Delamination Toughening of Carbon-Epoxy Laminate with a Polyethylene-*co*-Methacrylic Acid (EMAA) Self-Healing Modifier

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Abstract

One promising new technology to repair delamination damage in composite materials is the use of so-called self-healing (or mendable) polymers. This paper presents research into the repair of delaminations in carbon fibre-epoxy using the mendable thermoplastic agent polyethylene-*co*-methacrylic acid (EMAA). This healing agent was able to restore the mode I interlaminar fracture toughness by a novel repair mechanism. The recovery to the interlaminar toughness after healing was dependent on the concentration of EMAA, and improvements of up to three times higher than the original toughness were achieved. The EMAA was also capable of restoring the mode I fatigue delamination resistance. The healing mechanisms responsible for the repair of delamination damage and restoration of the delamination resistance under static and fatigue interlaminar loading are described in the paper.

1. Introduction

Fibre-polymer composite materials used in aircraft structures are prone to multiple layers of delamination cracking from service-induced stresses from through-thickness loads, edge stresses, environmental degradation, and foreign object impact damage, such as bird strike and hail stone impact. Also, delamination growth in polymer matrix composites under cyclic through-thickness loading is a long-standing problem for composite structures subjected to in-service fatigue. Such delaminations can change from no growth to rapid crack growth (unstable crack growth) with relatively small changes in the applied loads because of the high sensitivity of fatigue growth rate to the stress intensity range. Delamination is the most common and, in most instances, the most severe type of damage to composite materials used in aircraft structures. Conventional repair methods for delamination damage including removal of the damaged material, mechanically fastened repairs, and adhesively bonded repairs which are expensive and time-consuming [1]. Furthermore, current damage tolerant design incorporates large margins in order to account for the reduction in structural performance due to delamination damage [2]. A notable example is the so-called 'no growth' design regulation applied to primary composite structures for civil and (many) military aircraft [3]. This regulation mandates that delamination cracks must not grow under fatigue loading over a specific period of in-service use. This can lead to overweight and inefficient structures; hence, increasing both manufacturing and operating cost. Hence, the aerospace industries both in civil and military sectors are continuously seeking new strategies to avoid these problems.

One promising new technology to repair delamination damage in composite materials is the use of so-called self-healing (or mendable) polymers. These polymers, which are typically thermoplastic-based materials, repair delamination damage by flowing into and filling the cracks when melted at elevated temperature. After this treatment the composite material is cooled to room temperature, and this will partially or completely heal delamination cracks and restore the mechanical properties of the damaged composite. These self-healing technologies comprise microcapsules [1, 4-7], microvascular fibres [2, 7, 8] or mendable polymers [1, 7, 9, 10] and each of them have different healing and toughening mechanisms, healing efficiency and repeatability [1, 11].

Recent research has revealed that poly[ethylene-*co*-(methyacrylic acid)] (EMAA) can be used as an effective mendable thermoplastic for epoxy matrix composites [12-14]. The self-healing mechanism and repair efficiency of EMAA in epoxy was studied by Meure et al. [13], and found that it possessed a unique healing mechanism that is different from any mendable polymer systems, by forming a discrete insoluble phase that reacts with epoxy resin at elevated temperature and thus, results in healing. Meure and colleagues [13] measured a healing efficiency of over 100% in the fracture load of damaged epoxy by the inclusion of EMAA as small second phase particles. A novel pressure delivery mechanism that involves a condensation reaction between the EMAA and epoxy resin at elevated temperature (about 150^{o}C) is the main healing mechanism [14, 15]. Hydroxyl-acid reactions catalyzed via tertiary amine at the EMAA-epoxy interface produces volatiles (e.g. water) that phase separate into tiny bubbles within the EMAA. The internal pressure within the bubbles increases as the volatiles expand during healing at high temperature and this causes the hot, viscous EMAA to flow into the cracks and other open flaws within the epoxy matrix. The internal cracks were healed by a strong adhesive bond forming between EMAA and epoxy when the EMAA solidifies upon cooling. The self-healing process using EMAA can be repeated multiple times without any significant loss in the healing efficiency [12, 13]. Meure et al. [12] also reported that self-healing repair of delamination cracks in carbon fibre-epoxy laminates can be achieved not only by using EMAA particles but also using EMAA fibres. The EMAA was able to recover more than 100% of the mode I interlaminar fracture toughness after healing laminates containing delamination cracks. Such healing efficiency is equivalent or greater than that reported for composites using the microcapsule or microvascular repair methods. Despite this substantial restoration in toughness, the interlaminar toughening mechanisms that cause such improvement are not well understood. Also, there is no research on the healing efficiency and toughening mechanisms of EMAA under interlaminar fatigue loading. To date, no studies have reported on the healing efficiency of fatigue delamination cracks in composite laminates using either capsulated, microvascular or mendable techniques.

