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Material properties

Improved thermo-oxidative stability of three-dimensional and four-directional braided carbon fiber/epoxy hierarchical composites using graphene-reinforced gradient interface layer

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ABSTRACT

In order to improve the thermo-oxidative stability of three-dimensional and four-directional braided carbon fiber/epoxy composites, we introduced a gradient interphase reinforced by graphene nanoplatelets (GN) between the carbon fiber and the matrix, with a liquid phase deposition strategy. Both the interlaminar shear strength and the flexural strength of the composites were improved after thermo-oxidative aging at 140 °C for various durations (up to 1200 h). The interfacial reinforcing mechanisms are explored by analyzing the structure of the interfacial phase, thermal conductivity, weight loss, surface topography, fiber/matrix interfacial morphology and thermomechanical properties of the composites. Results indicate that the GN-reinforced gradient interphase provides an effective shield against interface oxidation, assists in thermal stress transfer, and restricts the movement of the different phases of materials at the composite interface.

1. Introduction

Carbon fiber (CF) polymer matrix composites (PMCs) are extensively employed in the aerospace industry owing to their high specific mechanical properties [1]. However, degradation of the properties of the constituents, i.e., fiber, matrix and fiber/matrix interphase, occurs when the CFPMCs are exposed to oxidative environments at high temperatures. In particular, fiber/matrix interface oxidation governs the oxidation rate of the CFPMCs [2,3]. For traditional laminated composites, the occurrence and development of microscopic damage (fiber/matrix debonding or micro-cracks) promote delamination [4], which degrades the structural properties and reduces service life of CFPMCs. However, the emergence of new advanced aerospace applications ("hot" structures) has given a new impetus to the development of CFPMCs with higher thermo-oxidative stability (TOS).

Compared to conventional laminated composites, the integrated structure of three-dimensional (3D) and four-directional (4Dir) braided composites can improve the TOS of the CFPMCs by enhancing the ability of the fibers to bear the external load and resist delamination, despite the deterioration in the matrix resin and interface properties after thermo-oxidative aging (TOA) for long periods of time [5]. Unfortunately, however, the flexural strength (FS) of the 3D-4Dir braided composite decreases significantly when the fiber/matrix interface performance declines extensively after TOA [6].

Although substantial research has been conducted on improving the interface performance of CFPMCs, only a small portion has focused on the effect of interface

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performance on the TOS of CFPMCs [3,7]. Bowles [7] studied the effects of fiber surface modification on the TOS of Graphite/PMR-15 composites and noted that the AS-4G composites exhibited a higher interlaminar shear strength (ILSS) compared to AS-4 composites at room temperature. However, after TOA, the ILSS of the AS-4G composites (in the above study) was somewhat worse than that of the AS-4 composites. This was attributed to the 1.5 % low temperature epoxy sizing at the fiber/matrix interface of the AS-4G composites. The low temperature epoxy sizing on the fiber surfaces degraded rapidly, resulting in diffusion of air along the fiber/matrix interfaces, accelerating the interface oxidation. This example illustrates that the CFPMCs with higher bonding strengths at room temperature do not necessarily have higher TOS after TOA. Furthermore, it demonstrates that the TOS of the reinforced material itself has a great influence on the TOS of CFPMCs. The introduction of GN into conventional continuous fiber reinforced PMCs to create hierarchical reinforcement structures is currently of significant interest owing to the GN's unique structure, outstanding strength and modulus, and excellent electrical and thermal properties [8,9]. Recent research demonstrates that, by incorporating GN, significant improvements are achieved in CFPMCs, especially for the fiber/matrix interphase and the matrix-dominated out-of-plane performance, such as interfacial shear strength [8,10], impact strength [8] and fatigue resistance [9]. However, there are few studies on the properties of GN-reinforced CFPMCs after TOA.

Therefore, the purpose of the current investigation was to determine the role of a GN-reinforced hierarchical interface on the TOS of a 3D-4Dir braided/epoxy composite. The specimens were thermally oxidized at 140 °C for various durations. After exposure to the high temperature, composites were characterized to: (1) determine the weight loss and reduction in FS and ILSS at different exposure times, (2) observe the corresponding micro-cracks and surface damage, and (3) understand the reinforcement mechanism of the GN-reinforced hierarchical interface.

