

ASSESSMENT OF COMPOSITE DELAMINATION SELF-HEALING UNDER CYCLIC LOADING

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SUMMARY

Recently, the promise of self-healing materials for enhanced autonomous durability has been introduced using a micro-encapsulation technique where a polymer based healing agent is encapsulated in thin walled spheres and embedded into a base polymer along with a catalyst phase. For this study, composite skin-stiffener flange debonding specimens were manufactured from composite prepreg containing interleaf layers with a polymer based healing agent encapsulated in thin-walled spheres. Constant amplitude fatigue tests in three-point bending showed the effect of self-healing on the fatigue response of the skin-stiffener flange coupons. After the cycling that created debonding, fatigue tests were held at the mean load for 24 hours. For roughly half the specimens tested, when the cyclic loading was resumed a decrease in compliance (increase in stiffness) was observed, indicating that some healing had occurred. However, with continued cycling, the specimen compliance eventually increased to the original level before the hold, indicating that the damage had returned to its original state. As was noted in a previous study conducted with specimens tested under monotonically increasing loads to failure, healing achieved via the micro-encapsulation technique may be limited to the volume of healing agent available relative to the crack volume.

Keywords: *Self-healing, Delamination, Fatigue, Micro-encapsulation, z-pins*

INTRODUCTION

Recently, the promise of self-healing materials for enhanced autonomous durability has been introduced using the micro-encapsulation technique [1]. In this technique, a polymer based healing agent is encapsulated in thin walled spheres and embedded into a base polymer along with a catalyst phase (fig.1).

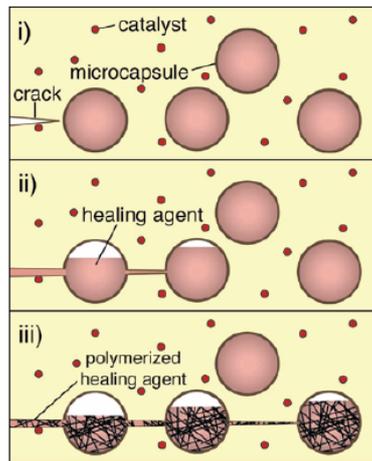


Fig.1 Micro-encapsulation technique for self-healing

As a crack develops and grows in the base polymer, the spheres fracture and release the healing agent, which reacts with the catalyst and polymerizes, thereby healing the crack. Using a height-tapered Double Cantilever Beam (DCB) fracture specimen, White, et al., demonstrated recovery of 90% of the virgin fracture toughness of the base polymer [1].

Prepreg Material

The micro-encapsulation technique was recently applied to the resin matrix of a fiber reinforced composite material [2]. An interleaf prepreg material was manufactured where the interleaf layer contained the micro-encapsulated healing agent and the catalyst (fig.2) .

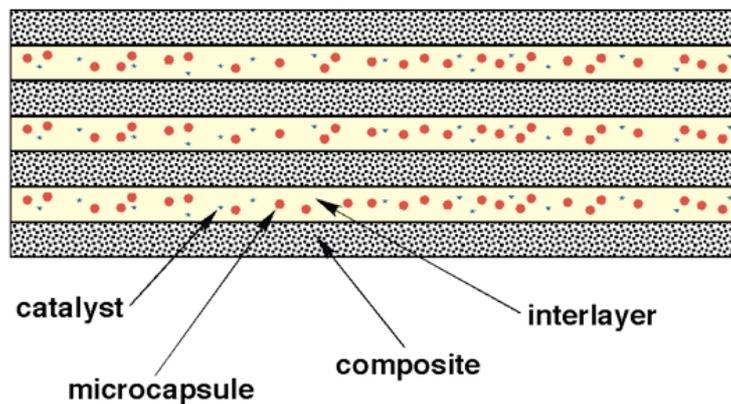


Fig.2 Composite prepreg with micro-encapsulated interleaf layer

The base matrix resin consisted of Epon 862® with an Epicure 3274® curing agent. This material cures at room temperature, achieving a tack-free cure overnight and a full cure in seven days. Polymer shell microcapsules, 10-100 µm in diameter, were produced and filled with a dicyclopentadiene (DCPD) healing agent. A wet-winding process was used to produce the composite prepreg, where IM7-GP 12K carbon fiber bundles passed through a resin bath before being wound onto the mandrel. Microcapsules and catalyst were