This paper presents an experimental study into the self-healing repair of delamination cracks in a carbon fibre-epoxy laminate using mendable EMAA particles or fibres. This paper extends the original work by Meure et al. [12] by assessing the delamination crack growth process and interlaminar toughening mechanisms which result in the high self-healing efficiency of epoxy matrix laminates containing EMAA. The study identifies the mechanism responsible for the high recovery in delamination toughness of mendable laminates following self-healing using EMAA. Also, investigation on the repair of fatigue-induced delamination cracks and the recovery of the interlaminar fatigue properties of carbon fibre-epoxy laminate with EMAA was conducted. The healing efficiency was quantified by measuring changes to the delamination crack growth rate of the EMAA laminates before and after healing under mode I interlaminar cyclic loading. The capability of EMAA to repair fatigue cracks and restore the fatigue crack growth resistance was assessed for multiple healing cycles. The mechanisms controlling the repair of fatigue cracks and the restoration of the interlaminar fatigue properties were investigated. The paper also examines similarities and differences in the healing of delamination cracks which grow under static and fatigue mode I interlaminar loading conditions.

2 Materials and Experimental Methodology

2.1 Mendable carbon fibre-epoxy laminates

The self-healing properties of EMAA were evaluated for a 20-ply thick carbon fibre-epoxy laminate with a cross-ply ([0/90]) stacking pattern. The carbon was a plain woven fabric and the epoxy was diglycidyl ether of bisphenol A (DGEBA) mixed with triethyltetramine (TETA) at the stoichiometric ratio of 100:13 w/w epoxy to amine. Four types of carbon-epoxy laminate containing different amounts of EMAA fibres and one type of laminate containing EMAA particles were studied, and these materials are listed in Table 1.

The particles were produced by cryogenic grinding and were spherical in shape with diameters between 250 and 425 µm. The particles were blended into the uncured epoxy at 15% volume content. The other laminate contained a woven mesh of EMAA fibres. Plies of the mesh were located between the two middle plies of the laminate, which was the location of delamination crack growth. The EMAA was confined to this region, rather than being placed between every ply, because the self-healing repair efficiency was assessed for delamination cracking along the mid-thickness plane of the laminate. In addition, EMAA plies were located at the neighbouring ply of the middle plies to repair multiple delaminations in the event of crack branching. An unmodified laminate without EMAA was manufactured using the same carbon fabric and epoxy resin as the mendable laminates.

The laminates with and without EMAA were fabricated using the wet hand lay-up process. The laminates were cured and consolidated at 70°C and 2 MPa for 1 hr and then post-cured at 150°C for 30 mins at ambient pressure. The glass transition temperature of the epoxy after post-curing was 142°C (tan δ max). The carbon fibre content and thickness of the laminates are given in Table 1 and the mendable laminates were thicker due mainly to the volume occupied by the EMAA.

Table 1. Carbon fibre-epoxy laminates used to study the healing efficiency of EMAA.

Composite	**EMAA Self-Healing Agent**[1]	**Average Carbon Fibre Content (vol%)**	**Average Laminate Thickness (mm)**
Control laminate	No EMAA	40%	3.5
EMAA particle laminate	EMAA particles (250-425 µm size range): volume content of 15%	26%	5.0
EMAA fibre laminate – (2 ply/50 µm)	EMAA mesh with 2 plies of 50 µm diameter fibres	31%	3.7
EMAA fibre laminate – (2 ply/100 µm)	EMAA mesh with 2 plies of 100 µm diameter fibres	30%	3.9
EMAA fibre laminate – (4 ply/50 µm)	EMAA mesh with 4 plies of 50 µm diameter fibres	32%	3.9
EMAA fibre laminate – (4 ply/100 µm)	EMAA mesh with 4 plies of 100 µm diameter fibres	30%	4.0

EMAA added between two central plies and between these plies and their neighbouring ply

