STATIC AND DYNAMIC LOADING BEHAVIOR OF HYBRID EPOXY COMPOSITE WITH ALUMINA NANOPARTICLES

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Abstract

Particulate reinforcement in conjunction with 2 phase composites provides a unique behavior as a hybrid composite system. The interactions at the interface often lead to changes in the performance characteristics. Attempt is made to disperse spherical alumina particulate reinforcement with a 110nm mean diameter, into conventional fiber reinforced plastics (FRP's) of epoxy and fiber glass. Alumina particles are also treated using tris-2-methoxyethoxy vinyl silane (T2MEVS) to attain functionalization for better adhesion. Mechanical tests conducted revealed notable changes from the conventional composites as seen from the double cantilever beam tests. Tensile tests and fatigue tests are also performed to investigate the static and dynamic behavior of these hybrid composites.

1 Introduction

Hybrid composite material systems that consist of traditional laminate composite reinforced with alumina nanoparticles are fabricated via advanced material processing techniques. Mechanical behavior of composite material systems is highly dependent not only on their constituents but also on the geometric interactions of the constituent materials. The recent activities in the research and development of polymer nanocomposites provide a new class of matrix for conventional laminate composites [1]. These matrix materials are however two phase, and will provide new challenges for integration with conventional composites. Various research work in polymer nanocomposites have largely focused on the use of layered silicates (clay), carbon nanotubes and nanofibers. While layered silicates are dispersed as platelets; carbon nanotubes and nanofibers as one-dimensional ropes, work on nanocomposites with inorganic compounds like spherical alumina are also being conducted but not as extensive as clay, carbon nanotubes and fibers. In both thermo-plastic and thermoset polymers, changes in mechanical properties has been reported for both clay and nano-sized carbon reinforcements. Ajayan et al [2] reported an increase of 20% and 24% in tensile and compressive modulus with the inclusion of 5%wt of carbon nanotubes in epoxy. Koutsos et al [3] reported a 20% increase in both Young's modulus and ultimate strength with multi-walled nanotubes reinforced epoxy, and an improvement of 50% in Young's modulus and 45% in ultimate strength was reported when a co-polymer was synthesized with epoxy resin. Clay polymer nanocomposites however showed 5.4% decrease for every 1% increase in the particulate loading in both compressive strength and flexural strength as reported by Akbari and Bagheri [4]. Interestingly Kornmann et al [5] reported a 50% increase in fracture toughness in epoxy reinforced with 10% weight composition of organosilicate clay.

In this present work, epoxy composites reinforced with S2 fiberglass fabric reinforcement and alumina nanoparticles are studied for their static and dynamic behavior. Pre-processing of alumina involves particulate functionalization to improve the interface bonding characteristics among the fiber and matrix constituents of the composite laminate. Dispersion schemes by grafting alumina particles in epoxy resin and on fabric reinforcement are employed [6]. Effect of particulate reinforcements is studied for both the non-treated and functionalized alumina. Functionalization of alumina nanoparticles is performed using a silane compound with organo-functional terminals. J Zhang et al [7] showed silane improved the interfacial shear strength and flexural strength of various resin systems with improvements ranging from 11 – 54% for the shear strength and 2.5 – 10.4% in the flexural strength. Gillespie et al [8] showed the effect of silane coupling agent on the interface strength of adhesively bonded alumina with reported increases of 83.3% for methacrylate functional agent and 117% for styrylamine functional agent. Fracture toughness studies of unsaturated polyester-alumina polymer nanocomposite for various alumina volume fractions by M Zhang and P Singh [9] showed improvements ranging from 23 – 133.3% for volume fraction ratios of 0.5 - 4.5% when functionalized alumina is used compared to the non-functionalized reinforced polyester.

Our earlier studies have established that alumina influence the performance of reinforced epoxy plastics and hybridized composite laminates [6]. Alumina weight composition of 1.5% of resin weight is used in these material systems. The material system configuration and processing methodologies employed are addressed briefly in the next section. The subsequent discussion focuses on comparative studies of mechanical characterizations as it relates to the static and the dynamic behavior of the hybrid epoxy composite system in this work.

2 Materials and Fabrication

2.1 Material Systems

Hybrid composites made up of epoxy, fiberglass fabric and alumina nanoparticles are fabricated by a two stage processing method. The initial stage is the integration of the nanoparticles in the primary materials (resin or fiber) and the second stage is the consolidation of a laminate composite by VARTM processing [10]. The initial stage involves the dispersion of particulate in the resin or the grafting of the alumina particles onto the fabric surface creating an interface between the fabric plies.

