The loading rate effect on Mode II fracture toughness of composites interleaved with CNT

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Abstract The loading rate effect on Mode II interlaminar fracture toughness (ILFT) is examined in this study with interleaved epoxy/carbon fabric laminates tested under dynamic conditions. Specifically, an SP1 protein-treated carbon nanotube-reinforced epoxy leaf is inserted at the midplane of the laminates, and the fracture properties are measured by the crack lap shear method at two different loading rates. Whereas our preliminary study performed under quasi-static conditions showed that this specific interleaving generated an ~85% improvement in the Mode II ILFT, the current work shows that the occurrence and magnitude of the improvement depend on the loading rate and crack velocity, with different effects on the initiation and the instable/stable propagation stages. Improvements in Mode II ILFT for both the crack initiation and propagation phases can reach up to ~145% for certain dynamic loading conditions.

Keywords Nanocomposites, Carbon nanotubes, Fracture toughness, High-rate loadings, Delamination, Interleaving

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Introduction

One of the common failures in laminated composite materials is delamination caused by microcracks in the laminated structure that grow under a sufficiently high shear stress. Improvement of the mechanical properties in general and particularly of the interlaminar fracture toughness (ILFT) is one of the most investigated issues in the field of composite materials. Delamination of composite structures under Mode I (opening mode of failure where the stress is perpendicular to the crack) or Mode II (shear mode of failure where the stress is in parallel to the crack) loadings is frequent and has been studied extensively, wherein different configurations of the edge notch fracture (ENF) test are utilized in opening and shear modes, respectively. A limitation of this test is that it is often performed under slow loading in quasi-static conditions that do not simulate adequate practical scenarios. In fact, the service conditions of many composite structures, such as those that are used in aircraft, marine, automobile, and sports industries, generate fatigue-related behavior and dynamic crack growth.

Over the years, different solutions have been utilized for the delamination problem, working to increase the fracture toughness of laminated composites under a shear stress field. In many of them, the increased delamination resistance has been accompanied by a deterioration of other mechanical properties, such as strength and stiffness. More recently, with the remarkable progress in nanotechnologies, minute quantities of different types of nanoreinforcement, e.g., CNT, have been mixed into the polymer matrices to produce improved ILFT without a penalty of reduction of other essential mechanical properties and with no weight gain.

Accordingly, in our preceding study, we have applied a modified interleaving technology, based on the original concept of placing a thermoplastic polymer leaf at the midplane of the laminated composite. The guiding concept of our study was that nanoparticles for improved fracture toughness should be placed specifically at critical zones of high stress concentrations, e.g., regions of potential delamination, instead of dispersing them wastefully throughout the matrix. Thus, a thin leaf of epoxy/protein-treated carbon nanotubes (f-CNT) was inserted in the midplane of a carbon fabric–epoxy composite laminate (see Fig. 1), generating a remarkable improvement in the ILFT without deteriorating the static properties. Essential details of the SP1 protein can be found in the literature.
Because the ability to resist crack propagation in composite materials depends on the interfacial properties of the matrix and fibers, the environmental conditions and the loading rate and mode, an effective test method is required, which is more sensitive than the classic ENF test to the broad range of such factors that affect fracture toughness.8 Regarding the rate factor, there are many studies in the literature of the behavior of polymer composites under dynamic conditions, which cover different materials, test specimens, and modes of loading.9–15 The diversity of those studies and of their results indicates that such studies ought to be case specific; hence, every material and testing configurations must be studied particularly.

In view of that, the present research was undertaken to broaden the scope of our previous work6 – on Mode II fracture toughness of a CNT interleaved laminate (described in Fig. 1) – to study the effect of loading rate. However, unlike the studies on rate effects cited above, where conventional test methods (such as Mode II center-notch-flexure under impact loading, or end-notch-flexure under different loading rates or under drop-weight impact), here the crack lap shear (CLS) test under extremely controlled rate and failure mode conditions was employed.

**Experimental section**

**Materials and preparation of samples**

Prepregs of J.D. Lincoln, INC, product name L-930 were used. The prepregs are based on a carbon fiber fabric (50–70 wt.%)/bisphenol A/epichlorohydrin-based epoxy resin with a ply thickness of approximately 0.25 mm. Epoxy LY1556 with anhydride hardener HY917 and accelerator DY070 was chosen as the matrix for the interleaf.

Multi-walled carbon nanotubes C-100 (CNT) (Arkema), functionalized in a non-covalent mode by SP1 protein with a maximal CNT-to-protein weight-to-weight ratio of 20:1 (e.g., t-CNT), were supplied by SP Nano Ltd. Additional information on the SP1 protein and the CNT treatment process can be found in7. The wt.% of t-CNT in the epoxy matrix was 0.5 wt.%, wt.% of CNT pristine in the epoxy matrix was 0.5 wt.%, and of SP1 0.025 wt.%.

