EPOXY-AMINE BASED SELF-HEALING IN HIGH TEMPERATURE CURED EPOXY

Henghua Jin[1,4], Chris L. Mangun[2], Dylan S. Stradley[3], Nancy R. Sottos[3,4], Scott R. White[1,4,*]

[1] Aerospace Engineering, University of Illinois at Urbana Champaign
[2] CU Aerospace 2100 S. Oak St. – Suite 206, Champaign, IL, 61820, USA
[3] Materials Science and Engineering, University of Illinois at Urbana Champaign
[4] Beckman Institute for Advanced Science and Technology, University of Illinois at Urbana Champaign, Urbana, IL, 61801 USA
Email: swhite@illinois.edu

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ABSTRACT

A dual-microcapsule epoxy-amine based self-healing system has been developed for use in high temperature cured epoxy. One capsule contains a modified aliphatic polyamine (EPIKURE 3274) while the second capsule contains a diluted epoxy monomer (EPON® 815C). The reactive amine microcapsules are prepared by vacuum infiltration of EPIKURE 3274 into hollow polymeric microcapsules, as shown in Figure 1(a). Epoxy microcapsules are prepared by an *in situ* polymerization method [1], as shown in Figure 1(b). Both types of capsules were incorporated into an epoxy matrix (EPON® 828/DETA) at various concentrations.

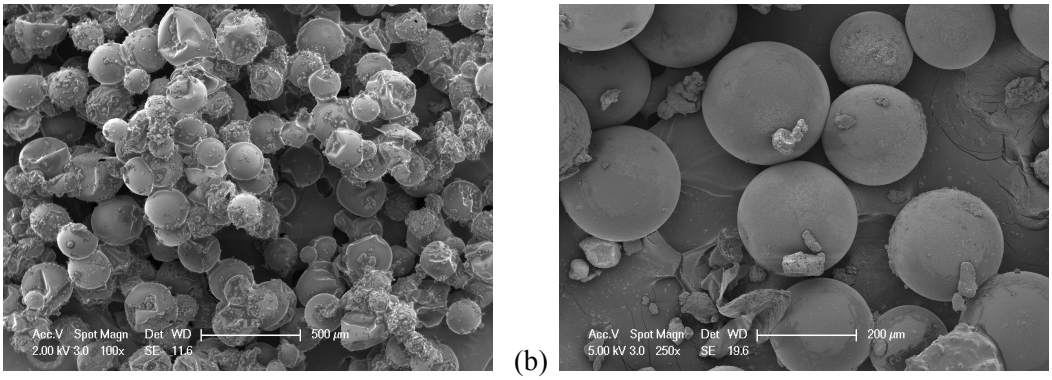

Figure 1. Scanning electron micrographs of (a) 3274 amine microcapsules, (b) EPON 815C microcapsules.

Fracture of the epoxy matrix ruptures both types of capsules releasing their respective core materials into the crack plane. Upon mixing of both healing agents, polymerization occurs at room temperature without any external intervention. Recovery of mode-I fracture toughness was measured using tapered-double-cantilever-beam (TDCB) specimens [2]. Specimens were allowed to heal for 48 h at room temperature following the virgin fracture test.

TDCB specimens cured at room temperature were used to investigate healing performance of various capsule ratios of epoxy to amine while holding the total capsule loading at a constant 10wt%. The optimal healing performance (85.1 ± 13.0%) was obtained at a ratio of 4 to 6 for 3274 amine capsules to EPON 815C capsules as shown in Figure 2(a). The amount of amine required to effectively cure the epoxy deviates from the stochiometric ratio of EPIKURE 3274 to EPON 815C (4:10) indicating that some of the 3274 amine is lost during specimen preparation. This may due to diffusion of 3274 amine from the core which then reacts with the epoxy matrix, perhaps forming a secondary barrier. As such, additional amine capsules are required to optimally react with the 815C to form a strong healing bond. With the optimal ratio of amine to epoxy capsules established, a set of experiments was carried out to determine the effect of total capsule concentration (results shown in Figure 2(b)). In general there is an increasing healing efficiency as the total capsule concentration is increased. There is a local maximum

at 10 wt% but the highest healing efficiency of 91% is obtained with 15 wt% capsules. There is also a steady increase in virgin toughness as the capsule concentration is increased due to toughening mechanisms in epoxy that have been well-documented in the literature such as crack pinning [3].

