FATIGUE INDUCED MICROCRACKING IN COMPOSITE TANK LAMINATES

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ABSTRACT

The development of linerless composite tanks fabricated with carbon fiber reinforced, polymer matrix composites is an enabling technology that will help reduce vehicle weight and increase payload capacity of future aircraft and spacecraft. A challenge in designing these tanks is selecting and characterizing composite material systems that show strong resistance to microcrack formation under cyclic mechanical loads caused by repeated fill and drain cycles of the tank. This paper studies the growth of damage as a function of mechanical cyclic load for three novel, microcrack-resistant materials that were purpose-designed by Composite Technology Development, Inc. (CTD) for linerless composite tank applications. Cross-ply [0/90], composite laminates were subjected to uniaxial cyclic loading at room temperature. The development of microcracks in the transverse plies was monitored as a function of cycles for different target strain levels. The experimental results show significant differences in microcrack formation among the material systems. The test data from one of the material systems is analyzed within the framework of microcrack fracture toughness. An analytical relationship is established between the rate of growth of microcracks in the composite and the microcracking energy release rate of the material.

KEYWORDS

Fatigue, microcracking, composites, damage.

INTRODUCTION

The development of lightweight, reusable, linerless tanks constructed from carbon fiber reinforced, polymer matrix composites will be essential to the success of future missions ranging from the aerial platforms to reusable launch vehicles (RLVs) [1,2]. Current state-of-the art tanks used for aerospace applications are fabricated as metal-lined, composite-overwrapped pressure vessels (COPVs). Lightweight linerless composite pressure vessels, due to the lack of a liner, provide for the lightest possible weight solution for a given set of requirements. In addition, elimination of the metallic liner can reduce part count, lower fabrication costs, and eliminate problems associated with thermal expansion mismatch between the metal liner and composite overwrap [3]. However, the use of linerless composite tanks also gives rise to several
additional challenges. In the absence of a metallic liner, the composite shell must not only support the thermo-mechanical loads (due to cryogenic liquid temperatures, external environments and internal pressures), but must also act as a permeation barrier for the fluid inside the tank. This latter requirement presents a significant obstacle, given the propensity for microcrack development in the polymer matrix under cyclic thermo-mechanical loading. Microcracking in composite laminates affects not only the structural integrity of the tank, but may ultimately lead to leakage of the fluid through the tank walls. Thus, the long-term use of linerless composite tanks hinges on the development of microcrack-resistant materials and the analytical characterization of damage growth and its resulting influence on the structural performance of these materials.

The objective of the current study is to investigate and model the development and growth of microcracks in carbon-fiber-reinforced, polymer-matrix composites under cyclic mechanical loads. Three different polymer matrix systems, all developed by Composite Technology Development, Inc. (CTD) to provide superior microcracking resistance at both ambient and cryogenic temperatures, were tested. The development of microcracks in the transverse (90°) plies of cross-ply [0/90]s laminates subjected to various cyclic stress levels was monitored over time. The results from the different material systems are compared to assess the level of microcracking resistance of these novel materials developed for composite tank applications. Finally, the experimental results are presented within the framework of an analytical model capable of defining how damage (microcrack density) evolves as a function of cyclic loads. The model, based on the concept of ‘microcrack fracture toughness,’ aims to relate microcrack density to the local material response and the global constitutive behavior under cyclic load. The performance of the composites under cyclic thermal loads or other thermo-mechanical loads is not investigated in this study.

MATERIALS AND EXPERIMENTAL PROCEDURES

Three sets of cross-ply [0/90]s laminates were fabricated for the experimental study by a hand-lay-up process using Toray T700 graphite fibers and three different matrix systems. These cross-ply laminate samples represent the building block of the laminate structure in the cylindrical section of a filament wound composite tank [1]. The elastic moduli of the unidirectional plies within these laminates ranged from 121 to 138 GPa in the longitudinal direction and 4 to 8 GPa in the transverse direction. The three different matrix systems, designated as CTD-DP5.1, CTD-525, and CTD-15XQ, are toughened epoxies and were developed by CTD specifically for use in linerless composite tank applications. CTD-DP5.1 and CTD-525 are room-temperature cure systems, suitable for wet filament winding processes, and have shown excellent performance against microcrack formation at cryogenic temperatures. CTD-15XQ is a high-temperature cure system that is designed for tow-preg winding applications.

