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Mechanical properties of graphene platelets reinforced syntactic foams

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ABSTRACT

Graphene platelets (GPs) are two-dimensional thin plates containing few layers of graphene sheets. Compressive and tensile behaviors of epoxy-based syntactic foams with pristine GPs as additives are discussed in this article. Four sets of syntactic foams containing 0, 0.1, 0.3, and 0.5 vol.% of GPs were fabricated and tested. The volume fraction of microballoons in all syntactic foam samples was kept constant at 30%. Results indicated that the compressive and tensile moduli of the syntactic foams were significantly improved as compared to samples that did not contain GPs. The addition of GPs also enhanced the tensile strength while the compressive strength was only slightly increased. Optimal property improvements were obtained for very low filling fraction of approximately 0.3 vol.%. Samples with higher volume fraction of GPs (0.5%) showed deterioration in mechanical properties when compared to other GP containing samples. Transmission microscopy study indicated formation of voids enclosed by undispersed GPs in the samples which could explain the decline of the properties. High matrix porosity could also play an important role in this observation. Utilizing surface modified GPs could allow incorporation of higher volume fraction of GPs homogeneously, thus improving the mechanical properties of the syntactic foams.

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1. Introduction

Syntactic foams are fabricated by dispersing hollow microballoons in a polymeric matrix. The presence of the microballoons provides a closed cell porosity thereby reducing the weight, and preventing thermal transport and moisture absorption in the material. In addition, the microballoons provide high compressive strength, high damage tolerance, and good dimensional stability to the material. Due to these advantages, syntactic foams can find applications in aerospace industries as a core materials and ablative barrier coatings [1]. However, syntactic foams are ductile in compression and extremely brittle in tension. Their tensile strength decreases further with an increase in the volume fraction of microballoons [2]. In addition, results from microstructural analysis of tensile and shear fracture surfaces revealed that failure of syntactic foams is matrix-dominated [3]. In foam-core sandwich structural members, compressive, tensile, and shear properties are critical and need improvement. Specifically, the strength and reliability of these structural members, when used for aerospace applications, is very important since their failure could lead to serious life and property loss. Hence, interest in utilizing the advantage of lightweight syntactic foams for aerospace sandwich structures drove researchers to explore different reinforcement methods to improve their properties. Most of the studies on matrix reinforcement have been focused on the use of long and short micro-sized fibers [4–9]. Results indicated improvements in shear and tensile properties by adding low volume fraction (<5%) of fibers. However, the fiber addition did not improve the compressive properties of the syntactic foams. It was also observed that, addition of higher volume fractions of fibers did not improve any of the properties, rather decreased the compressive properties of the foams. Ultrasonic imaging techniques showed the presence of a greater number of voids in foams with higher volume fractions of fibers, so that degrading the mechanical properties of the foams. In addition, micro-sized fibers in general soften the matrix and have poor interfacial interaction with the matrix.

Nanoclay has also been used to reinforce syntactic foams. Reports indicated that nanoclay significantly delayed crack initiation and growth and resulted in enhancement in tensile strength and toughness [10,11]. The increase in the strength was attributed to the reinforcing effect of the nanoclay particles. The incorporation of nanoclay resulted in larger amount of interface between nanoclay particles/platelets and the matrix thereby increasing the amount of energy required to de-bond the interfaces between the glass microballoons, nanoclay particles, and matrix resin. However, the inclusion of nanoclay decreased the tensile modulus as a result of a large increase in fracture strain and toughness of syntactic foams. In addition, increase in strength was accompanied with increase in the stiffness and brittleness, resulting in poor damage tolerance properties [12,13]. It was also observed that nanoclay syntactic foam structures showed lower load bearing capacity than

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that of the plain syntactic foam structures. This was due to the pronounced effects of shear stresses that resulted deformation and fracture of the syntactic foam in shear mode. In other report, addition of 2 vol.% of nanoclay decreased the compressive strength of syntactic foams [14]. When the volume fraction was increased to 5 vol.%, compressive strength was improved for lower density foams, whereas a decrease in strength was observed for the higher density foam. However, the compressive modulus was observed to decrease upon addition of 2 and 5 vol.% of nanoclay. In order to obtain better exfoliation of clay particles in the matrix, researchers have limited the nanoclay loading below 5 vol.%, with the minimum loading being 1 vol.%. The addition of such volume fractions of nanoclay affects the density of syntactic foams significantly. For example, addition of 2 and 4 vol.% of nanoclay increased the foams density by 17% and 26%, respectively [15]. From the report of another work, 3–11% increase in density with addition of 2 vol.% nanoclay was observed [10]. The inclusion of 5 vol.% nanoclay has also resulted in the increase of the density by 11–20%. Such increase in density of the syntactic foams could limit their use in light-weight applications.