dispersed in the base resin bath and deposited onto a carrier fabric on the top of each composite layer containing the base resin only with no healing agent. This composite plus interlayer ply was then wound on the mandrel. Continuous prepreg sheets were produced and cut.

Specimen Manufacture

The self-healing composite prepreg was used to manufacture six-inch long by one-inch wide composite coupon specimens, consisting of a skin and a stiffener flange tip, designed to study skin/stiffener debonding (figure 3) [3,4]. In addition, through-thickness reinforcement, in the form of 0.5% by volume pultruded carbon z-pins, was included near the flange tips to improve the resistance to debonding [5]. By combining these two technologies, it was hoped to obtain a synergy between the damage tolerance provided by the z-pins and the durability provided by the self-healing matrix. Panels were layed up and cured on a vacuum debulk table at 95°F for 48 hours using a 15 psi vacuum bag pressure. Specimens were manufactured with 14 plies in the skin with a $[45/90/-45/0/45/0/-45]_s$ orientation and 10 plies in the stiffener flange with a $[45/90/-45/0]_s$ orientation. Each of the plies in the skin and stiffener flange contained an interleaf layer with the micro-encapsulated healing agent.

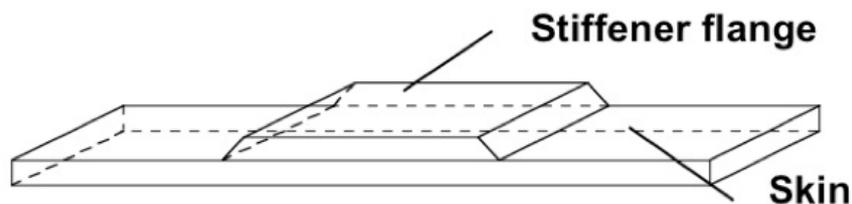


Fig.3 Skin/stiffener debonding specimen

Test Procedure

Three-point-bend tests, with a five-inch span between the rollers, were performed to measure the onset and growth of skin/stiffener debonding. In the initial study described in reference 5, only slow monotonically increasing loads were applied, whereas in the current study, cyclic loads were applied. Tests were performed using the central solid load nose instead of a roller, as shown in figure 4, to allow a good view of damage growth from both flanges during the test.

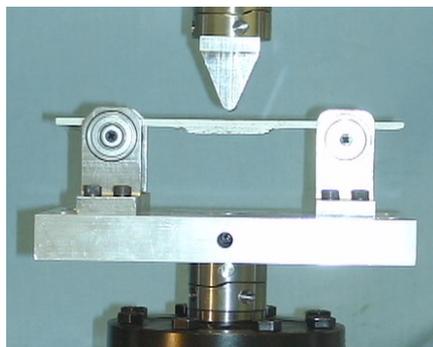


Fig.4 Three-point bend test set-up

Specimen edges were painted white, using a thin coat of spray paint, to easily detect the onset and growth of damage. Tests were performed in a 5-kip hydraulic test stand.

For the initial study, specimens were loaded monotonically to failure in three-point bending to measure the skin/stiffener debonding strength and the recovered strength after healing [5]. Monotonic loading was stopped after the onset of damage. A few specimens were subjected to reverse bending during the hold time of 24-48 hours to effectively clamp the fracture surfaces together under pressure before reloading (fig.5).