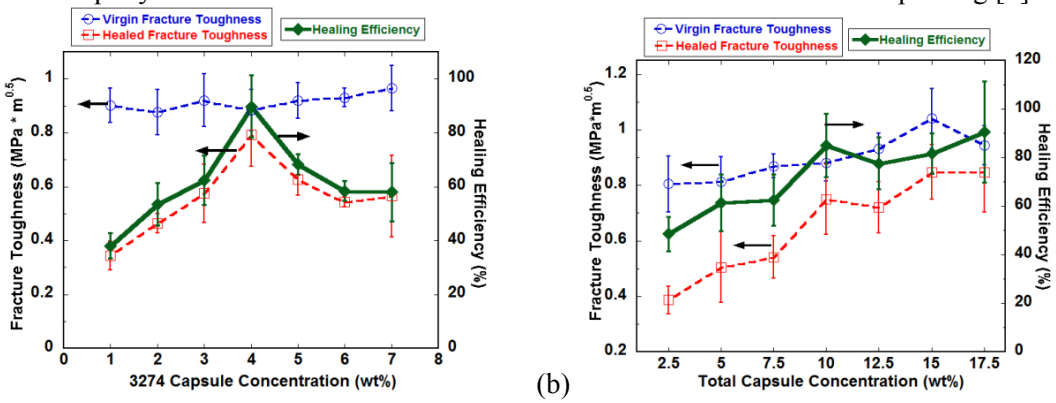

Figure 2. Fracture toughness of virgin and healed tests and corresponding healing efficiencies. (a) Effect of weight ratio of 3274 amine capsule to 815C on healing performance. (b) Effect of total microcapsules on healing performance.

A set of specimens containing 4 wt% 3274 amine capsules and 6 wt% 815C epoxy capsules was prepared and then either post-treated at 100 °C for 1 h to fully cure the epoxy matrix or at 121°C for various times. The fracture tests for specimens cured at 100°C for 1 h resulted in a healing efficiency of 49.8 ± 6.6%, while the healing efficiencies for specimens cured at 121°C decreased quickly from 85.1% to 45.9% after the first hour and held fairly steady to a final value of 35.1% after 8 hours. EPON 815C capsules should be stable until about 200 °C as evidenced by TGA analysis. Therefore, the reduction of self-healing efficiency might be due to the leakage/instability of the 3274 amine capsules since the TGA analysis indicates the shell wall does not provide a robust and stable shield to the core material. This is an important factor and will be thoroughly analyzed to determine improved methods of protection to retain high healing efficiency as the processing temperature is increased.

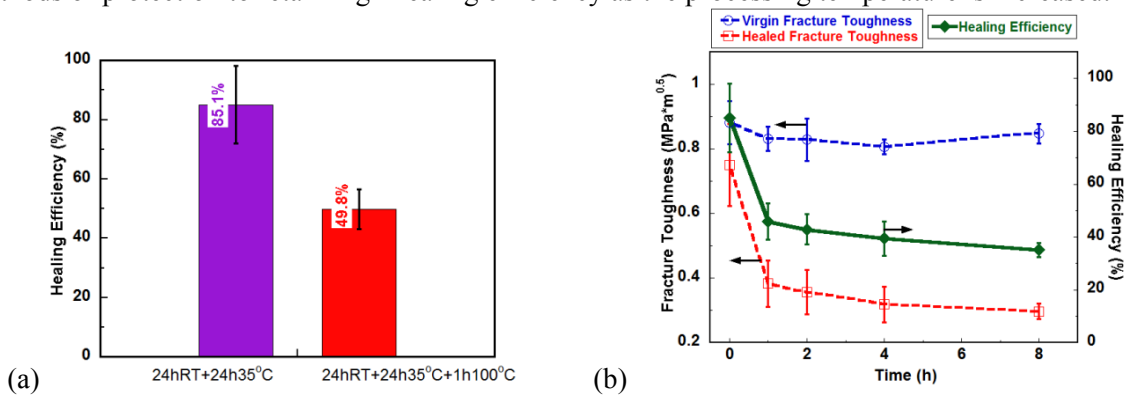

Figure 3. Healing efficiencies for specimens post cured at (a) 100°C for 1 hour, (b) 121°C for various times.

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