BIOINSPIRED AND BIOMIMETIC SELF-HEALING OF ADVANCED COMPOSITE MATERIALS

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ABSTRACT

Lightweight, high strength, high stiffness fibre reinforced polymer composite materials are leading contenders to improve the efficiency and sustainability of many forms of transport. They offer immense scope for incorporating multifunctionality due to their hierarchical internal architecture. One limiting factor in their wider exploitation is relatively poor performance under impact loading, a crucial aspect of any safety critical design, leading to a significant reduction in strength, stiffness and stability. This results in conservative design and higher mass structures. Self-healing has the potential to mitigate damage resulting from impact, thereby improving design allowables or offering other benefits such as reduced maintenance and inspection schedules. The work presented in this paper shows that either compartmentalised hollow-fibre or continuous vascular network self-healing approaches can be used for the repair of advanced composite structures. In the nearer term, the specific placement of self-healing plies or individual fibres to match a critical damage threat has been shown to repair internal matrix cracking and delaminations throughout the thickness of a laminate when assessed in both a flexural and compressive loading state. In the longer term, integration of a pervasive, circulatory vascular network within the foam core of a composite sandwich structure has also been shown to offer a marked benefit. The network has negligible influence on structural performance whilst being able to provide reattachment of the foam core and laminate skin after impact damage. In the case studied, a sizeable recovery in flexural and compression after impact strength, and restoration of primary failure mode was observed. Such systems offer significant potential for restoring structural integrity to a composite component during service and prolonging residual life after a damage event.

1. INTRODUCTION

Advances in materials technologies have been largely responsible for major performance improvements in many engineering structures and continue to be key in determining the reliability, performance and cost effectiveness of such systems. Lightweight, high strength, high stiffness Fibre Reinforced Plastic (FRP) composite materials are leading contenders to improve the efficiency and sustainability of many forms of transport. In addition, they offer immense scope for incorporating multifunctionality due to their hierarchical internal architecture. One limiting factor in their wider exploitation is relatively poor performance under impact loading, a crucial aspect of any safety critical design, leading to a significant reduction in strength, stiffness and stability. For example, Carbon Fibre Reinforced Plastic (CFRP) used in aerospace applications is typically assigned an allowable strain level of <0.4% [1,2] whereas commercially available carbon fibres typically have a strain to failure of around 1.5%. This results in conservative design and higher weight structures. The inability to plastically deform results in energy absorption via the creation of matrix cracks and delaminations which can be difficult to detect visually. Self-healing has the potential to mitigate damage resulting from an impact event, thereby providing an opportunity to improve the design allowables for FRP’s or offer other benefits such as reduced maintenance and inspection schedules.

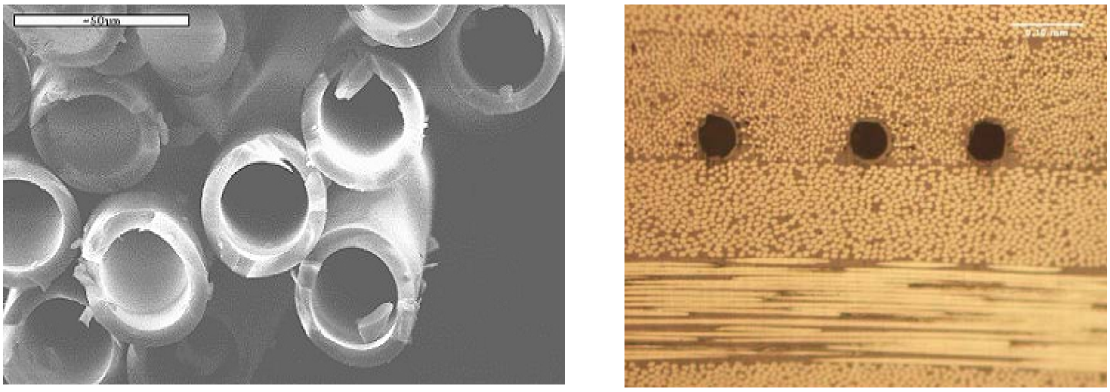
Conceptual inspiration from nature is not new, and many engineering approaches have been inspired by observing natural systems. The healing potential and repair strategies of living organisms is increasingly of interest to designers seeking lower mass structures with increased service life who wish to progress from a conventional damage tolerance philosophy. Naturally occurring ‘materials’ have evolved into highly sophisticated, integrated, hierarchical structures that commonly exhibit multifunctional behaviour [3]. Bioinspired self-healing using hollow fibres embedded within a composite structure has been investigated at different length scales in several materials by various authors [4-7]. Hollow glass fibres are used in preference to embedded microcapsules [8-15] because they offer the advantage of being able to store functional agents for self-repair as well as integrating easily with and acting as a reinforcement [16-20]. A typical hollow fibre self-healing approach used within composite laminates could take the form of fibres containing a one-part resin system, a two-part resin and hardener system or a resin system with a particulate catalyst or hardener dispersed within the matrix material [7].

