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High sensitive damage sensors based on the use of functionalized graphene nanoplatelets coated fabrics as reinforcement in multiscale composite materials

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ABSTRACT

Functionalized graphene nanoplatelets networks created through glass fiber fabrics were used to detect and locate damage in multiscale composite materials. The electrical behavior of multiscale composite materials was strongly influenced by microstructural features. Coated fabrics presented high sensitivity to breakage of fibers due to the preferential orientation of f-GNPs through fibers. This sensitivity was higher when damage was induced perpendicular to the fiber direction and the region where damage could be detected was bigger in the case of locating the measuring electrical channels perpendicular to fiber direction. These phenomena are related again to the morphology of the electrical network through the coating. Due to the insulating character through thickness of composites, detection and location was limited to layers of fabric of the composite. Nevertheless, self-sensors had the capacity of detecting and locating damage with high sensitivity by means of abrupt increases in electrical resistance induced by breakage.

1. Introduction

During last decades, development of structural health monitoring (SHM) systems has increasingly attracted the interest of industry [1–3]. Internal damage is difficult to detect in continuous fiber reinforced composite materials during service. For this reason, the integration of sensors that could provide the state of the structure would be advantageous [4]. As an example, wind farms are moving to new emplacements, such as off-shore, where conditions are more aggressive making inspection and maintenance operations more costly and risky [5–8].

Particularly, the use of structural materials with self-sensing capabilities could be beneficial as it avoids the use of external sensors [9]. The incorporation of electrically conductive nanoparticles (carbon nanotubes, carbon nanofibers, silver nanoparticles …) into the epoxy matrix has been widely studied to achieve sensing properties in polymer based composite materials [10–13]. Once enough quantity of nanoparticles is incorporated (above the percolation threshold), the composite material become electrically conductive due to the creation of an electrical network formed by adjacent and overlying nanoparticles [14,15]. This network created by the nanoparticles is the responsible of self-sensing properties. If the material is strained, the electrical network is modified and so the electrical resistance [16,17]. In the case of damage, it causes a discontinuity in the electrical network, provoking the breakage of electrically conductive paths and then, the augment of the electrical resistance [18,19]. But the use of nanoreinforcement leads to one important issue to take into account: the incorporation of nanoparticles in composite materials makes necessary the development of new structural models that asses size effect at nanoscale evolving structural mechanics [20–24].

The use of graphene nanoplatelets (GNPs) as nanoreinforcement has been demonstrated to result in high sensitivities [25–27] due to their 2D character [28], which makes the electrical network more susceptible to strain and damage [29].

In the present work, self-sensors composite materials based on the coating of glass fiber fabrics with a mixture of functionalized GNPs and sizing agent are characterized. Damage was induced in different configurations directly in coated fabrics and multiscale composite materials in order to elucidate dependence on damage orientation and sensitivity.

2. Experimental

2.1. Materials

Epoxy resin was Diglycidyl Ether of Bisphenol A (DGEBA), commercially named *LY556*, cured with an aromatic amine (*XB3473*), both

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purchased to Huntsman. Reinforcement of composite materials was unidirectional glass fiber fabric denominated *Hex-Force® 01031 1000 TF970*, type *E fabric UD 4H Satin*, purchased from *Hexcel*. The fabric is unbalanced with a weight distribution of fiber of 87/13 (warp/weft), the thickness of each layer is 0.24 mm and the nominal weight 305 g/m^2. The sizing agent, which particles were incorporated in, was provided by *Nanocyl* (*Sizicyl™ X1*).

Graphene nanoplatelets (f-GNPs) used in this work were NH_2 functionalized and were purchased from *Cheaptubes* with the name *Grade 4 Graphene Nanoplatelets – NH_2*. The thickness of f-GNPs was below 6 nm and the average lateral size of 5 µm.

2.2. Methods

2.2.1. Manufacturing of multiscale composite materials

Dispersion of the f-GNPs was carried out using probe sonication (*Hielscher UP400S*). The mixture was constituted by 5 wt% of f-GNPs into a solution of distilled water and sizing agent in a proportion of 1:1 in weight. Sonication was applied during 45 min using a cycle of 0.5 s and 50% of amplitude. Once dispersion process was completed, glass fiber fabrics were impregnated with the mixture by dip coating and dried at 150 °C (24 h).

Coated fabrics were used as reinforcement in multiscale composite materials. Vacuum assisted resin infusion molding (VARIM) was used in order to manufacture composite materials. Previous to the infusion, the resin was degassed at 80 °C (15 min) under vacuum and then mixed with the hardener in a stoichiometric ratio (100:23 wt). Curing was performed maintaining the vacuum at 140 °C (8 h).

