MICRO-HYSTERESIS AND DAMAGE DETECTION IN NOTCHED COMPOSITES

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Abstract

While carbon fiber composites have excellent mechanical properties, a much lower cost alternative exists in glass fiber composites. The main drawback of glass fiber composites, however, is their poor performance in fatigue. Mechanisms of fatigue failure consist predominantly of matrix-dominated damage accumulation and growth that propagates into the fibers resulting in ultimate fatigue failure.

Incorporating multi-walled carbon nanotubes (CNTs) into the matrix tends to inhibit the formation of large cracks due to the large density of interfaces provided by the CNTs and CNT bridging across nanoscale cracks. The resulting distributed nanocrack growth slows damage propagation resulting in a low-cost material possessing improved fatigue strength and durability.

1 Introduction

Prior fatigue studies of glass composites have shown that fatigue life is dominated by fatigue cracking in the matrix that subsequently propagate and rupture the fibers. Once a significant number of fibers fracture, the composite laminate fails shortly thereafter since the fibers carry most of the load [1-3]. A recent review of fatigue theories is given in [4]. Crack propagation and the resulting loss in stiffness is a gross overall symptom of damage in composites. Damage can, however, be generated well before microscopic-level crack initiation and propagation occurs. Other studies have concentrated on determining the fracture energies involved in crack propagation in composites, particularly in delamination fracture, a common failure mode [5]. Such studies show that a single crack that propagates in the matrix or fiber/matrix interface in a composite is associated with a low level of absorbed fracture energy.

In typical composite laminate configurations designed to carry structural loads under cyclic conditions, carbon composites show little degradation with load cycling compared to glass composites. This observation is at odds with delamination fracture energy measurements that show that carbon composites require an order-of-magnitude *lower* energy to propagate relative to glass composites. The explanation for the low degradation rates for carbon composites lies in the fact that the modulus of carbon fiber is much higher than that of glass fiber thereby restricting the cyclic strain imposed on the matrix. This explanation has been verified recently on oriented “rope” CNT-reinforced epoxy composites. The fatigue strength did not change with cycling in a manner similar to that shown by conventional carbon fiber composites [6].

Other studies on the effect of CNTs fracture behavior of the matrix polymer have shown that small additions of CNTs (0.1 – 0.2 weight %) resulted in a 40% increase in the fracture toughness of the polymer [7]. Microscopy studies show a very high density of nano-scale cracks in the matrix that are attributed to the improvement in the fracture toughness of the matrix polymer.

There have been, however, no studies on the effect of CNTs on composite strength when subjected to cyclic loading. While the effect on modulus has been shown to be small due to the low volume fraction of the CNT content (0.1 to 1% range) [8], the presence of CNTs can be significant in restricting damage. In this work, we investigate the effect of carbon nanotubes (CNTs) on the life of structural composites subjected to static and cyclic loading. Damage mechanisms in conventional composite laminates consist of the formation of micro-cracks in the matrix that initiate and propagate under cyclic loading, eventually causing fiber failure and fracture of the composite. The addition of CNTs is expected to decrease the scale of damage mechanisms by several orders of magnitude resulting in an increase in the absorption of strain energy through the creation of a multitude of fine nano-scale cracks. In addition, fiber bridging at the nanoscale increases energy absorption through the participation of nanotubes in the fracture process. This effect should increase the damage tolerance of the composite and make it more resistant to damage growth under cyclic loading.

We have incorporated functionalized thermosetting (epoxy) polymers containing uniformly distributed nanofibers (CNTs) into conventional glass fiber composites. Our recent efforts have established that these resins exhibit a relatively uniform distribution of the nanofibers in the polymer with little agglomeration and clumping. This is important for processing our proposed nanofiber-modified composites. The failure mechanisms in composite systems that contain CNTs, both under static and cyclic loading were examined and crack propagation mechanisms determined both in the neat polymer matrix and in the composite.

2 Materials, Processing, and Experimental Methods

2.1 Materials

The thermosetting polymer resin/hardener used in this study was EPON 826 / Epikure 3234, both manufactured by Hexion Specialty Chemicals Inc. (Houston, TX). The EPON 826 resin was functionalized with 1% by weight of multi-walled carbon nanotubes by Nanoledge (Clapiers, France). The fiberglass used was Hexcel 7500 (Fullerton, CA), a 0.28 mm thick plain weave fabric.

2.2 Composite Processing

Both the CNT and non-CNT functionalized [0/90] fiber reinforced composites were manufactured using a wet layup method. Eight plies of fiberglass fabric were wet with the mixed and degassed resin/hardener pair. The residual resin was removed, and the epoxy was cured through the use of a heated platen press held at 80°C and 580 kPa for one hour.