2.2 Static and fatigue interlaminar fracture toughness testing

The healing efficiency of the EMAA particles and fibres was quantified by measuring the recovery to the mode I interlaminar fracture toughness using the double cantilever beam (DCB) test. The DCB specimens were 130 mm long, 15 mm wide, and the unmodified and mendable laminate specimens contained a pre-crack that was 35 mm or 43 mm long, respectively. Both static and fatigue DCB tests were performed; under static loading the crack opening displacement was slowly increased monotonically (i.e. strain effects on the delamination toughness can be ignored) and under fatigue loading a cyclic crack opening displacement was applied. The static interlaminar fracture toughness test was performed by applying a crack opening load to the pre-cracked end of the DCB specimen at a constant crosshead (crack opening) displacement rate of 2 mm/min in close accordance to ASTM D5528-01(2007)e3 specifications.

The fatigue test was performed by applying a cyclic load to the DCB specimens in mode I displacement control at the frequency of 10 Hz. The R ratio, defined as the minimum crack opening displacement normalised to the maximum crack opening in one load cycle, was 0.1. The growth of the delamination was measured over a short length (typically 5-10 mm) under a constant fatigue stress intensity range (ΔG_I) to measure the average crack growth length per load cycle (d*a*/d*N*). The strain energy release rate range was varied between ~5 J/m^2 and ~1500 J/m^2 to measure the fatigue crack growth rate over ten orders of magnitude (i.e. d*a*/d*N* from 10^{-9} mm/cycle to 10 mm/cycle). This data was used to produce Paris fatigue curves for the unmodified and mendable laminates.

The mendable laminates were healed by heating to 150°C for 30 mins within an oven after static and fatigue DCB testing. The laminates were then retested under the identical DCB test condition to measure the healing efficiency of the EMAA under static and fatigue loading. The efficacy of the EMAA to repair the laminate and restore the interlaminar toughness properties was determined for five repetitions of the healing process, which were all performed under identical test conditions.

3 Results and Discussions

3.1 Healing of static delamination cracks

The static interlaminar fracture toughness (G_{Ic}) values for the unmodified and mendable laminates in their before healing condition are shown in **Fig. 1**.

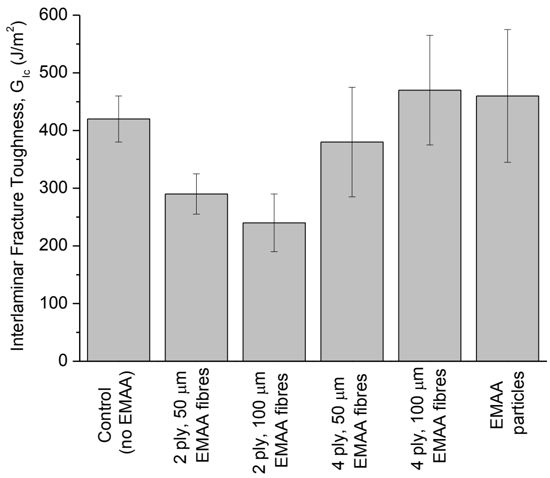


Fig. 1. Mode I critical strain energy release rates (G_{Ic}) for the control and mendable EMAA laminates in the original condition.

The fracture toughness of the laminate was not changed significantly by the EMAA particles or fibres. After thermally-activated healing at 150°C for 30 mins; however, the fracture toughness of the mendable laminates increased over 100% of their original toughness as shown in in **Fig. 2** (with the exception of the material with the lowest amount of EMAA [2 ply/50 µm fibres]).