One gram of t-CNT or CNT powder was added to 100 g of LY1556 epoxy. Dispersion by manual mixing was followed by probe sonication using Hielscher Ultrasonics processor UIP1000 (1000 W) for 10 min.

The specimens were prepared by stacking of twenty-two layers of prepreg fabric in a [0/90] configuration into 150 × 150 × 4 mm laminates as presented in Fig. 1. In order to form an artificial initiation crack, a Teflon film of 15 mm width and 20 μm thickness was inserted in the laminate between the 11th and 12th plies (at the midplane) as shown (Fig. 2a).

The non-cured interleaves, which were prepared by mixing of all the compounds (LY1556 pure or with the t-CNT/CNT pristine/SP1 protein addition/HY917/DY070) in a separate flask, were introduced utilizing small brush at the midplane of the laminate.
lamine from edge to edge (Fig. 1). Curing was performed under a pressure of 1.7 MPa at 130 °C for 30 min. The laminate was cut to the specimens of dimensions 150 × 10 × 4 mm. Five different types of laminates were prepared. The laminates were labeled according to the interlayer type as summarized in Table 1.

**CLS test**

CLS test was performed in order to examine Mode II (shear) fracture behavior of the composite materials, although usually this kind of test is used in order to calculate mixed-mode (I and II) fracture toughness. The specimen for CLS test is presented in Fig. 2a and consists of 2 parts: lap and strap. During the loading (tension), the crack propagates between lap and strap, while strap is fixed at the lower extremity, as shown in Fig. 2b.

**High strain-rate tensile tests**

High strain-rate tensile tests were conducted using servo-hydraulic machine at different strain rates until the total failure of composite specimen. The test machine is equipped with a launching system. The composite specimen is positioned between the load cell (upper extremity) and the moving device (lower extremity) as presented in Fig. 2b. Prior to the contact between the sliding bar and the hydraulic jack, the latter one is accelerated over a straight displacement of 135 mm in order to reach the nominal crosshead velocity before loading begins. Once the contact occurs, the specimen is then subjected to a tension at a constant load rate. The damping joint placed between the slide and the hydraulic jack may attenuate partially the wave effects caused by the dynamic shock.

In order to prevent the mixed mode of failure of the composites and ensure the maximum shear failure, special ‘arms’ were used (Fig. 2b) which suppressed the Mode I component during the loading.

Ultra high-speed camera was used during each test to record all specimen failures and calculate crack extension and speed.

The CLS specimens were loaded using servo-hydraulic machine at load rate of 4 and 8 m/s. Load–displacement records were obtained during the test, and the crack front location was recorded during crack propagation. The energy release rate ($G_{II}$) was calculated according to16–18:

$$G_{II} = \frac{P_c^2}{2b^2Et}$$  \hspace{1cm} (1)

where $P_c$ is the critical load for crack initiation/propagation, $b$ is the specimen width, $E$ is the laminate modulus, and $t$ is the total thickness.

The $G_{II}$ values for initiation were calculated from the critical $P_c$ value at the onset of pre-crack propagation.

**Morphology characterization**

The fracture morphology of the specimens and t-CNT exfoliation in the epoxy matrix was characterized utilizing a high-resolution scanning electron microscope Sirion, and the operating voltage was 5.00 kV. The samples were coated with an Au–Pd nano-layer using a SC7640 Sputter.

**Results and discussion**

We have recently shown that under quasi-static conditions by interleaving of a protein-treated CNT/epoxy layer at the midplane of a carbon fabric-reinforced epoxy laminate, the Mode II ILFT of the composite material is increased by 85% (~3.48 kJ/m$^2$) compared to the control laminate without an
interleaf (~1.88 kJ/m²). In view of the results under quasi-static conditions, it became appropriate to broaden the study to include dynamic conditions that are more relevant to typical service conditions of such structures. Also, as pointed out above, dynamic measurements might have an additional benefit by uncovering new characteristics of the composite material – not seen under quasi-static loadings.

As previously stated, we focus on the t-CNT sample in comparison with four reference samples that consist of interleaves based on the separate constituents of the t-CNT leaf.

Figure 3 presents a typical force–time curve as received during the dynamic Mode II loading (the specific curve is for control specimen loaded at crosshead speed of 8 m/s).