24 x 200 mm tensile specimens were cut from the cured sheets using a diamond blade wet saw. A 6.4 mm hole was drilled in the center of each specimen to create a stress concentration to ensure that the specimens would not break in the grips of the testing machine. Aluminum tabs were also bonded to the ends of the specimens to reduce the chance of breaking in the grips. The specimens were aged for 10 days at around 20°C before testing.

2.3 Experimental Methods

Specimens were tested until failure using an MTS servo-hydraulic testing machine, digitally controlled by an Instron Labtronic 8400. Data acquisition was performed using Labview with a sampling period of 5 ms.

Prior to fatigue testing, the monotonic tensile strengths of both CNT and non-CNT functionalized composite samples were established for use as reference points when determining the loads to apply in the fatigue tests.

Both types of samples were then tested in tension-tension fatigue at a maximum stress of 70, 60, and 30% of their monotonic strengths, all with R-values of 0.15. The loading frequency used was 3 Hz to eliminate sample heating.

Representative specimens were chosen and their fracture surfaces were excised and coated with a 2.5 nm layer of Pt using a Bal-Tec Med 020 sputter coater. The samples were then imaged using a Hitachi S-5000 cold field emission SEM with an accelerating voltage of 10 kV.

3 Results and Discussion

3.1 Fatigue Life

The fatigue life data for the specified loading conditions can be seen in Figure 1. A significant increase in the number of load reversals to failure for each loading case was observed for the samples that contained the CNT functionalized resin. This increase in fatigue life is attributed to the distributed nanocrack growth as well as the crack bridging that occurs as a result of the incorporation of CNTs into the matrix.

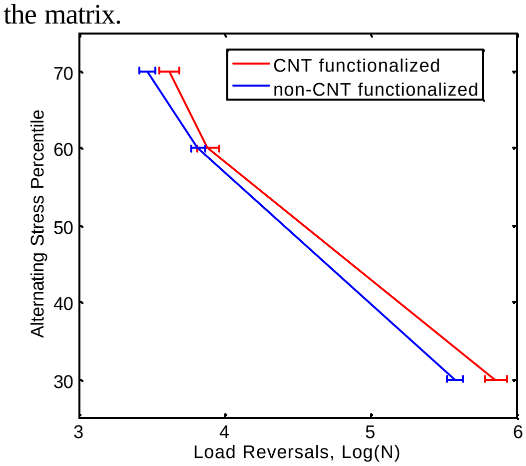

Fig. 1. S-N curve for CNT and non-CNT samples

3.2 Hysteresis

Hysteresis per cycle has recently been shown to be a low-cycle predictor of overall fatigue life in composites [9]. Figure 2 shows two representative samples that were cycled at 70% of their monotonic strengths. There is a noticeable decrease in the slope of the hysteresis per cycle vs loading cycles in the specimen containing CNTs relative to a specimen without CNTs. Final failure, characterized by a rapid

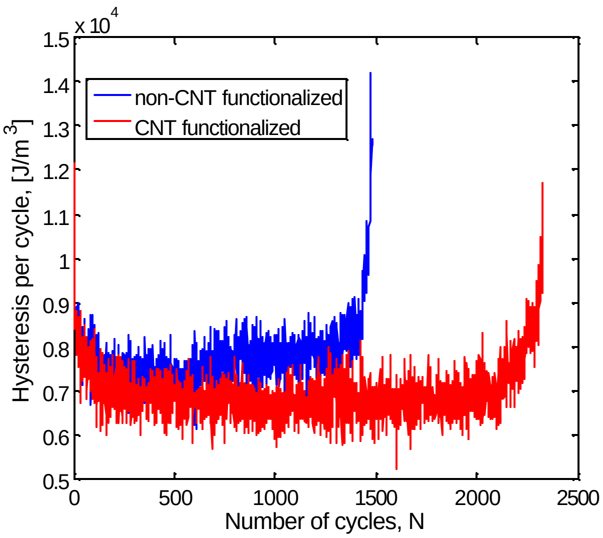

Fig.2. Hysteresis per cycle for CNT and non-CNT functionalized samples

increase in hyteresis per cycle, also occurs significantly earlier as shown in the figure.