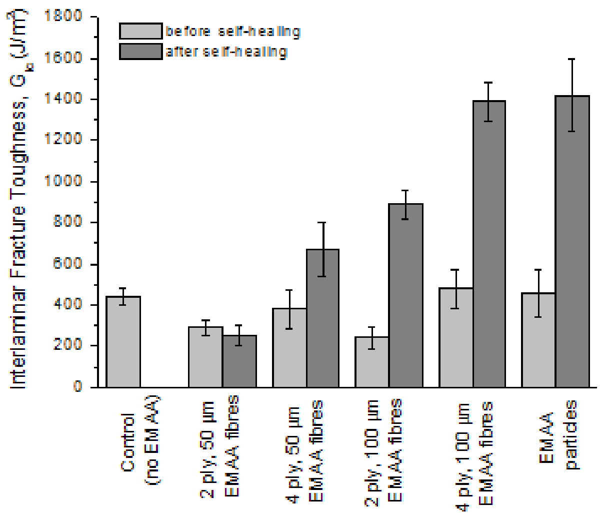
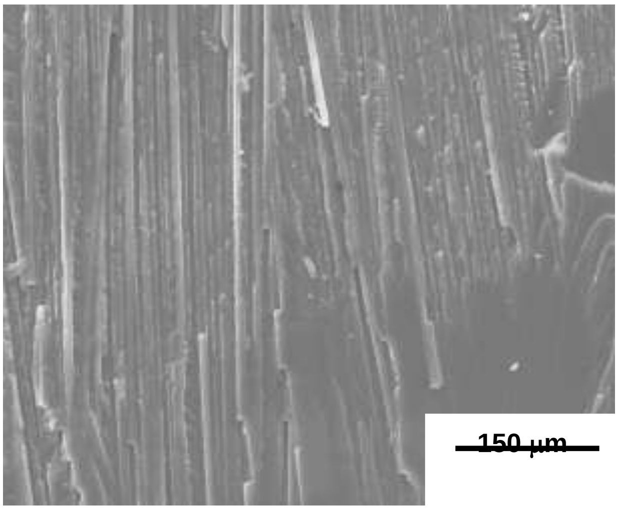


Fig. 2. Mode I interlaminar fracture toughness (G_{Ic}) for the control and mendable EMAA laminates in the original condition (before healing) and after self-healing.

This shows that the EMAA particles and fibres were both effective at healing the delamination crack and restoring the static fracture toughness. Moreover, **Fig. 2** also shows that healing efficiency, which is defined as the fracture toughness after healing compared to before healing, increased with the EMAA content, and at the highest concentration the recovery in toughness was over 300%. The high self-healing efficiency is consistent with the work by Meure et al. [12, 13] who measured a large recovery in the fracture load of epoxy resin and carbon fibre-epoxy laminates containing EMAA.

The cause of the high healing efficiency of the mendable laminate is the novel self-healing delivery mechanism of EMAA into delamination cracks. **Fig. 3** shows the delamination fracture surfaces of the laminate containing EMAA particles before and after healing. (Similar fracture surfaces were observed for the laminate containing EMAA fibres).

The fracture surface showed no obvious signs of the EMAA before healing, which was dispersed as small second-phase particles within the epoxy matrix (These particles are not easily observed due to the lack of phase contrast with the epoxy matrix). However, after healing, discrete phase regions of highly porous EMAA occurred on the delamination fracture surface. Meure et al. [13, 14] and Varley et al. [15] reported that hydroxyl-acid condensation reactions catalysed via tertiary amine occurs at the EMAA-epoxy interface at the healing temperature (150ºC in the present investigation). The reactions produce volatile by-products (e.g. water) that phase separate into tiny bubbles within the hot, molten EMAA. The high internal pressure of these bubbles assists in spreading the molten EMAA to fill up cracks.



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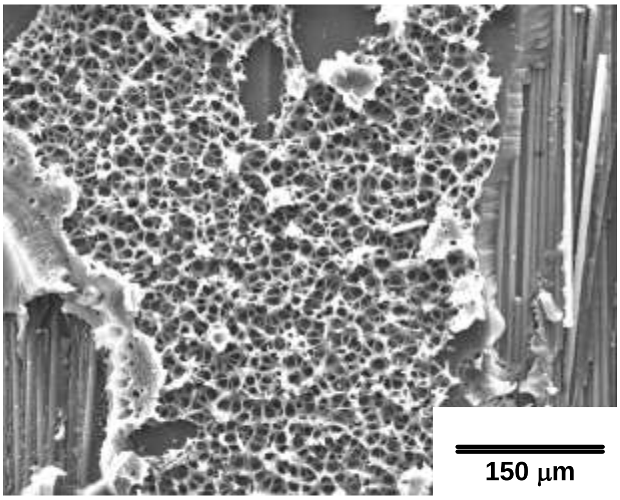
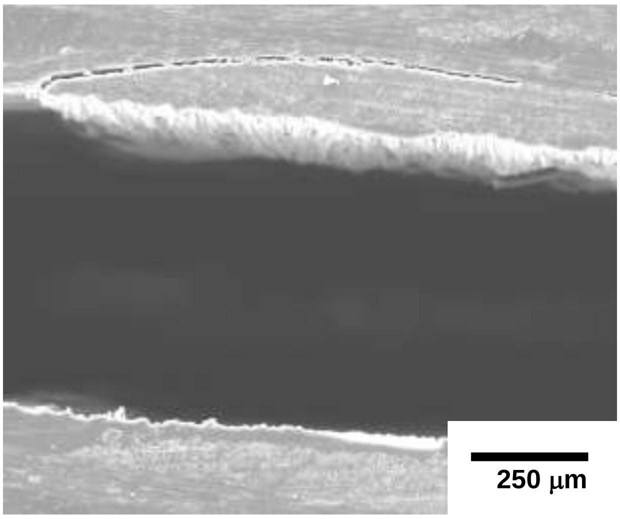