The cross-ply laminates were fabricated with a nominal fiber volume fraction of 60% and overall thickness of approximately 1.15 mm, and were subsequently cut into uniaxial test coupons with a nominal length of 220 mm and width of 12 mm. Fiberglass end tabs were bonded to the test specimens for gripping. Constant-amplitude fatigue tests were conducted at room temperature using an MTS servo-hydraulic load frame. An extensometer with a gage length of 50.8 mm was attached to the samples to monitor strains. The samples were cycled in load control at R=0 (zero to max load), at levels corresponding to maximum strain values ranging from 0.5 to 1.0%. The tests were conducted to a maximum of 10,000 cycles at a frequency of 1 Hz.

The cyclic tests were performed in stress-controlled mode where the maximum stress magnitude was dictated by the initial strain values in the virgin (undamaged) laminate. The maximum limit for the load cycle was computed from specified strain levels at the start of the mechanical cycle test and was kept constant throughout the cycles. Therefore, the target strain values mentioned in the subsequent sections refer specifically to the strain level in the undamaged laminate that was used for computing the maximum stress level for the cyclic tests. Strain levels of 0.67% and 1% were selected based on the design strain level in the cylindrical section of the tank with a burst factor of 2.25 and 1.5 respectively (see Table 1). Additional strain levels of 0.5% and 0.75% were chosen to provide a more complete set of data for assessing the effects of strain level on microcrack growth rate.
During the tests, the formation of microcracks in the transverse (90°) plies was monitored using a handheld digital microscope with 100x magnification. To detect microcracks at specific cyclic intervals, the test was paused and the specimen loaded to approximately 70% of the maximum load level for the cycle. Each side of the sample was scanned with the hand held microscope over a gage length of approximately 100 mm. The average microcrack density was calculated by dividing the number of counted microcracks by the total gage length. The rate of damage growth (microcrack density) was determined as a function of applied cycles.

TABLE 1
RELATIONSHIP BETWEEN CYCLIC STRAIN LEVEL AND THE TANK’S BURST FACTOR

<table>
<thead>
<tr>
<th>Longitudinal failure strain of T700 carbon fiber reinforced composite</th>
<th>~1.5%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hoop strain in the tank at its burst pressure</td>
<td>1.5%</td>
</tr>
<tr>
<td>Hoop strain at operating pressure for tanks designed for burst factor = 2.25</td>
<td>0.67%</td>
</tr>
<tr>
<td>Hoop strain at operating pressure for tanks designed for burst factor = 1.5</td>
<td>1.0%</td>
</tr>
</tbody>
</table>

EXPERIMENTAL RESULTS

Microcracks in the transverse (90°) plies were observed and photographed periodically throughout the tests using the digital microscope. Examples of microcracks in the specimens are shown in Figure 1. These microcracks typically extended across the thickness of the 90° plies and stopped at the interface of the 0° plies. After continued cycling, the stress intensity at the tip of the microcrack occasionally caused an interfacial crack to develop at the interface of the 0° and 90° plies, causing a partial delamination as shown in Figure 2. In most cases, these delaminations were confined to a localized area around the transverse microcracks.

**Figure 1**: Examples of microcracks in transverse (90°) plies of a cross ply [0/90]s laminate.
Microcrack density measurements were recorded as a function of applied cycles for each specimen. The results were then averaged at each strain level for comparison among the material types. Three samples were tested at each strain level. The average microcrack density measurements (microcracks/mm) from the transverse plies are shown in Figures 3 through 5 for each of the matrix systems: CTD-DP5.1, CTD-525, and CTD-15XQ, respectively.

The rate of damage growth in CTD-DP5.1 is shown in Figure 3. This material was cyclically loaded to maximum strain values of 0.67, 0.75, and 1.0%. Over the first 2000 cycles, the damage growth rate at the 0.67 and 0.75% strain levels for the composite specimen made with CTD-DP5.1 was very similar. The samples tested at the 0.67% level were continued to 10,000 cycles. It is evident that the rate of damage growth was fairly steady throughout these tests, and a saturation point was never attained. For the samples tested at the 1.0% strain level, the microcrack density increased rapidly, eventually causing specimen failure between 200 and 800 cycles.

As shown in Figure 4, the CTD-525 material was tested at maximum strain values of 0.5, 0.67, 0.75, and 1.0%. The rate of damage growth at 0.5% was extremely small, but increased steadily with increasing strain values. At a strain level of 1.0%, the microcrack density rapidly increased until specimen failure occurred between 700 and 1000 cycles. The third material, CTD-15XQ, was only tested at strain values of 0.5 and 0.67%, as shown in Figure 5. The damage growth rate in this material was observed to be much higher than in the other two materials at similar strain levels.