Carbon nanostructures, carbon nanotubes (CNTs) and carbon nanofibers (CNFs) could be other potential reinforcing filler, because of their small size, high aspect ratio, and superior mechanical properties. In order for these fillers to influence the properties of syntactic foams, they need to be homogeneously dispersed in the matrix. Although several methods of dispersing the nanostructures have been explored, obtaining homogeneous dispersion has remained a fundamental challenge. In addition, these nanostructures are still very expensive to produce. Consequently, only few reports have been published on the use of these nanostructures to reinforce syntactic foams [16–18].

Graphene platelets (GPs) are novel potential fillers that can be used to reinforce syntactic foams. GPs are two-dimensional graphene thin plates containing few layers of graphene sheets. They have excellent performance characteristics compared to other carbon nanostructures. Their surface area is higher than other carbon nanostructures which can provide stronger filler–matrix adhesion, thus maximizing the stress transfer [19]. GPs are also currently being produced in bulk quantities with low cost [20]. These nano-materials have been used as fillers in polymeric composites to tailor the mechanical properties and produce high performance composites [19]. Comparative study on the mechanical properties of epoxy nanocomposites with GP, single-walled CNT (SWCNT), and multi-walled CNT (MWCNT) additives has been done at a filler weight fractions of 0.1 ± 0.002% [21]. Results indicated that GPs significantly out-perform CNT additives [21]. It was observed that addition of very low volume fraction of GPs improved the tensile modulus, tensile strength, fracture toughness, and fracture energy properties of the polymer composite compared to the plain (no-fillers) composite. The effect of these novel fillers on the mechanical properties of syntactic foams has not been studied yet. This paper discusses the mechanical characterization of syntactic foams reinforced with graphene platelets (GP-SFs). Compressive and tensile properties of GP-SFs at different volume fractions of GPs are evaluated and presented in this work.

2. Experimental

2.1. Materials and fabrication

The GPs used for this work were pristine and supplied by Cheap Tube Inc., USA. They had diameters of 1–2 μm, surface area >700 m^2/g, and purity of >99 wt%. GPs were first added in Toluene and sonicated for 5 min. The toluene/GPs suspension was added into epoxy (DER 332, DOW chemicals) and sonicated for additional 10 min. In order to remove the solvent, the mixture was kept in a degassing chamber at 60 °C for 15 h. After adding S38 glass microballoons (3 M Company, US) and the curing agent (DEH 24, DOW chemicals), the slurry was poured in silicone rubber molds. The samples were then allowed to cure for 24 h at room temperature and post cured for 3 h at 100 °C. Four sets of tensile and compression test samples containing different volume fractions of GPs were fabricated. All fabricated samples comprised of 30% by volume of glass microballoons. The dimensions of the samples for tensile and compression tests were respectively 147.5 × 28.5 × 7.5 and 24.5 × 24.5 × 13 mm. For the compression tests, specimen shape and size were determined according to ASTM C 365/C 365M-05; whereas, the guideline used for the tensile specimen geometry and dimension was ASTM D 3039/D 3039. Table 1 shows the volume fractions, weigh fractions, density, and porosity of the samples.

2.2. Characterization methods

Scanning electron microscopy (SEM) (FEI Quanta 3D FEG Dual Beam FIB/SEM) was used for failure analysis. Fracture samples were sputter coated with thin gold layer before imaging to make their surface conducting. SEM and transmission electron microscope (TEM) (JEOL JEM-1011 TEM) were used to study the GPs dispersion in syntactic foams.

Tensile and compression tests were conducted using QTEST 150 universal testing equipment. The compression test was performed at a crosshead speed of 0.5 mm/min. The samples were compressed to about 50% of their initial height. On the other hand, tensile tests were carried out at a constant deformation of 1 mm/min. The samples were gripped at opposite ends with a gage length of 50 mm. Load and displacement data obtained from the testing system was used to produce the stress strain plots. Tests were performed on at least five samples from each set of compression and tensile samples.

3. Results and discussions

One of the advantages of syntactic foams is their low density achieved due to the inclusion of hollow microballoons in a polymer matrix. Several reinforcements (glass fibers, carbon fibers, CNTs, etc.) have been used to improve the properties of syntactic foams. These reinforcements usually have higher densities as compared to the constituents used for fabricating traditional syntactic foams. Addition of high volume percent of these reinforcements may significantly affect the overall density of the syntactic foams and could limit their applications. Nevertheless, several reports indicated that only very low volume percent of reinforcements is required to bring improvements in the properties of syntactic foams. Accordingly, the maximum volume fraction of the GPs used in this study was limited to 0.5%. The densities of the fabricated syntactic foams were obtained experimentally by measuring the weight and volume of at least five specimens. The densities of the syntactic foams with increasing vol.% are listed in Table 1. It can be seen that no significant variation in the density is observed due to the addition of GPs.

Attempts to study the dispersion of the GPs in syntactic foams using SEM were not successful. Due to their planar geometry, only the edges of the GPs embedded in the matrix were exposed, and differentiating the platelet edges from the edges of fractured microballoons was extremely difficult. Although it was possible to distinguish the GPs from microballoon fragments using TEM (see Fig. 1a), it was challenging to accurately determine the GPs dispersion as the areal coverage of the TEM grid was too small and reliable statistics could not be derived. The TEM study,