Alumina at 1.5% weight composition of resin mass is used in the hybrid composites formed. The added alumina particulates are untreated or functionally treated with tris-2-methoxyethoxy vinylsilane (T2MEVS) during processing prior to dispersion in the resin or atomized spray coated onto the fabric surface. In all, five different hybrid composite material configurations are fabricated. Variations are based on the particulate alumina dispersion scheme and alumina functionalization. Material nomenclature for different hybrid composite material configuration used in the static and dynamic studies is shown in Table 1.

Table 1: Nomenclature of material system configurations

Material	Configuration
MAT 1	Baseline: EPON™ S2 Glass
MAT 2	Resin Modification with pristine Alumina
MAT 3	Resin Modification with Functionalized Alumina
MAT 4	Fiber Modification with pristine Alumina
MAT 5	Fiber Modification with Functionalized Alumina

Hybrid composite panels were fabricated for these above material system configurations for the tensile, double cantilever beam and fatigue tests. The composite panels for double cantilever beam tests were fabricated with a Teflon insert to provide an initial crack size of 50.8mm as shown in figure 2.

The following sub-sections briefly discuss the processing methodologies employed in the dispersion of alumina particulate reinforcement in the hybrid composite material systems.

2.2 Resin Modification

Laminate hybridization preprocessing is carried out by dispersion of alumina nanoparticles (functionalized and non-functionalized) in the neat resin. Functionalization is achieved using tris-2-methylethylvinyl silane (T2MEVS). Exfoliated and intercalated dispersion is obtained by sonication at 20 KHz and 500watts. Laminate consolidation is achieved by resin infusion through vacuum assisted resin transfer molding (VARTM) using the modified resin [10].

2.3 Fiber Modification

Alumina particles are laced onto interfacing fabric plies for the fiber modification during processing of the hybrid composites. Atomized spray of functionalized and non-functionalized alumina particles are grafted onto interfacing plies to achieve an exfoliated and intercalated dispersion on fabric surface. Alumina is pre-dispersed in polar solution and then sonicated before grafting onto the fabric surface. Functionalization is achieved by treating alumina with T2MEVS before atomized spraying onto the surface of the interfacing fabrics. Laminates are then fabricated by conventional VARTM processing using the modified fabric layers and neat resin [10].

3 Results and Discussion

The following sections discuss the static and dynamic tests performed characterizing the behavior of epoxy and hybrid composite systems in this work.

3.1 Static Behavior: Tensile Tests

Tensile tests were conducted to the specifications of ASTM D3039 standards [11]. The specimens of all fabricated material systems are cut to ASTM specifications shown in figure 1.

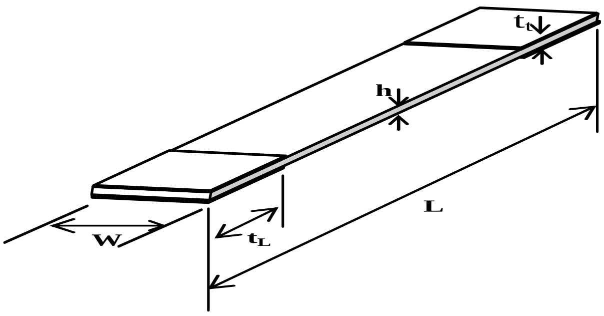

Figure 1: ASTM D 3039M – 0 Tensile test Specimen (L = 330mm, W = 25.4, h = 3.175mm, t_L = 50.8mm and t_t = 6.35mm)

The sample size of the coupons used in the tests for each material system configuration shown in Table 1 was ten. Test results for tensile tests are shown in table 2. The tests results show that no significant changes were observed in the ultimate tensile strength and Young's modulus. All reported results are within the same margin of variation. However a slight increase in ultimate tensile strength was observed for the fiber modified hybrid composite with functionalized alumina.

Table 2: Tensile test result for hybrid composite materials

	Y. Modulus (GPa)	Std Dev.	COV	UTS (MPa)	Std Dev.	COV
MAT 1	25.016	1.249	4.990	569.458	21.168	3.718
MAT 2	25.927	1.510	5.825	559.239	27.097	5.892
MAT 3	25.592	0.892	3.485	512.361	38.019	4.951
MAT 4	24.717	1.937	7.838	513.98311	39.6799	0.077
MAT5	26.031	2.057	7.902	536.97398	33.4259	6.225

The hybrid epoxy composites with alumina nanoparticles did not show much improvement in the mechanical properties via tensile tests. This can be attributed to the dominance of the directional strength of the fiber in all hybrid composite material systems fabricated. The results obtained here indicate that influence of particulate reinforcements could be potentially seen in performance characterization tests where the resin system and the interaction at the resin and fiber interfaces exist.