(b)

Fig. 3. Fracture surface of the mendable laminate (containing EMAA particles) (a) before and (b) after self-healing.

The increase of the fracture toughness of the mendable laminates after self-healing (**Fig. 2**) [except the 2 ply/50 µm fibre material] was due to the formation of large-scale bridging traction zone of EMAA along the delamination crack. **Fig. 4** shows the crack in a mendable laminate before and after healing.



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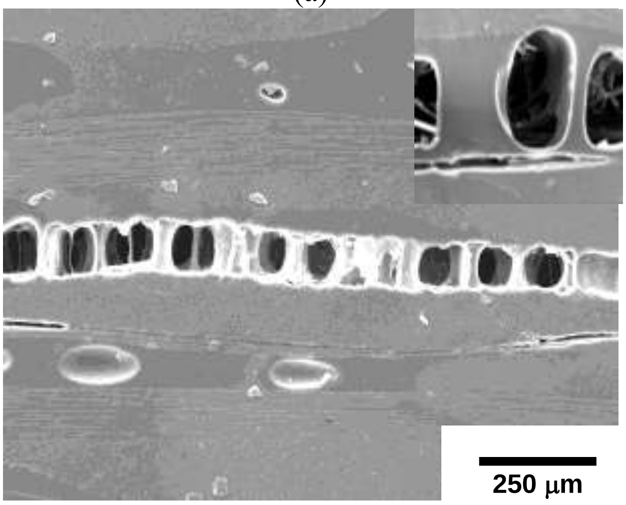


(b)

Fig. 4. Scanning electron microscopy images of the delamination crack in a mendable laminate (a) before self-healing and (b) after self-healing. The insert photograph in (b) shows a higher magnification view of the crack bridging ligaments. Note the absence and presence of crack bridging ligaments of EMAA before and after healing, respectively.

The delamination after healing was reformed by firstly repairing the original crack by thermal activation of the EMAA and then repeating the mode I DCB static test. Crack bridging was absent before healing; while after healing, high-density thin ligaments of EMAA bridged across the delamination. The EMAA adhered strongly to the delamination faces due to the formation of covalent bonds (via acid-oxirane and/or acid-hydroxyl reactions) and hydrogen bonds upon self-healing at the EMAA-epoxy interface [14, 15], and therefore crack growth occurred through the EMAA (cohesive failure) rather than along the EMAA-epoxy interface (adhesive failure). The EMAA was deformed in tension under increasing crack opening displacement. The bridging ligaments extended for a distance of 10-12 mm behind the delamination front until they broke at high crack opening displacement. The formation of bridging ligaments after healing (but not before healing) is attributed to the flow of EMAA along the delamination crack and the strong adhesive bond at the EMAA-epoxy interface during the healing process. Because EMAA is a ductile thermoplastic material which is capable of large-strain plastic deformation, bridging ligaments were formed behind the crack tip and these ligaments were able to transfer stress across the delamination and thereby lower the stress at the crack tip, resulting in increased static fracture toughness after healing.

The only mendable laminate that was unable to form a large-scale bridging traction zone after self-healing is the one with the least amount of EMAA (2 ply/50 μm fibre). The amount of EMAA in this laminate was sufficient to heal the delamination crack, but it was insufficient to increase the fracture toughness after self-healing (**Fig. 2**). Fractographic examination of this material revealed the absence of a large-scale crack bridging zone after self-healing. It is believed that this laminate contained an insufficient amount of EMAA to form bridging ligaments during crack growth, and so the fracture toughness was not increased above the original material. This shows that a minimum concentration of EMAA is required for interlaminar toughening of the laminate following self-healing.