Figs. 4 and 5 present the plots of $G_{II}$ as a function of the crack extension and velocity at two different loading rates – 4 m/s (Fig. 4a and b) and 8 m/s (Fig. 5a and b), respectively – of the five samples that have been examined (Table 1). The results represent testing of two similar sets. The plots constitute the crack growth resistant curves (R-curves) that characterize the points of fracture initiation and the regions of stable (linear) and unstable (non-linear) crack growth prior and beyond the dotted lines, respectively, as shown in Fig. 4a and 5a. The points where the vertical line intercept the traces (the instability points) are related to the fracture toughness of each composite laminate, which is an intrinsic feature of the material.

First, considering the onset of fracture, it is seen that the t-CNT interleaved laminate exhibits higher $G_{II}$ of ILFT of initiation values: at a loading rate of 4 m/s, the fracture resistance increases from ~1.5 kJ/m² (control) to ~1.75 kJ/m² (t-CNT), which is ~17% improvement (Fig. 4a); and at a loading rate of 8 m/s, the initiation $G_{II}$ increases from ~0.22 kJ/m² (control) to ~0.54 kJ/m² (t-CNT), which is ~145% improvement (Fig. 5a).

Generally, as can be seen in the graphs of Figs. 4 and 5, the fracture resistance increases as the crack extension/velocity increase for all the samples. There is, however, a number of noticeable differences related mostly to the loading rates. Under the lower loading rate (Fig. 4), the crack instability point is reached at around a crack extension of 0.03 m, beyond which $G_{II}$ approaches a plateau. Below and at the instability point, a small improvement (~17%) of the ILFT $G_{II}$ from ~1.5 kJ/m² (control) to ~1.75 kJ/m² (t-CNT) is observed; thereafter, the control sample exhibits the highest fracture toughness values throughout the crack extension/velocity range. Under the higher loading rate (Fig. 5), the crack instability point is reached at a significantly higher crack extension of about 0.10 m, beyond which $G_{II}$ continues to increase monotonically. Here, the t-CNT interleaf contributes to a dramatic increase in the fracture toughness of about 70% compared to the control along the whole crack extension/velocity range. Obviously, the loading rate affects the fracture resistance through its effect on the crack stability and velocity.

The straightforward expression of crack stability is its velocity. Looking specifically at the $G_{II}$-crack velocity relationship (Figs. 4b and 5b), we see that under the two loading rates (4 and 8 m/s), the crack velocity increases – and so do the $G_{II}$ values – as the initial crack propagates, until a steady-state plateau (that is more distinct at 4 m/s loading) is attained. The crack extension starts at a relatively low velocity and as the fracture process proceeds the crack velocity grows. Under dynamic loading, the crack initiation occurs before the critical fracture load is reached – unlike the case of quasi-static loading, where the crack initiates at maximum loading, as in 6. At a loading rate of 4 m/s, the maximum fracture toughness value of $G_{II} \approx 3$ kJ/m² is reached at a crack velocity of 1000–1700 m/s, while at a loading rate of 8 m/s, the maximum $G_{II}$ value is higher – ~3.5 kJ/m², (for the t-CNT sample), and is attained for a crack velocity over 2500 m/s, which is below the expected steady-state plateau. Based on the quasi-static $G_{II}$ values, it is estimated that under quasi-static loading, the initial crack velocity at fracture onset is around 3000 m/s.
between the CNT and the carbon fibers of the fabric; thus, the adhesion between the epoxy matrix and carbon fibers increases. As the adhesion between the matrix and the fibers increases, the cohesive failure is promoted which ensures the increase in the $G_{II}$ for crack initiation. In addition to the cohesive failure, there is an interfacial failure that is not less important because it is required for the formation of bridging CNT that constitute the main energy absorption mechanism in composites. Actually, the optimal combination of both cohesive and interfacial failure provides the best value of ILFT of the composite material. The CNT bridging mechanism can be clearly seen in Fig. 7 that reveals the significance of t-CNT addition to the midplane of the laminate, with reference to the other interleaves (epoxy, epoxy-CNT, and epoxy-SP1), where no significant improvements or no improvement at all has resulted.

Fig. 7 presents the fracture surfaces of two different specimen types: the t-CNT (Fig. 7a and b) and the CNT (Fig. 7c and d) specimens at a loading rate of 8 m/s. As was already mentioned, the bridging mechanism, where the t-CNT binds to the carbon fiber on the one hand and to the epoxy matrix on the other, is seen in Fig. 7b, implying that the bonding between the three components – epoxy matrix, t-CNT, and carbon fiber – is effective. For the untreated CNT specimens, however, Fig. 7c and d shows that the dominant fracture mechanism is pullout, as seen by both the protruding CNT and the corresponding cylindrical holes in the matrix (both marked with circles). This emphasizes the crucial binding capability of the SP1 protein.