Fig.5 Reverse bending clamp-up

In the current study, constant amplitude fatigue tests were performed in three-point bending to assess the effect of self-healing on the fatigue response of the skin-stiffener flange coupons. Tests were performed in load control, at a cyclic frequency of 5 Hertz, and R-ratio of 0.1, with maximum cyclic load levels corresponding to 50% and 70% of the average load at delamination onset from the static tests in the initial study. Fatigue tests were held at the mean load for 24 hours after the onset of damage before resuming the cycling loading.

TEST RESULTS

Before examining the fatigue results in the current study, it is helpful to review the results of the initial study utilizing only monotonically increasing loads. Most tests exhibited a loss in stiffness after cracking and reloading, as shown in figure 6, indicated that no healing had occurred following unloading and reloading.

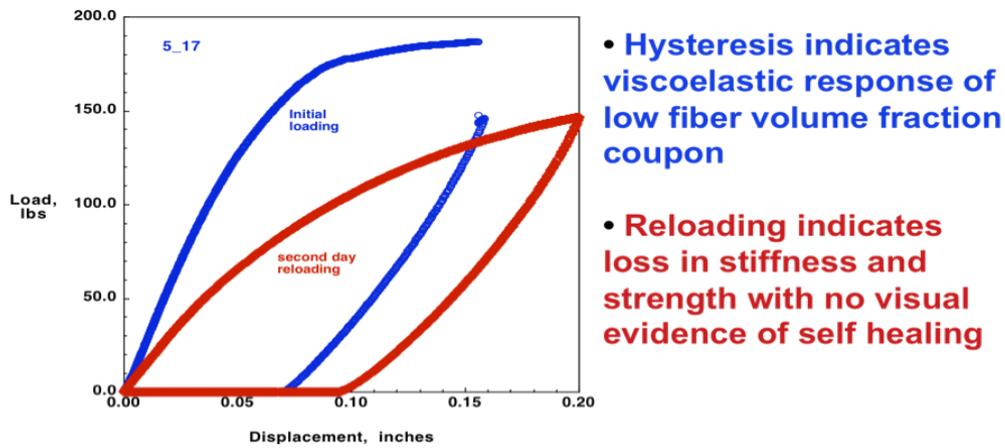


Fig.6 Response of self-healing coupon test [5]

Micrographs showed that delaminations could migrate to the top of the interleaf layer due the asymmetric loading, and hence, bypass the embedded capsules (fig.7).