The healing efficiency of the EMAA laminates was determined for five repair cycles, and the recovery to the static fracture toughness values after each repair are given in **Fig. 5**.

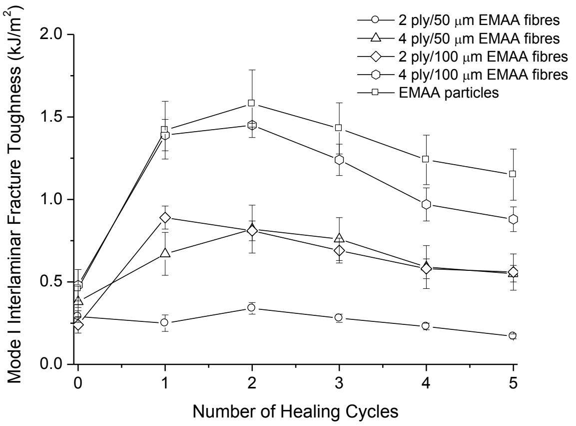
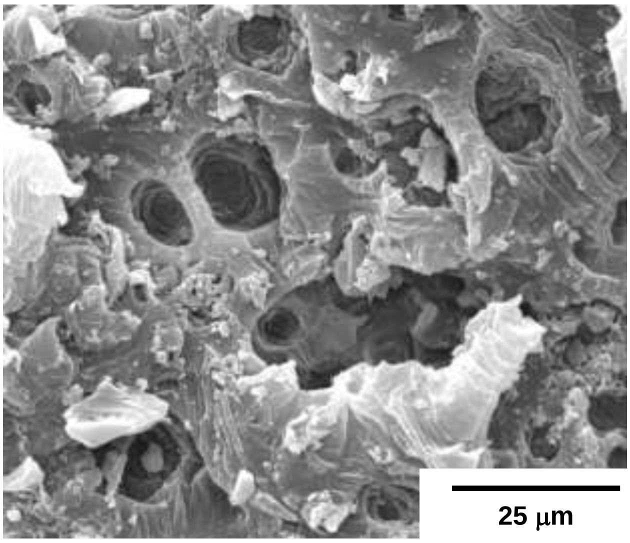


Fig.5. Effect of number of healing cycles on the mode I interlaminar fracture toughness (G_{IC}) of the mendable laminates.

The high fracture toughness gained with the first healing cycle was retained with the second and third healing cycles for both the laminates containing EMAA particles or fibres. However, the average fracture toughness value then declined with further healing cycles. Similar behaviour has been reported for the multiple healing of epoxy resin [13] and carbon fibre-epoxy laminate [12] containing EMAA. The decrease of the healing with the number of healing cycles is because the reactive compounds in the epoxy were depleted by multiple reactions processes with the EMAA. Fractographic analysis showed that after each healing cycle the EMAA formed second phase regions on the delamination fracture plane which then plastically deformed into bridging ligaments with repeated crack growth. For example, 6 shows planar and cross-sectional views of the delamination crack in an EMAA laminate following five healing cycles, and its appearance is similar to the crack after one healing cycle (**Fig. 4(b)**)

Hence, in each healing cycle, the capability of the EMAA rich regions to rebind with the epoxy matrix along the delamination fracture plane, and to then reform the bridging traction zone upon repeated delamination crack growth, is the controlling mechanism for the retention of high static fracture toughness with multiple healing.



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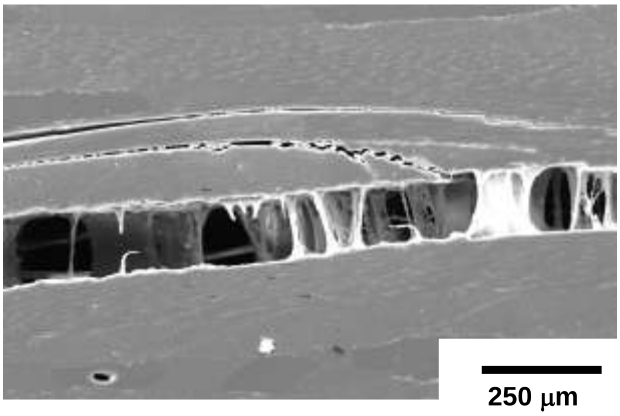


(b)

Fig. 6. (a) Fracture surface showing porous EMAA phase after five healing cycles. (b) EMAA bridging zone along the delamination crack after five self-healing cycles.