2.2.2. Characterization of coated glass fabrics and multiscale composite materials

Electrical conductivity of the multiscale composite materials was measured along three orthogonal directions by using a source-meter unit instrument *Keithley 2410*. Silver paint was used at the contact surfaces in order to minimize the contact resistance.

Scanning electron microscopy (SEM), *Hitachi S-2100N*, was used to analyze the microstructure and distribution of the f-GNPs through the composite materials.

Sensitivity to damage of f-GNPs coated fabrics was tested inducing controlled linear damage by cutting the fabrics in different steps. During the evolution of the linear damage, the electrical response was simultaneously measured with a source-meter *Agilent 34410A*. In order to measure the electrical resistance of the fabrics, two copper electrodes, which define the measuring channel, were attached to the surface of fabrics by using silver paint to reduce electrical contact resistance and then fixed with hot melt adhesive. Configurations of the electrical contacts vary from one test to another and, for that reason, they are schematized in the correspondent figure.

Damage sensing capabilities of composite materials were studied under standardized 3-point bending and interlaminar shear strength tests, following the ASTM D790 [30] and D2344 [31], respectively. In the case of 3-point bending tests, copper electrodes were attached on the surfaces of samples with a separation of 15 mm using silver paint in order to minimize the electrical contact resistance (Fig. 1a). Two channels were defined, one on the compression subjected surface and other on the tensile subjected one. In the case of interlaminar shear strength tests, contacts were located at a distance of 21 mm, which coincides with the edge of the samples, on both surfaces (Fig. 1b). This configuration permits to obtain two measuring channels, sectioning the volume of the sample.

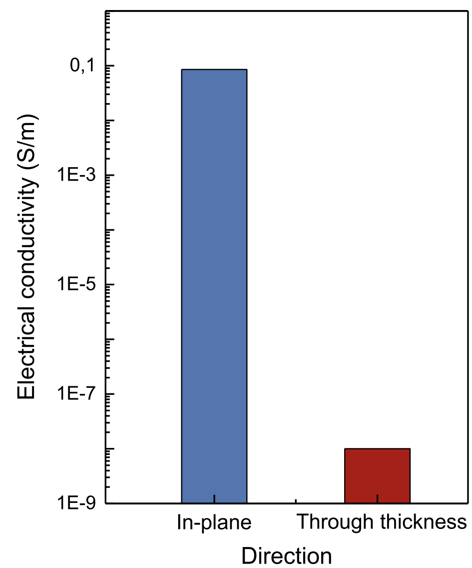

Fig. 2. Electrical conductivity of multiscale composite materials.

3. Results and discussion

3.1. Electrical conductivity and microstructural features of multiscale composite materials

Electrical conductivities of the multiscale composite material in the in-plane directions and through the thickness are shown in Fig. 2. The electrical conductivity in the in-plane direction was in the order of 10^{-2} S/m. Nevertheless, through the thickness the electrical conductivity was found to be lower than 10^{-7} S/m. This difference of more than 5 orders of magnitude is due to the disposition of f-GNPs. Similar results were obtained by G. J. Gallo et al. [18] in carbon nanotubes reinforce composites and can be justified by microstructural features.

Fig. 3 shows representative SEM images showing the microstructure of the multiscale composite materials. It can be observed that f-GNPs were localized in the vicinities of the fabric layer (Fig. 3a, in red) and the nanoreinforcement did not penetrate into the glass fabric (Fig. 3b, in blue). Although penetration into the fabric was not observed, some accessible spaces, which were opened to the surface, between fibers and tows were found to be occupied (Fig. 3c–e, in red). But there was an area poorly covered by the f-GNPs (Fig. 3, in blue), causing the insulating character through the thickness. The electrical resistance through the thickness is defined by serial resistances formed by

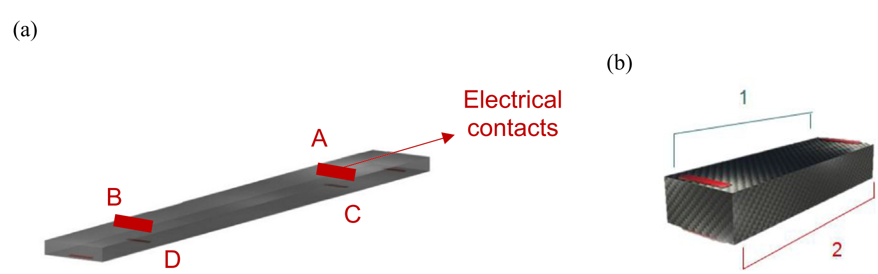

Fig. 1. Schematic representation of mechanical tests showing the location of the electrical contacts: (a) three-point bending and (b) interlaminar shear strength tests.