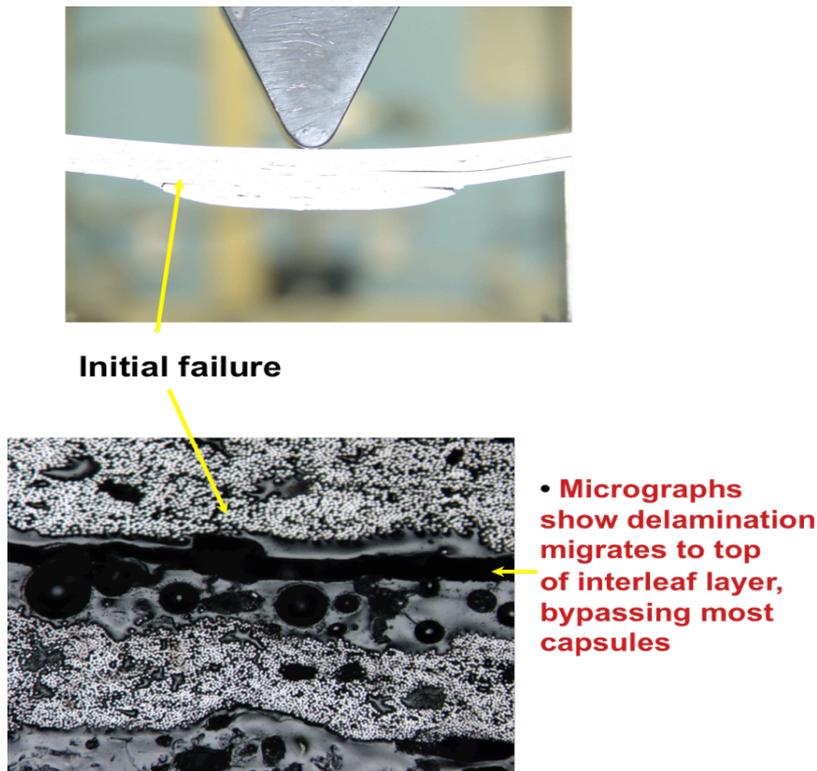
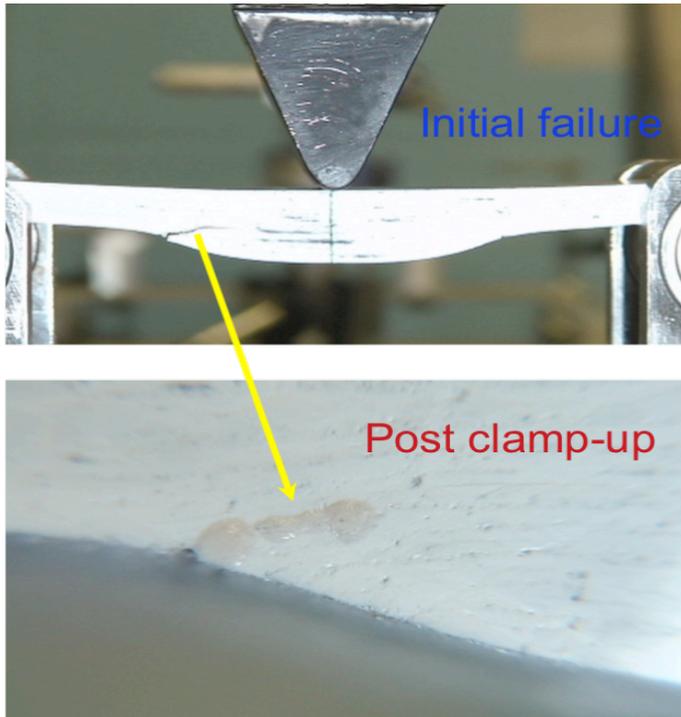


Fig.7 Micrographs showing delamination migration [5]

A few test specimens were loaded until initial failure was observed and then were clamped in reverse bending before reloading. In one case, healing was observed as evidenced by healing agent that leaked to the specimen edge forming a visible “scar” (fig.8).



Visual evidence of self healing “Scar” after 2 day clamp-up

Fig.8 Physical evidence of self-healing in coupon test with clamp-up [5]

This specimen retained 96% of its original strength upon reloading, indicating healing had occurred (fig.9).

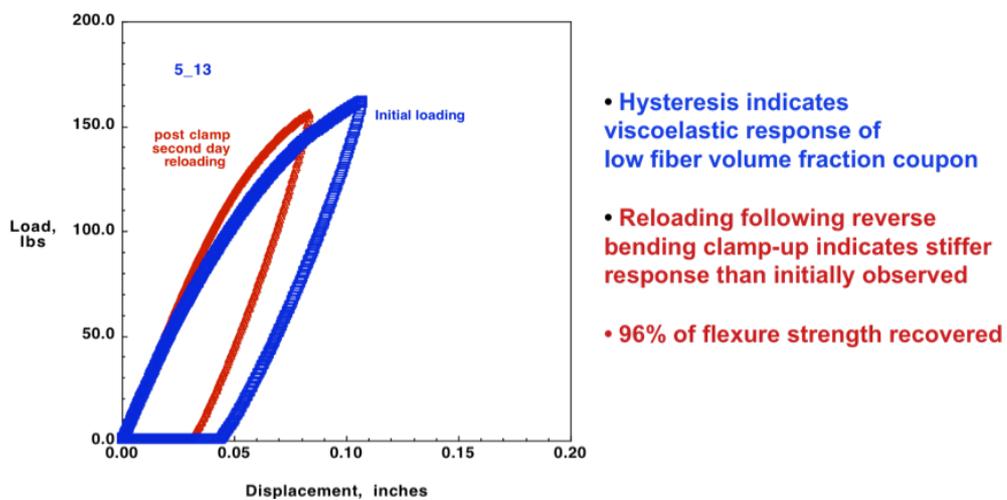


Fig.9 Response of self-healing coupon test with clamp-up [5]