Considering the loading rate effect, Fig. 6 presents plots of crack velocity against crack extension for the t-CNT and the control samples under the two loading rates, where it is clearly seen that at the same crack extensions, the crack velocity under the 8 m/s loading rate is notably higher. Based on all these observations, it is concluded that fracture toughness is significantly higher at high crack velocities, where it becomes more sensitive to energy dissipation mechanisms of crack front interactions.

To identify the energy dissipation mechanisms and as fracture toughness correlates with fracture surface features, a SEM comparative fractography was performed.

First, we refer to the improvement of the ILFT in laminates with incorporated t-CNT leaf attributed to SP1 protein treatment of the CNT, shown to have created strong interactions between the t-CNT and epoxy and t-CNT and carbon fibers. These interactions result from the presence of graphite-specific binding peptides on the SP1 N-terminus originally engineered to react with the CNT. So, the SP1 protein of the SP1-CNT assembly – in which on the one hand the protein binds to the CNT to form a complex and on the other it bonds covalently to the epoxy matrix – also binds to the carbon fiber. In fact, the SP1 protein plays a role of a coupling agent between the CNT and the carbon fibers of the fabric; thus, the adhesion between the epoxy matrix and carbon fibers increases. As the adhesion between the matrix and the fibers increases, the cohesive failure is promoted which ensures the increase in the $G_{II}$ for crack initiation. In addition to the cohesive failure, there is an interfacial failure that is not less important because it is required for the formation of bridging CNT that constitute the main energy absorption mechanism in composites. Actually, the optimal combination of both cohesive and interfacial failure provides the best value of ILFT of the composite material.

The main question that stems of these results pertains to the loading rate effect: For example, why the t-CNT sample in the crack propagation stage does not exhibit an improved ILFT at a loading rate of 4 m/s, while a significant improvement is observed under 8 m/s. Because the fracture toughness is sensitive to crack velocity, which in turn is determined by the loading rate, the answer is given in part by comparing the respective crack velocities at a given crack extension. Whereas under a loading rate of 4 m/s, the maximum $G_{II}$ value of above 3.0 kJ/m² is attained already at a crack velocity of 1500 m/s, at a loading rate of 8 m/s, for the same crack velocity, the corresponding $G_{II}$ value is less than 2.5 kJ/m². To demonstrate the effect of crack velocity, Fig. 6 presents plots of crack velocity against crack extension for the t-CNT and the control samples under the two loading rates, where it is clearly seen that at the same crack extensions, the crack velocity under the 8 m/s loading rate is notably higher. Based on all these observations, it is concluded that fracture toughness is significantly higher at high crack velocities, where it becomes more sensitive to energy dissipation mechanisms of crack front interactions.

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**Conclusions**

Two main conclusions can be drawn from this research pertaining to the specific composite system studied here and more generally to loading rate effects on delamination fracture toughness.

In this study, we expand the scope of the quasi-static study to a higher range of loading rates, showing that a very small content of protein-treated CNT, interleaved into the composite structure, produces a significant improvement in the Mode II interlaminar fracture resistance of carbon fabric/epoxy laminate. The improvement is achieved by interleaving the protein-treated CNT layer specifically into the highest stress concentration region – the specimens loaded at different rates: 4 m/s (a–c), 8 m/s (d–f). For every sample, the roughness measurements were performed at different spots of the epoxy matrix in close proximity to a carbon fiber or a carbon fiber imprint (marked by the squares in the images), and the surface roughness was recorded at 3–4 sites at each spot.

A qualitative visual scrutiny of the SPM micrographs suggests a priori that t-CNT samples loaded at 8 m/s (Fig. 9d–f) generate much higher surface areas during fracture propagation than the same samples loaded at 4 m/s (Fig. 9a–c). Furthermore, the coverage of the carbon fibers by the epoxy matrix is significantly more extensive in the first rather than in the latter. The quantitative roughness measurements support the qualitative observations wherein the average roughness of the t-CNT samples tested at 8 m/s is approximately 300 nm (Fig. 9d–e) compared with 160 nm for the 4 m/s samples. This implies that excessive energy is dissipated at higher loading rates through cohesive fracture and roughness of the epoxy matrix.
midplane of the laminate. In addition, it is demonstrated that such improvements depend strongly on the loading rate.

The loading rate effect is expressed through the crack velocity, wherein at higher crack velocities during the crack expansion process, higher energies are dissipated – particularly in the protein-treated CNT laminates. High magnitude crack velocities are implicit under high loading rates, e.g., 8 m/s. In the latter scenario, the stored elastic energy prior to the onset of delamination results in crack initiation velocities at burst rates similar to those recorded under quasi-static conditions. Hence, the observed improvements in the ILFT, particularly in the protein-treated CNT laminates, occur only at high crack velocities.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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