of a few graphene layers stacked together in a platelet morphology, have emerged as a potential candidate for obtaining functional

and structural composite materials with improved properties due to their superior mechanical properties such as modulus and strength, excellent electrical conductivity, good thermal conductivity and low cost [10–13]. One of the most important parameters in manufacturing GnP/CF hybrid composites is the quality of the GnP dispersion. The intrinsic Van de Waals interactions existing between graphene nanoplatelets force them to aggregate, contributing to the development of local stress when an external force is applied on the composite part. This can significantly affect the strength of the composite. For example, Zhang et al. [14] reported that the degree of GnP dispersion in a fiber sizing solution strongly influenced the mechanical properties of the corresponding composites. GnP can be well dispersed in a small number of organic solvents such as N-methyl-pyrrolidone (NMP) [15]. The adding of GnP in fiber reinforced composites attracted some interest recently [16,17], especially in terms of mechanical and electrical properties. It was reported that it could also prevent the delamination/buckling of the fibers [18], and promote the redistribution of stresses around cracks starting near the fiber surface, thereby delaying the crack propagation in the interphase region [14]. Nevertheless, scaling up the manufacture of GnP coated CFs and their composites is still a challenge, because previous studies were focused on lab-scale processing which implementation in the CF industry seems to be compromised in electrophoretic deposition (bath process) [19], chemical vapor deposition (CVD) [20] or solvothermal methods (harsh conditions) [21].

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A new method for producing GnP coated CFs by a simple dip-coating procedure was studied in our group. Uniform coating of the CF surface, with a high density of well-dispersed GnP particles was achieved, as shown in Fig. 1(a). During the process, GnP were dispersed by sonication in a NMP/epoxy solution, and the CFs were dipped into the GnP/NMP/epoxy suspension by hand, then GnP coated CF was dried in oven at 75 °C for 1 h and 60 °C for 3 h. This method can be easily scaled-up for an eventual continuous production of GnP coated CFs at an industrial scale. It was also found that a concentration of 3 wt.% of GnP in the coating solution led to the highest value (52 MPa) if the interfacial shear strength (IFSS) measured by a fragmentation test. An increase of the interfacial adhesion measured by a single fiber test usually corresponds to an increase of the mechanical properties of unidirectional composite that are sensitive to interfacial adhesion, such as off-axis properties (90° flexural strength, transverse tensile properties) and interlaminar shear properties [22]. Unidirectional composite plates at high fiber volume fractions were fabricated in order to fully understand what is the influence of the incorporation of GnP particles in the interphase region compared to other interactions (fiber–fiber interactions, dispersion of the single fibers in the matrix, void content linked to the manufacturing process, ...).

In this study, CFs were coated with GnP using a straightforward continuous solution coating process, and unidirectional CF-epoxy composites were manufactured using prepreg and lay-up methods. Mechanical tests including inter-laminar shear strength, 90° flexural tests and 0° flexural tests, as well as electrical conductivity measurement were used to examine the effect of the adding of GnP in the interphase region. The fracture surfaces after flexural testing were also observed by scanning electron microscopy (SEM).

2. Experimental

2.1. Materials

The exfoliated graphene nanoplatelets (GnP) used in this study were GnP-C-750 from XG Sciences Inc. Their average surface area is 750 m²/g. As received, the particles are agglomerated into a few microns size aggregates, but when dispersed properly in the appropriate solvent, their diameter is around 100 nm. An X-ray photoelectron spectroscopy (XPS) analysis gave elemental concentrations for oxygen and carbon of 7% and 93%, respectively.

Intermediate modulus PAN-based AS4 CFs (12 k) surface treated and unsized were supplied by Hexcel Co. Their average diameter was 7.1 μm. The surface of the fibers was continuously treated by UV/ozone oxidation with an MSU developed surface treatment in our laboratory. More information about the process and the CF surface properties generated by this surface treatment can be found elsewhere [23,24]. The results of this UV surface treatment was the combined effect of surface functionalization and limited etching of the surface also created a topography that was adapted for chemical bonding and mechanical interlocking with the matrix.