3.3 Fracture Surface Analysis

Through use of high-resolution scanning electron microscopy, the fracture surfaces of the samples have been im aged for analysis. Figure 3 shows a large number of individual CNTs protruding from the fracture surface. It is believed that the longer of the tubes have pulled out from the opposite fracture surface while the shorter of the tubes broke at or near the interface. A single hole (white arrow) corresponds to a tube that was pulled out from this surface and left embedded in the opposite surface. It is this process of tube pullout and tube fracture that is believed to contribute to the increased fatigue resistance of the CNT functionalized composites.

Also of interest is the distribution of CNTs in the matrix. Figure 4 shows a representative region that contains an even distribution of CNTs. However, there were areas with relatively large entanglements of CNTs that were not wet by the polymer matrix (white arrow, inset). As in [10], it is believed that these large entanglements are detrimental to the overall strength and fatigue life as they behave effectively as voids rather than as reinforcement.

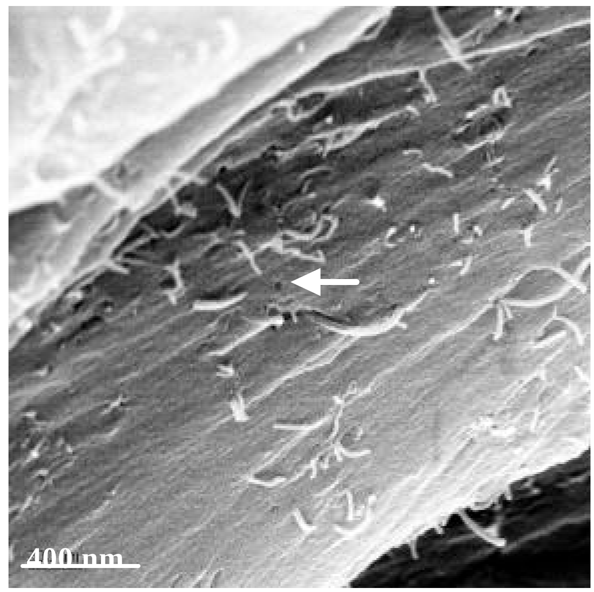

Fig. 3. SEM micrograph showing fractured and pulled-out CNTs

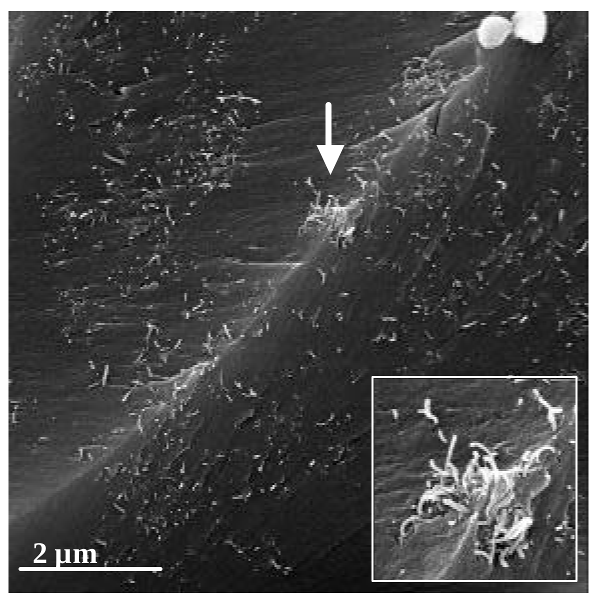

Fig. 4. SEM micrograph showing the CNT distribution and large entanglements

4 Summary and Conclusions

The effect of the addition of CNTs to the polymer matrix of fiberglass composites has been studied. It has been found that these new composites show promise as a means of improving the otherwise poor fatigue performance of fiberglass structures.

At low stress levels, the CNT functionalized composites had lives approaching twice that of the non-functionalized composites. At higher stress levels the effect of CNTs exhibited and increase in fatigue life of 40%. In addition, composites containing CNTs exhibited a lower level of hysteresis per cycle and a lower rate of increase of hysteresis per cycle with cycling. Correlation between the slope of the hysteresis per cycle vs number of cycles curve and overall life has been suggested earlier for non-CNT based composites.

SEM micrographs showed the physical interactions between CNTs and the polymer matrix, exhibiting CNT breakage and pull-out as two processes through which crack propagation in the matrix is impeded, leading to reduced hysteresis losses per loading cycling and corresponding increased fatigue life.

5 Future Work

Monitoring hysteresis per cycle has been shown to be a sensitive indicator of damage early in the cyclic history of composites. In planned future studies, the effect of CNT additions in increasing fatigue life will be studied for a variety of lay-ups and composite hybrid fiber combinations. The aim is to develop a methodology for the design of fatigue-resistant low-cost composites that contain small fractions of nanofibers.

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