In the current study, none of the fatigue tests exhibited visual evidence of healing similar to the “scar” observed in the post-clamp up static test shown in figure 8. However, in about half of the fatigue tests there was some evidence that self healing had occurred in the form of a compliance decrease (stiffness increase) after a hold in the cyclic loading. Figure 10 shows specimen compliance as a function of load cycles for a $P_{\max}=70\% P_{\text{ult}}$ test. The specimen was held at the mean load for 24 hours following the onset of damage. Upon reloading, a decrease in compliance (increase in stiffness) was observed indicating some healing had occurred. However, with continued cycling, the specimen compliance eventually increased to the original level before the hold, indicating that the damage had returned to its original state. This behavior was observed in four of the six fatigue tests performed at $P_{\max}=70\% P_{\text{ult}}$. The remaining two fatigue tests performed at $P_{\max}=70\% P_{\text{ult}}$ showed no change in compliance after the hold, as shown in figure 11.

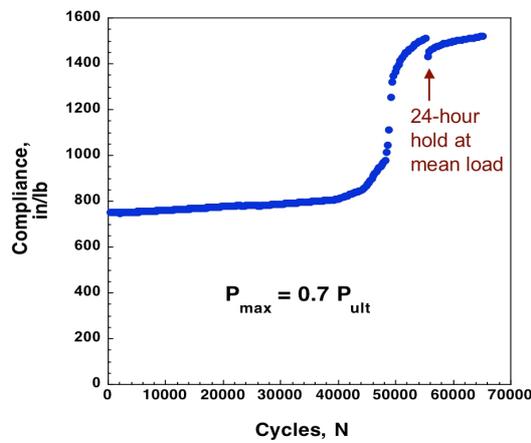


Fig.10 Compliance change as a function of fatigue cycles for a $P_{\max}=70\% P_{\text{ult}}$ test

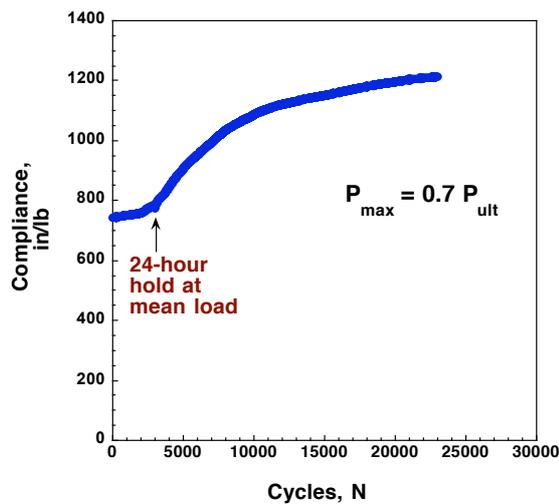


Fig.11 Compliance change as a function of fatigue cycles for a $P_{\max}=70\% P_{\text{ult}}$ test

Figure 12 shows specimen compliance as a function of load cycles for a $P_{\max}=50\% P_{\text{ult}}$ test. The specimen was held at the mean load for 24 hours following the onset of damage. Upon reloading, a decrease in compliance (increase in stiffness) was observed indicating some healing had occurred. However, with continued cycling, the specimen compliance eventually increased to the original level before the hold, indicating that the damage had returned to its original state. This behavior was observed in three of the six fatigue tests performed at $P_{\max}=50\% P_{\text{ult}}$. The remaining three fatigue tests performed at $P_{\max}=50\% P_{\text{ult}}$ showed no change in compliance after the hold, as shown in fig.13.