Many of the current self-healing approaches being studied are likely ultimately to be limited by the volume of healing agent available, or will only be capable of healing a single or few repeated damage events. In the longer term, self-healing via a vascular system potentially offers a replenishable healing system that can be tailored for individual design cases and has the capacity to fill large damage volumes [21]. Pilot studies on vascular self-healing have been reported for polymer coatings [22] and in composite sandwich structures [23]. Likewise, studies are underway to help design and implement such vascular networks [24,25]. Vascular self-healing offers enormous potential in sandwich construction because this configuration is susceptible to low-velocity impact of blunt objects. A skin-core disbond and void of considerable volume is formed under the impact site [26] and the residual compressive strength can be reduced by over 25% [27-30]. A low density, closed-cell foam core is the ideal host for a vascular network with the brittle fracture of the skin-core disbond releasing the liquid healing components [23]. This work is discussed in a colleagues paper [31].

2. HOLLOW GLASS FIBRES FOR SELF HEALING

A bespoke Hollow Glass Fibre (HGF) making facility [32,33] is used to produce HGF between 30-100μm diameter with a hollowness of around 50%, Figure 1a). These are embedded within either Glass Fibre Reinforced Plastic (GFRP) or CFRP , Figure 1b), and infused with uncured resin to impart a self-healing functionality to the laminate. During a damage event some of these hollow fibres will fracture, thus, initiating the recovery of properties by ‘healing’ whereby a repair agent passes from within any broken hollow fibres to infiltrate the damage zone and acts to ameliorate the critical effects of matrix cracking and delamination between plies and, most importantly, prevent further damage propagation. This release of repair agent mimics the bleeding mechanism in biological organisms (e.g. human thrombosis).

The self-healing fibres can be introduced within a laminate as additional plies at each interface, at damage critical interfaces or as individual filaments spaced at predetermined distances within each ply. The precise configuration will depend upon (1) the nature and location of the damage, (2) the type of repair resin, and (3) the influence of the operational environment. In order to understand and optimise the healing process, two studies were undertaken in both glass and carbon reinforced epoxy systems respectively. A translucent glass/epoxy laminate provides good visualisation of both damage occurrence and the healing process when viewed with transmission microscopy, however, a carbon/epoxy laminate is opaque and therefore, to enhance visualisation, an UV fluorescent dye (Ardrox 985) was added to the healing resin in both studies.



a) b)

Figure 1: a) Typical hollow glass fibres (30µm diameter and 50% hollowness). b) Hollow glass fibres embedded in a fibre reinforced polymer laminate