2. Experimental details

2.1. Materials

T700-12K CF (Toray) was used for this study. An epoxy resin JC-02A based on diglycidyl ether of bisphenol A (Changshu Jaffa Chemical Co., Ltd.) with hardener JC-02B (improved methyl tetrahydrophthalic anhydride) and accelerant JC-02C (tertiary amine) was used for the matrix. Graphite powder with an average diameter of 10 μm was purchased from Qingdao AoKe ShiMo Co. Ltd., China. Concentrated H_2SO_4 (98%), concentrated H_3PO_4 (85%), $KMNO_4$, acetone and 30% H_2O_2 (Tianjin Fengchuan Co., Ltd.) were used to make the GN.

2.2. Preparation of GN-coated CFs

The processes for making graphite oxide (GO), GN, and GN-coated CFs are detailed in reference [11]. The difference between the GN-coated CFs used in our study and those in reference [11] is that the former were made with commercial CFs without any treatment. The CFs absorbed 1 wt.% GN, relative to the composites. A GN concentration of 1 wt.% was chosen because previous researchers [11] have shown that this proportion is the best choice to improve the FS and ILSS of CF/epoxy composites at room temperature.

2.3. Preparation of 3D-4Dir braided CF/epoxy composites (BCs), 3D-4Dir braided GN-coated CF/epoxy composites (BGCs), and neat resin (NR)

Both the 3D-4Dir braided commercial CF preforms and 3D-4Dir braided GN-coated CF preforms were manufactured by the intertwining or orthogonal interlacing of four sets of yarns-braiders to form a 3D sheet fabric [12]. The 3D-4Dir braided architecture is illustrated in Fig. 1(c). It is characterized by almost all the braider yarns being offset at different angles between the in-plane and through-thickness directions, which can be seen clearly from the

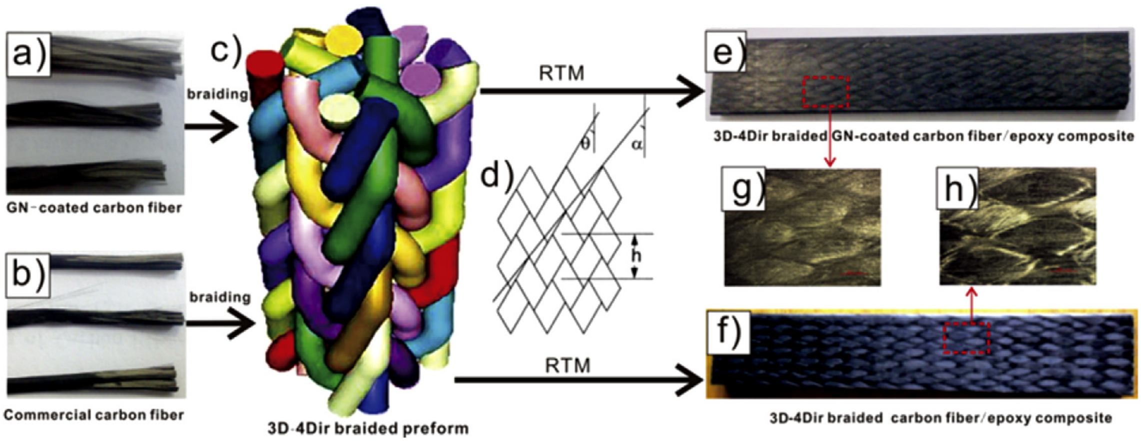

Fig. 1. Graphene nanoplatelet(GN)-coated carbon fibers(CFs) (a), commercial CFs (b), three-dimensional(3D) and four-directional(4Dir) braided architecture (c), an idealized model of the 3D-4Dir braided preform surface (d), a flexural specimen of 3D-4Dir braided GN-coated CF/epoxy composites (BGCs) (e) and 3D-4Dir braided CF/epoxy composites (BCs) (f), zoom on the surface of BGCs (g) and BCs (h).