3.2 Healing of fatigue delamination cracks

The effect of mode I cyclic loading on the fatigue crack growth rate for the unmodified and EMAA laminates in their original condition is shown in **Fig. 7**.

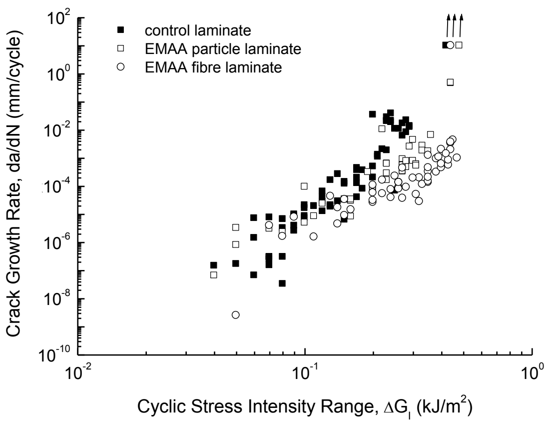
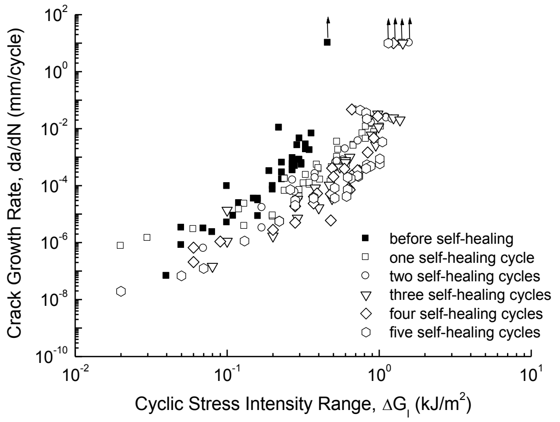


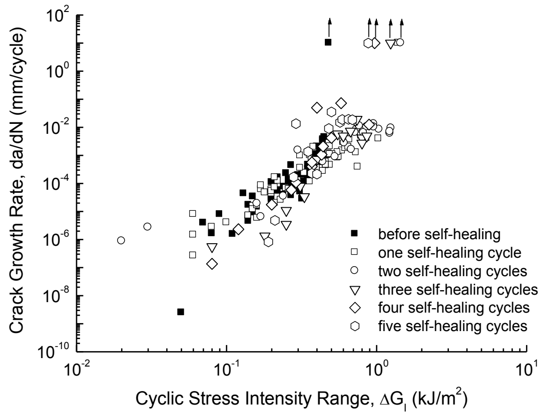
Fig. 7. Fatigue delamination crack growth curves for the unmodified and mendable laminates in the original condition.

The fatigue crack growth rate, da/dN, is the average distance of delamination crack growth per load cycle. Significant scatter in the fatigue crack growth rate data was measured, and this was because the delamination did not always grow at the same rate under a constant cyclic stress range, due to the slip/stick nature of the crack growth process. Both the unmodified and mendable laminates have similar curves with a linear log-log relationship between the delamination growth rate and the cyclic strain energy release rate ranging from 70 to 400 J/m^2. The fatigue strain energy release rate thresholds for the unmodified and mendable laminates were approximately the same (ΔG_I ~ 70 J/m^2), below which no crack growth occurred. The maximum cyclic stress intensity range to induce rapid delamination crack growth in the three laminates was also the same (ΔG_I ~ 400 J/m^2). The similarities revealed that EMAA in the original condition does not change the delamination fracture resistance under fatigue loading. This is consistent with the static fracture toughness values which were similar for the unmodified and EMAA laminates before healing (**Fig. 1**).

The effect of multiple healing cycles on the fatigue crack growth curves for the laminates containing EMAA particles or fibres are shown in **Fig. 8**.



(a)



(b)

Fig. 8. Fatigue crack growth rate curves for the mendable laminates containing (a) EMAA particles and (b) EMAA fibres before and after self-healing.

