RESPONSE OF SOLVENT-BASED SELF-HEALING SMART MATERIALS UNDER FATIGUE

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ABSTRACT

Self-healing of epoxy matrix could prove essential in increasing the reliability and service life of fiber-reinforced polymers. The principle of encapsulated liquid healing agents dispersed in the matrix has been demonstrated successfully using different chemistries and different testing setups. In addition, progress has been made in the field of smart composites regarding crack detection and closure, using for example fiber optics and shape memory alloy fibers. An active response is given to a detected and localized damage event by the combined action of crack closure and local heating.

We report here on encapsulated EPA solvent based epoxy materials with embedded SMA fibers tested in fatigue loading of TDCB sample geometries. Healing in solvent-based capsule systems occurs through solvent diffusion, swelling of the polymer on the crack faces thus enabling further reaction of residual monomer and bonding crack faces which are in contact.

Epoxy samples loaded with EPA microcapsules fared substantially better than samples without capsules or samples with inactive control solvent microcapsules. This behavior is attributed to a crack blunting effect: as the EPA diffuses into the matrix, the modulus decreases while the epoxy becomes more ductile. As a result, complete crack arrest at relatively high stress intensities $K_{ic}$ was observed.

TDCB samples containing capsules and embedded SMA wires were tested statically until complete fracture, activated through the SMA wires for healing and finally tested in fatigue after healing. Such healed samples exceeded the virgin properties in fatigue response. Secondly, samples with or without SMA wires were tested in fatigue for 20,000 cycles at high stress intensities, followed by a rest period (with SMA wire activation, if applicable) for different durations and further tested in fatigue. Sample response was improved by SMA wire activation and longer healing rest periods.

Overall, EPA solvent capsules are beneficial to the fatigue life of an epoxy matrix, by combined effects of crack blunting during loading and crack healing at rest periods.
1. INTRODUCTION

Fatigue cracks are an important damage scenario in the service life of composite materials. In this work, an epoxy matrix with a solvent-filled capsule healing system based on Caruso et al.'s work [1], combined with embedded shape memory alloy (SMA) fibers was investigated under dynamic testing [2]. The effect of solvent on the epoxy during fatigue testing is analyzed through both continuous experiments, as well as with rest periods and SMA activation.

2. MATERIALS

The epoxy resin was Epon 828, a DGEBA resin (Brenntag) which was cured with diethylenetriamine (DETA, Sigma Aldrich) in the stoichiometric ratio of 100:12. Curing took 24h at ambient temperature followed by post-curing 24h at 35°C (PC1). The SMA wire used in this study was a martensitic NiTiCu alloy with respective composition of 44.86/45.08/10.06 and a diameter of 150µm (Furukawa Techno Material).

3. METHODS

TDCB samples were cast in silicone molds and spacers were used to minimize the amount of microcapsules per sample by casting in two steps: first the surrounding matrix consisting of pure epoxy resin, then the self-healing capable, 15wt% microcapsule loaded resin. The microcapsules were produced using the urea-formaldehyde (UF) microencapsulation protocol established by Blaiszik et al. [3] using a stirring rate of 400rpm and retaining only the microcapsule fraction between 125 and 355µm of diameter. For samples with integrated SMA wires, knots were introduced at their ends and these were aligned using free-hanging 50g weights before the silicone molds were closed, thereby clamping the SMA wires at 3 equally spaced locations (as done previously [2]).

Fatigue testing involved a tension-tension triangular wave form with an stress intensity ratio of R=K_{min}/K_{max}=0.1 a frequency of 5Hz and for active healing in samples with integrated SMA wires, an electrical current of 0.5A per wire was applied during the break.

4. RESULTS

In the first instance, we specifically compared plain epoxy to the well-known DCPD/Grubbs self-healing epoxy and the EPA solvent-loaded epoxy. Representative results are presented in Fig.1. At constant 0.47MPam^{1/2} loading, a crack in the plain sample progresses rapidly while in the DCPD/Grubbs sample, crack progression was slower. EPA solvent microcapsule loaded TDCB samples also showed a reduced crack progression rate as compared to the plain epoxy, but after about 4h for this sample, complete crack arrest at these relatively high loading conditions was observed. This behavior was observed for samples loaded with solvent microcapsules even if the time until crack arrest varied.
In plain epoxy, crack progression is fast while in the DCPD/Grubbs sample crack progression is slower. In the EPA solvent microcapsule loaded TDCB sample, crack arrest occurred after 4h ($\Delta K=0.47\text{MPam}^{1/2}$).

Based on the observations above, we investigated the solvent effect on stress and strain distribution around the crack tip using the finite element method (FEM). The analysis was performed using ANSYS and a simple 2D crack tip model was generated. Fig. 2 shows the chosen geometry: the crack tip notch protruded by 100µm into the 400µm wide geometry. The crack tip was modeled by a simplified circular notch of 1µm of diameter, surrounded by a solvent-weakened layer of 4µm thickness (±2h of solvent diffusion). Using the symmetry of this problem, the bottom boundary was clamped while a stress of 1MPa was applied to the top boundary.

Figure 2: Equivalent von Mises stress and strain distribution around the crack tip for the pure epoxy case as well as the solvent-affected epoxy.

The model showed a slight increase in the strains and a substantial decrease of the stress in the crack tip. Knowing that the solvated epoxy showed an almost threefold increased elongation at break (5.7% vs. 2.0% in plain epoxy), the observed crack arrest is therefore due crack blunting.
After investigating continuous fatigue testing on virgin and healed samples, we also characterized the effect of resting periods during low-cycle fatigue tests. The tests were conducted on samples with or without SMA wires at a stress intensity of 0.50MPa m^{1/2} and the resting periods were 10min, 2h and 24h. Fig.3 shows a representative results for a 10min rest period is shown. The rest period on samples without SMA wires had no significant effect on the overall trend of the crack progression while in samples with SMA wires, crack arrest occurred.

Figure 3: For 10min rest periods, no effect was observed unless the SMA wires were activated, which led to crack arrest.

5. CONCLUSIONS

Fatigue performance of a self-healing epoxy with EPA solvent-filled UF microcapsules and SMA wires was investigated. Complete crack arrest for very high loading conditions was observed and this phenomenon is due to the reduced stresses in the crack tip due to the solvent. Furthermore, SMA activation led to crack arrest during low-cycle fatigue tests, which was not observed in samples without SMA wires.

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