3. SPECIMEN MANUFACTURE

In the manufacture of the self-healing GFRP, the HGF chosen had an external diameter of 60 µm ± 3 µm and an internal diameter of ~40 µm yielding a hollowness fraction (ratio of internal to external area) of ~55%. This larger fibre diameter (compared to Figure 1) gives a greater volume for healing agent storage. Once manufactured the individual fibres were consolidated within a resin film (913 epoxy, 42gsm, Hexcel Composites), which was selected to match the baseline laminate material. The healing resin is infused into the individual filaments using a vacuum assisted capillary action. Once the ends have been sealed (Bostik BondFlex 100HMA high modulus silicone sealant) the infused hollow fibre layers (which can now be considered as standard 'prepreg' plies) are incorporated into a laminate stacking sequence as required, and processed according to the resin film manufacturer's guidelines. A 16 ply composite laminate with a $[0°/+45°/90°/-45°]_{2s}$ stacking sequence manufactured from pre-impregnated E-glass/913 epoxy resin (Hexcel Composites) was selected for the first evaluation of the HGF self-healing approach. Self-healing filaments were introduced at four 0°/45° damage critical ply interfaces that were identified and reported previously [18]. An epoxy resin system (Cycom 823, Cytec) was selected as the healing resin for the following; compatibility with host laminate, availability as a two-part system permitting inclusion in separate storage filaments, low viscosity profile, and time to gelation of 30 minutes after mixing. Furthermore, it was observed experimentally that the individual components of the two-part Cycom 823 were sufficiently robust to survive the host laminate curing process (120°C for 1 hour) following infiltration into the embedded hollow fibres. Panels 200mm x 200mm x 2.5mm were prepared according to manufacturer's instructions. Each panel was then sectioned into coupons 20 mm x 50 mm for testing. After cutting, the edges of the samples are sealed with a two-part rapid curing epoxy system (Araldite Rapid) to prevent any healing resin loss through the exposed ends of the hollow fibres.

In the case of the CFRP self-healing laminates, an alternative laminate stacking sequence and distribution of HGFs to that above was investigated. The driver in this case being to minimize any detrimental effects on the higher performance of the CFRP whilst providing an effective self-healing function. Thus, the incorporation of HGF as discrete plies was deemed unsuitable for CFRP and a less intrusive approach was devised whereby a small number of individual HGF's were distributed within individual plies (Figure 1b)) to act as dispersed storage vessels for the healing agent. The distribution of HGFs within a CFRP laminate poses the challenge of balancing disruption to the host laminate architecture against delivery of adequate healing function. For the flexural loading specimens, HGF's were located at two 0°/45° interfaces with a spatial density of 70µm or 200µm respectively, to investigate the effect of HGFs on host laminate performance and the healing efficacy of different healing agent volume. For the CAI testing HGF were distributed at five 0°/45° interfaces with a mixture of spatial densities of 70µm and 140µm. This increase in the number of HGF locations and spatial density for the CAI testing arose from a need to provide greater healing resin volume at close proximity to the larger delaminations typically created after impact near the back face, whilst also providing sufficient healing resin to address the less significant damage towards the impact surface.

Pre-impregnated carbon fibre/epoxy resin (T300/914 Hexcel Composites) was selected as the host material. A quasi-isotropic stacking sequence of 16 plies was prepared as a 230mm x 160mm x 2.5mm plate. For the flexural testing, HGF was located at two 0º/-45º interfaces within the lay-up as follows:

(-45°/90°/45°/0°/HGF/-45°/90°/45°/0°/0°/45 °/90°/-45°/HGF/0°/45°/90°/-45°)

For the CAI testing, the following stacking sequence with healing potential at five interfaces with variation inf HGF spacing:

(-45°/90°/45°/HGF_{70}/0°/HGF_{70}/-45°/90°/45°/HGF_{70}/0°/0°/HGF_{140}/45°/90°/45°/0°/HGF_{140}/45°/90°/-45°)

The inclusion of HGF within the CFRP stack was such that short lengths (10-20mm) of exposed HGF protruded from the panel edges. This facilitated the vacuum-assisted infiltration of the HGF, after cure of the host laminate, with a two-part epoxy resin (Cytec Cycom 823) immediately prior to testing.