The EMAA was able to heal the fatigue crack upon heating the delaminated specimens at 150^{o}C for 30 mins. After the healing process, the delamination fatigue behaviour of the laminates was approximately the same as the unmodified laminate over most of the cyclic stress intensity range. These results indicate that EMAA is capable of fully restoring the fatigue crack growth resistance of the laminate. At high cyclic stress ranges (ΔG_I > 400 J/m^2), the healed laminates even exhibited enhanced fatigue properties, and this is likely due to the increase in the static fracture toughness of the mendable laminates. At different number of healing cycles, the fatigue crack growth curves were similar (within the bounds of scatter) and no evidence of small loss in static fracture toughness measured after the third healing cycle was observed in the fatigue behaviour.

From a fractographic examination of the laminates during interlaminar fatigue testing, no EMAA bridging zone occurred. For instance, **Fig. 8** shows the delamination fatigue crack in an EMAA laminate following healing, and no large-scale EMAA bridging zone was created behind the delamination front.

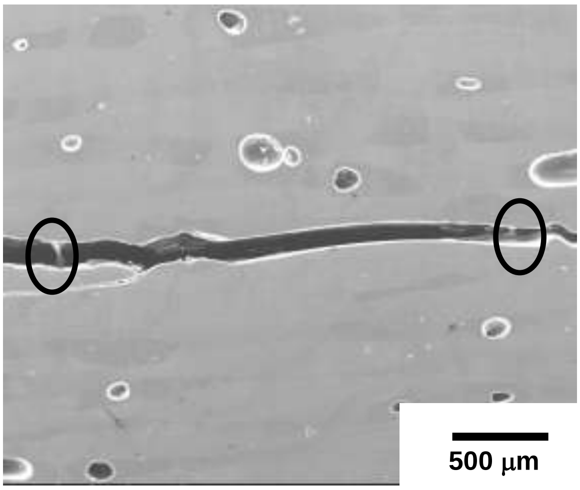


Fig. 5. Cross-section view of the fatigue delamination crack in a mendable laminate. Note the absence of a large-scale bridging traction zone. Only two thin bridging ligaments are visible, which are circled.

A very low density of bridging ligaments was observed along the fatigue crack, although these were much fewer in number than under static loading. The lack of improvement in the fatigue delamination resistance of the laminates after healing is therefore attributed to the short tensile fatigue life of the EMAA ligaments, which impeded the formation of a bridging zone. Fatigue failure occurred as a cohesive failure within the EMAA, and not an interfacial (adhesion) failure event at the EMAA-epoxy interface. The cause for the rapid fatigue failure of the ligaments has not been determined, although it is possible that the low-cycle fatigue failure of the EMAA ligaments under high strain cycling may be partly responsible. Furthermore, time-dependent (creep) plastic yielding of thermoplastics under tensile loads can lead to low rupture stress levels and short time to rupture. It is possible that creep-induced plastic flow of the EMAA under tensile fatigue loading causes the ligaments to permanently deform and break within a relatively small number of cycles; in other words, short loading time. This would explain the presence of ligaments under static loading (which is not time-dependent for the test conditions used here) and their absence under cyclic loading (which is time-dependent), although further investigation and analysis of the fatigue properties of the EMAA ligaments is required.

4 Conclusion

The mendable thermoplastic poly[ethylene-*co*-(methyacrylic acid)] is an effective agent for the healing of delamination cracks in carbon fibre-epoxy laminates formed under static or fatigue interlaminar loading. The addition of EMAA in the form of particles or fibre mesh was able to heal delamination cracks and increase the mode I interlaminar fracture toughness for carbon fibre-epoxy laminates. The self-healing efficiency increased with the EMAA content, and for the highest amount used in this study, the restored fracture toughness was more than double the toughness of the control laminate. EMAA is an effective self-healing agent because it flows over a large area of the delamination crack under the pressure delivery mechanism involving high-pressure bubbles. The EMAA formed a large-scale bridging traction zone behind the crack front which is the main toughening mechanism that promoted high static interlaminar toughness. The EMAA can reform this bridging zone with repeated self-healing and therefore the thermoplastic agent was able to repair and maintain high fracture toughness and high healing efficiency for multiple healing cycles.

Even though EMAA promoted high healing efficiency under static interlaminar mode 1 loading, it was less effective under fatigue loading. Nevertheless, the EMAA was able to fully recover the interlaminar fatigue resistance of delamination cracks. The laminates before and after multiple healing cycles possessed similar fatigue crack growth curves and this is due to the inability of the EMAA to form a large-scale bridging traction zone under cyclic loading, which occurs under static mode 1 loading.

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