3.2 Static Behavior: Double Cantilever Beam Tests

Mode I inter-laminar fracture toughness tests were performed to the specifications of ASTM D5528-94a [12] for the five material configurations shown in Table 1. Test coupons with Teflon insert are cut to ASTM test standard shown in figure 2.

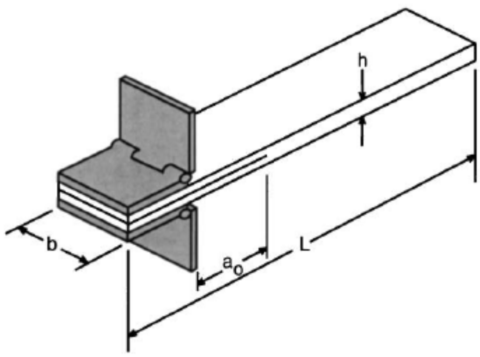

Figure 2: Double Cantilever Beam test specimen with Teflon inserts (L = 200mm, b = 25.4mm, h = 4mm and a_o =50.8mm)

Fracture toughness values for the mode I interlaminar tests were obtained from these double cantilever beam test results. Table 3 shows the fracture toughness values obtained for the five different material configurations.

Table 3: Fracture toughness of hybrid composite material systems in J/m^2

	Average	Std. Dev	Max	Min
Mat 1	347.12	82.28	483.33	273.81
Mat 2	377.87	19.87	410.94	360.84
Mat 3	411.94	60.88	457.87	313.28
Mat 4	398.03	64.85	481.45	328.35
Mat 5	448.01	83.62	543.98	345.78

These results show an improvement over the baseline material for all the hybrid composite material configurations. Improvements due to functionalization of alumina can also be inferred from these results. All hybrid composite material configurations showed improvement in fracture toughness compared to the baseline. An improvement of 8% and 12% in the fracture toughness over the baseline material system is observed for the non-functionalized alumina dispersed resin and non-functionalized fiber modification. The functionalized resin and fiber modification shows an increase of 18.4% and 29.4% respectively in the fracture toughness values compared to the baseline material. Functionalized alumina in modified resin (MAT 3) also shows a 9% increase over unfunctionalized modified resin material (MAT 2). A 12% improvement was also noted in functionalized alumina (MAT 5) against the unfunctionalized alumina (MAT 4) with fiber modification**.**

3.3 Fatigue Life

Fatigue characterization of the material systems is performed based on ASTM D3479 standard specifications. The test coupons are sized to ASTM 3039 standard shown in figure 1. The total life prediction and crack damage accumulation [12] approaches are employed for the behavior characterization with the dynamic fatigue tests.

Tension-tension fatigue tests are conducted at a single frequency of 2 Hz with continuous loading for damage accumulation in the composite material system. Stress loading ratio is maintained at 0.1. Maximum stress loading at 90, 80, 70, 60, 50, 40, and 30% of the ultimate tensile strength (UTS) were employed in various tests. These fatigue tests employed the baseline material system and the fiber modified material system with functionalized alumina. Functionalized fiber modified material system was selected for this study since it had the highest increase in the fracture toughness value based on mode I interlaminar tests discussed in section 3.2.

Semi-log plots were obtained from the test data for the fatigue life plots until fatigue failure by fiber breakage were observed. Three different curve fitting methods are used to determine a predictive tool and a characteristic fatigue model for these material systems. The curve fit models investigated include the following:

1. Logarithmic: The logarithmic curve fit is commonly used in non-linear monolithic material systems and has shown to describe approximate curve fit for the composites [13].

$$Y = A Log(x) + B$$

2. Power Law: The power law curve provides an approximation for coalescing multi-stage, multi-linear curve fitting of the fatigue test data into a single curve. This provides a generalized model and is gaining a lot of use in composites with correlation coefficients near unity [13]. The power law curve is given by:

$$Y = Ax^k + B$$

3. The Sigmoidal curves (reverse S-shaped): The S curve fit are frequently used in biology to describe growth patterns. The sigmoidal curve is the reverse S curve. In this work, the "Logistic" Sigmoidal curve is applied to the fatigue test data of the composite materials tested. The Boltzmann Sigmoidal curve has shown good fit in braided composites as reported by Kelkar et al [14]. The Logistic Sigmoidal fit used is given by:

$$Y = \frac{A_1 - A_2}{1 + \left(\frac{x}{x_0}\right)^p} + A_2$$

where, $Y = \frac{S}{S_{uts}}$, $x = \log_{10}(N)$, A, k, A_1, A_2, x_o, and p are the parameters in the equation.