Figures 6 and 7 compare the growth of microcrack density for the three materials at target strain levels of 0.67% and 0.75%, respectively. From Figure 6, it is evident that CTD-15XQ has much lower resistance to microcrack formation under mechanical cyclic load compared to the other two materials. This may be attributed to the differences in the post-cure residual stress state between the materials, as CTD-15XQ is a high-temperature cure epoxy, while the other two resins are cured at room temperature. During the cure of 15XQ, the transverse (90°) plies are restricted from contracting due to the difference in thermal expansion coefficients with the adjacent longitudinal (0°) plies. This creates a residual tensile stress state in the loading direction of the 90° plies. When mechanical cyclic load is imposed, the tensile residual stresses increase the net tensile stress in these plies, resulting in an increased rate of microcrack growth. It should be noted that the problem is exaggerated at cryogenic temperatures, where significant build-up of residual stresses can increase the rate of microcrack growth.

In Figure 7, the microcrack density measurements are compared between CTD-DP5.1 and CTD-525 at the strain level of 0.75%. Results indicate that there is a significant difference in the microcracking resistance for these two material systems despite the fact they are both room-temperature-cure systems. After 2000 cycles, the microcrack density in CTD-525 was approximately 0.2 microcracks/mm compared to only 0.01 microcracks/mm in CTD-DP5.1, indicating material selection and characterization is key to mitigating microcracks in linerless composite tanks made with these composites.
Figure 3: Microcrack density growth in transverse plies of CTD-DP5.1.

Figure 4: Microcrack density growth in transverse plies of CTD-525.

Figure 5: Microcrack density growth in transverse plies of CTD-15XQ.
ANALYTICAL MODELING

The experimental data were analyzed within the framework of ‘microcrack fracture toughness,’ proposed by Nairn et al [5-8]. The instant of formation of the microcrack is predicted when the total energy released by the formation of that microcrack reaches the critical energy release rate for microcracking, $G_{mc}$, or the microcracking fracture toughness. The greater the energy released, the tougher the material is against microcrack formation, and therefore the more resistant it is to formation of leak paths in the tank wall.

The strength of this approach is in the use of an energy analysis as a tool for understanding and predicting microcracking under a variety of conditions. The energy analysis is used in conjunction with experimental results to derive intrinsic material properties. For example, under static loading, a given material can be characterized by a microcracking fracture toughness parameter, $G_{mc}$. Once $G_{mc}$ is known, the nucleation and growth of damage (microcrack density) can subsequently be related to the local material characteristics (microcrack fracture toughness), global constitutive response (elastic properties), and external loading (thermo-mechanical loads) for any arbitrary ply thickness and orientation.
In the current study, the microcrack fracture toughness approach was applied to analyze the experimental data presented in the previous section for the CTD-525 material. This material was chosen for detailed analysis due to the quality of microcrack density measurements obtained at strain levels of 0.67, 0.75, and 1.0%. As derived by Nairn [5], the microcrack energy release rate, $\Delta G_m$, due to the formation of new microcracks under a uniaxial cyclic load reversal of $\Delta \sigma$ in a cross-ply laminate is calculated as

$$\Delta G_m = \left( \Delta \sigma^2 \frac{E_T^2}{E_o^2} \right) t_1 C_3 \left[ \frac{2 \chi \left( \frac{\rho}{2} \right)}{2} - \chi (\rho) \right]$$

where $C_3$ is a function of the transverse-ply modulus $E_T$ and the thicknesses of the unidirectional plies ($t_1$ and $t_2$), $E_o$ is the effective modulus of the cross-ply laminate, $\rho$ is the normalized microcrack spacing, and $\chi$ is given by

$$\chi (\rho) = 2 \alpha \beta \left( \alpha^2 + \beta^2 \right) \frac{\cosh(2 \alpha \rho) - \cos(2 \beta \rho)}{\beta \sinh(2 \alpha \rho) + \alpha \sin(2 \beta \rho)}$$

where $\alpha$ and $\beta$ are functions of the elastic properties of the material. Eqn. 2 can be used to plot the energy release rate, $\Delta G$, as a function of the microcrack density, or the damage parameter $D$. By experimentally measuring the rate of growth of microcrack density $D$ with the number of applied cycles of loading, $N$, a modified Paris law was proposed by Nairn et al [5] to relate the rate of increase in microcrack density per cycle, $dD/dN$, to the energy release rate, $\Delta G_m$, as

$$\frac{dD}{dN} = A(\Delta G_m)^n$$

where $A$ and $n$ are experimentally determined material constants. These two parameters are assumed to be unique to the material, and once determined, can be used for a variety of laminate geometries and architectures. This eliminates the need for repeated fatigue testing of different laminate configurations during tank laminate design.