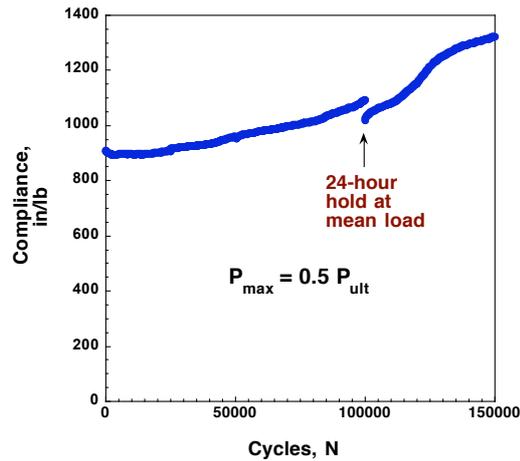


Fig.12 Compliance change as a function of fatigue cycles for a $P_{\max}=50\% P_{\text{ult}}$ test

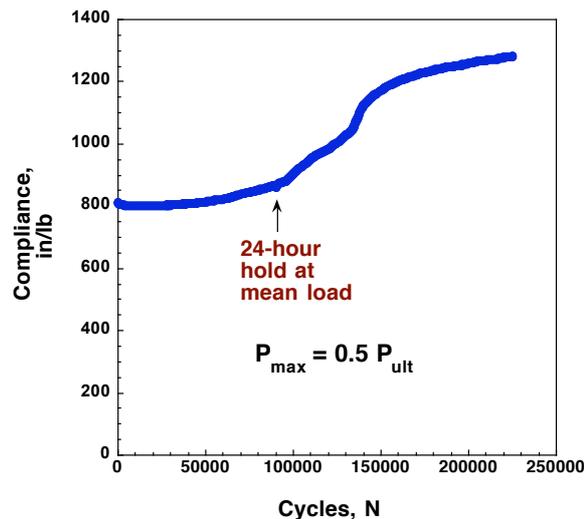


Fig.13 Compliance change as a function of fatigue cycles for a $P_{\max}=50\% P_{\text{ult}}$ test

DISCUSSION

In the static tests performed in the original study, healing was only achieved after holding specimens overnight in reverse bending to provide contact under pressure for a significant amount of time. For the fatigue tests performed in this study, by holding at the mean bending load for 24 hours where the crack faces remained open, only preliminary short-term benefits (in the form of a compliance increase) were observed for roughly half of the fatigue tests performed. The presence of the 0.5% density z-pins at the flange tip may have helped reduce the crack opening, and hence, reduced the amount of healing agent needed to fill the void. Even in these cases, the specimen compliance eventually increased with continued cycling to the original level before the hold, indicating that the damage had returned to its original state. As noted in reference [5], the micro-encapsulation technique may prove more robust when capsule sizes can be produced that are small enough to be embedded in the matrix resin without the need for using an interleaf layer. However, in either configuration, the amount of healing that can occur may be limited to the volume of healing agent available relative to the crack volume that must be filled.

CONCLUSIONS

Composite skin-stiffener flange debonding specimens were manufactured from composite prepreg containing interleaf layers with a polymer based healing agent encapsulated in thin-walled spheres. Constant amplitude fatigue tests in three-point bending showed the effect of self-healing on the fatigue response of the skin-stiffener flange coupons. After the cycling that created debonding, fatigue tests were held at the mean load for 24 hours. When the cyclic loading was resumed, a decrease in compliance was observed for roughly half of the specimens tested, indicating healing had occurred. However, with continued cycling, the specimen compliance eventually increased to the original level before the hold, indicating that the damage had returned to its original state. As was noted in a previous study conducted with specimens tested under monotonically increasing loads to failure, healing achieved via the micro-encapsulation technique may be limited to the volume of healing agent available relative to the crack volume.

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