Figure 3 shows the fatigue model for the baseline material system while figure 4 shows the fatigue life model for the fiber modified hybrid composite system with functionalized alumina. All three fatigue fit model parameters are shown in these figures.

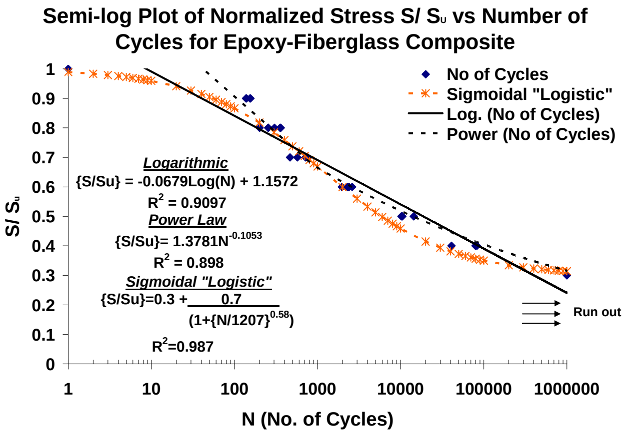

Fig. 3: Semi-log plot of normalized stress against number of cycles to failure for epoxy-S2 fiberglass laminate (MAT 1)

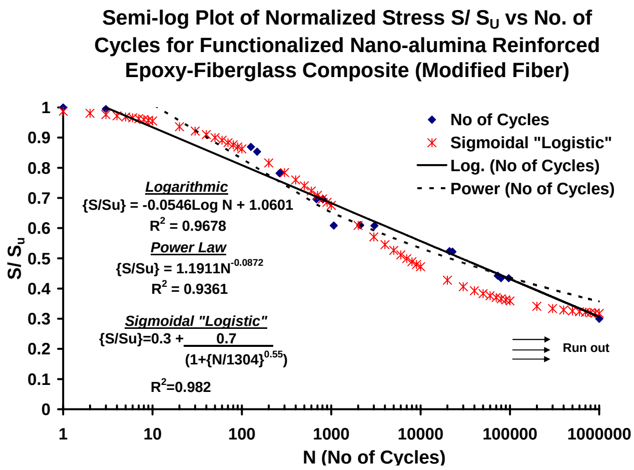

Fig. 4: Semi-log plot of normalized stress ratio against number of cycles to failure for nano-alumina (functionalized) reinforced epoxy-S2 fiberglass laminate (MAT 5).

Figures 3 and 4 show the fatigue curve fit profile and models for the conventional epoxy composites and the hybrid epoxy composites with alumina nanoparticles respectively. The results and fatigue life model fitting parameters show that alumina hybridization via fiber modification did not have any significant effect on the fatigue life of the formed composite. The correlation coefficients for all the fatigue model curves provide a reasonable fit. The coefficient constants of the fatigue curve models for conventional composites and hybrid composites show that there is no significant effect due to hybridization of fiber epoxy composite with alumina nanoparticles.

Though a significant difference did not exist among the different model fits, sigmoidal model shows a best fit with a correlation coefficient of 0.987 for the baseline epoxy composite and 0.982 for the hybrid epoxy composite compared to other models.

4 Concluding Remarks

Hybrid epoxy composites with alumina nanoparticles were obtained through particulate hybridization via resin and fiber modification in conjunction with laminate consolidation through VARTM processing. Hybrid composites were formed by dispersing the alumina particles in the neat resin or by grafting alumina nanoparticles onto the fabric surface. The mechanical behavior of the formed material is investigated to understand and characterize the influence alumina nanoparticles on the damage mechanism of the hybrid material system.

Investigations have shown improvements of 8% and 12% in the fracture toughness for resin and fiber modification respectively over traditional epoxy composite. Further improvements in fracture toughness studies are achieved when alumina particles are functionalized with an increase of 18% and 29% over the traditional epoxy composite for the resin and fiber modifications respectively. The results did not show any significant effect of the particulate reinforcement of the hybrid composite material system under tensile and fatigue loading. These results are consistent with of the reported behavior in hybrid composites formed with carbon nanotubes/ carbon fabric/ epoxy which showed an increase in the interlaminar shear strength but did not show any effect on the tensile properties [15].

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