Epoxy resin Epon 828 was supplied by Hexion Specialty Chemicals and was used as received. M-phenylenediamine (mPDA, Sigma–Aldrich Corporation, used as received) was used as the curing agent. 1-Methyl-2-pyrrolidinone (NMP, Sigma–Aldrich Corporation, used as received) was used as the solvent for the coating solution.

2.2. Preparation of GnP suspension as the coating solution

A mix of Epon 828 and mPDA (at a concentration of 7.5 wt.%) was dissolved in NMP to obtain a solution with a concentration of the mix of 4 wt.%. GnP was then added into the NMP/Epon 828/mPDA solution at a concentration of 3 wt.%, and was dispersed with a horn sonication device (750-Watt Ultrasonic Homogenizer, Cole-Parmer) using a 220 W output power for 2 h and with constant magnetic stirring. The GnP suspension was centrifuged at 500 rpm for 1 h to separate any remaining large agglomerates. Over 90% of the initial GnP was collected for use. This was determined by measuring the weight of GnP sediments after drying. The diameter of GnP particles after sonication and centrifugation was less than 100 nm, as seen in Fig. 1(b). The freshly prepared GnP suspension was transferred to the coating bath and continually stirred during the fiber coating procedure. The choice of the solvent, the concentrations of GnP, epoxy resin and curing agent in the coating solution and the sonication parameters have been optimized to obtained the highest value of interfacial shear strength with an epoxy matrix (measured by the single fiber fragmentation test), as reported previously.

2.3. Fabrication of CF unidirectional epoxy composites

2.3.1. Coating of the CFs

The fiber coating process is illustrated in Fig. 2(a). The non-coated and UV-ozone surface treated CFs were unwound and passed through the coating solution with the use of mirror-finished stainless steel rollers. The tow was spread as much as possible to insure a homogeneous coating of the fibers. The feeding speed of the CF tow was 0.3 m * min⁻¹. After immersion, the fibers were dried by successive exposure to heat in two tower-shaped furnaces. The temperature inside the furnaces was set at 125 °C and the total residence time of the fibers in the furnaces was ~6.5 min. The sized fibers were then wound onto a stainless steel mandrel which was transferred to an oven set at 60 °C for 3 h, in order to completely remove any remaining NMP. The comparative epoxy coated CFs (without GnP particles) were prepared with the same conditions, using a solution of Epon 828 + mPDA in NMP at a concentration of 4 wt.%.

2.3.2. Manufacture of prepregs

Prepregs were manufactured using a drum-winder technique, as shown in Fig. 2(b). The temperature of the resin pot, the traversing guide roller and the flattening pins was set at 52 °C. The CF tow

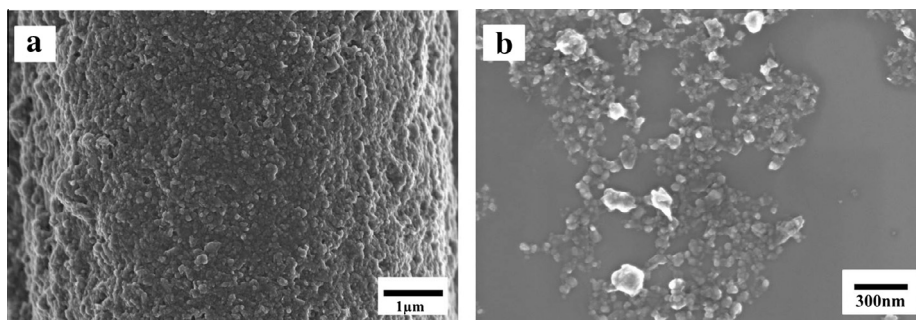


Fig. 1. SEM micrograph of (a) GnP coated CFs, and (b) SEM observations of GnP individual particles after sonication and centrifugation.

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