Experimental results of microcracking fatigue data in carbon/epoxy cross-ply laminates generated by Nairn et al [5] are shown in Figure 8. It can be seen from this figure that the energy release rate is constant at low microcrack density when there are no interactions between the microcracks. In this regime, the rate of increase in microcrack density is very high. As microcrack density increases, $\Delta G$ experiences a slight increase followed by a gradual decrease corresponding to a leveling off of the $D$ vs. $N$ curve. When the spacing between microcracks becomes small, the interactions between the microcracks become predominant. This results in the energy released by each new microcrack to decrease and the microcrack density level to saturate.

![Figure 8: Microcracking fatigue data for a cross-ply carbon/epoxy laminate [1].](image-url)
The microcrack growth data for CTD-525 from cyclic tests conducted at strain levels of 0.67, 0.75, and 1.0% are presented in Figures 9 through 11, respectively, along with the calculation of \( \Delta G \) (red curves) as a function of microcrack density from Eqn. 2. In these plots, the microcrack density, \( D \), is plotted along the abscissa to allow direct comparison of the \( D \) vs. \( N \) data with \( \Delta G \). At the 0.67% strain level, \( \Delta G \) is essentially constant over the range of recorded microcrack density measurements. It should be noted that, following an initial region of transitory behavior, the \( D \) vs. \( N \) data are well represented by a linear fit, indicating a constant rate of microcrack growth, \( dD/dN \), within the range of constant \( \Delta G \). This confirms the validity of Eqn. 4 within this regime of damage evolution.

For cyclic tests performed at strain levels of 0.75 and 1.0% (Figures 10 and 11), \( \Delta G \) is constant at lower microcrack densities. Subsequently it shows a slight increase of approximately 10% before decreasing as microcrack density increases, similar to the behavior shown in Figure 8. At the strain level of 0.75%, a linear region of \( D \) vs. \( N \) (constant \( dD/dN \)) is observed again, following the initial transitory behavior, within the region of constant \( \Delta G \). For cyclic tests performed at 1.0% strain level, a linear fit was applied to the \( D \) vs. \( N \) data within the region of constant \( \Delta G \). It is likely that the initial transitory behavior observed at the lower strain levels occurred prior to the initial measurement at this high strain level. The linear fit was not extended into the higher microcrack density measurements, as these corresponded to an increase in \( \Delta G \) as shown in Figure 11.

**Figure 9:** Microcracking fatigue data for CTD-525 at \( \Delta \varepsilon = 0.67\% \).

**Figure 10:** Microcracking fatigue data for CTD-525 at \( \Delta \varepsilon = 0.75\% \).
The values of $dD/dN$ obtained from Figures 9 through 11 are plotted on log coordinates against the corresponding values of $\Delta G$ in Figure 12. It is evident that the power-law relationship assumed in Eqn. 4 represents the data extremely well. This confirms that the modified Paris-type relation (Eqn. 4) proposed by Nairn [5], based on the microcracking energy release rate, $\Delta G_m$, can be used to model the rate of microcrack growth in composite laminates subjected to cyclic loading. However, the range of validity of this relation cannot be determined from the limited amount of data collected in this study.

CONCLUSIONS

Microcracking in composite laminates subjected to mechanical loads is an obstacle to the development of linerless composite tanks. This damage can affect not only the structural integrity of the tank, but may ultimately lead to leakage of fluid through the tank walls. In this study, the evolution of damage in the form of microcracks in transverse plies of a cross-ply laminate subjected to uniaxial cyclic loading was investigated. Carbon fiber reinforced composite laminates of cross-ply laminate configurations made from three different matrix materials were investigated in this study. These novel microcrack resistant matrix
materials were purpose-designed by CTD for linerless composite tank applications. Two of the matrix materials were room-temperature-cure epoxies suitable for wet winding and one was a high-temperature-cure system suitable for towpreg winding. Significant differences in microcrack resistance under cyclic mechanical loads were observed between the materials. The greater microcrack density in the high-temperature-cure system is consistent with the higher tensile residual stresses induced in the transverse plies during elevated-temperature cure. Substantial differences in microcrack evolution were also observed between the two room-temperature-cure epoxies, with one system (CTD-DP5.1) showing extremely low microcrack damage evolution.

The experimental data for one of the materials were analyzed within the framework of microcrack fracture toughness. The analysis assumes that microcracks form when the total energy released by the formation of that microcrack reaches a critical energy release rate, an intrinsic property of that material. Analysis shows that the rate of microcrack growth in the material under uniaxial cyclic loading can be related to the microcracking energy release rate of the material through a modified Paris law formulation. This indicates that the microcracking energy release rate may provide a useful technique for the prediction of damage growth in composite materials under cyclic mechanical load, in much the same way the stress intensity factor range is used to analyze microcrack growth in monolithic materials. Further characterization of this model will be useful for optimizing the design of linerless composite tanks using the novel microcrack resistant materials.

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