4. MECHANICAL TESTING

4.1 Flexural testing of GFRP self-healing laminates

Four-point bend flexural strength testing according to ASTM D6272–02 [34] was selected to characterize the strength and stiffness of the baseline and self-healed specimens. A support span to depth ratio of 32:1 with the loading noses positioned at one third of the span was selected. The tests were conducted using a loading rate of 5mm/min on a Roell Amsler hydraulic test frame fitted with a 25kN load cell. A Linear Potentiometric Displacement Transducer (LPDT) was used to record mid-span deflection, which was logged through a PC running Instron data acquisition software.

For all damaged samples, a three-point bend quasi-static indentation using a 5mm diameter spherical tup mounted on a Hounsfield H20K-W (20kN load cell) electromechanical test machine under load control (peak load limited to 2500N) was used to simulate low velocity impact damage by initiating matrix shear cracking and sub-critical delamination damage within the laminate prior to flexural testing. The sample was supported by a steel ring of 27mm outer diameter and 14mm inner diameter. Up to this load level the damage is contained within the laminate and can be likened to BVID: surface indent <0.3mm), minimal back face distortion/fibre break out. Furthermore, the shear crack/delamination distribution within the laminate forms the characteristic ‘pine tree’ distribution as would be expected from a low velocity drop weight impact event.

In the case of the laminate containing the self-healing function, this was permitted to heal by exposure to 100°C (from ambient) in an air circulation oven for a period of 2 hours. This was deemed acceptable as it allowed some degree of control and repeatability during the study and simulated the environment of the intended aerospace application. The flexural strength results for the five different laminate configurations (baseline, baseline + damage, self-heal + no damage, self-heal + damage, and self-heal + damage + healing) are given in Table 1.

Table 1: Summary of flexural strength and healing efficiency for self-healing GFRP

Specimen ID	**Specimen description**	**Flexural strength [MPa]**	**% retained strength**
Group A	Baseline laminate {[0°/+45°/90°/-45°]$_{2s}$} - no damage	668 ± 13	100
Group B	Laminate with self-healing plies at critical 45°/90° interfaces - no damage	559 ± 12	84
Group C	Damaged (2500N indentation) laminate	479 ± 32	72
Group D	Damaged laminate with self-healing plies - no healing	494 ± 7	74
Group E	Damaged laminate with self-healing plies - 2 hours self-healing at 100°C	578 ± 28	87

Table 1 indicates that the inclusion of hollow fibres gives an initial strength reduction of 16% (Group A vs. Group B). It was found that Group A and Group B had comparable low energy impact damage tolerance both in terms of damage size and residual failure strength viz. Group C and D respectively . After healing of the damage site was undertaken it was found that Group E had a residual strength of 87% compared to Group A and 100% compared to Group B.

4.2 Flexural testing of CFRP self-healing laminates

As for above, a support span to depth ratio of 32:1 and a support span to load span ratio of 3:1 were selected according to ASTM D6272-02 [34], which resulted in specimen dimensions of 100mm x 20mm x 2.6mm being cut from a plate with the use of a water-cooled diamond grit saw. Immediately prior to mechanical testing, the HGF within each specimen was infiltrated with a healing agent of pre-mixed, two-part epoxy resin (Cytec Cycom 823) using a vacuum assist capillary technique.

Quasi-static impact damage was imparted to each specimen as per the procedure described above. The indentations were limited to a peak load of 2000N. After indentation, the specimens were subject to 70ºC for 45 minutes to reduce healing resin viscosity (25cps) and facilitate infiltration into damage sites, Figure 2. This was followed by a cure schedule of 125ºC for 75 minutes. Whilst this process diverges from the original aim of achieving an autonomic healing function, the use of a pre-mixed healing resin and elevated temperature after a damage event allowed the Cycom 823 to reach an optimum cure status and for the study to demonstrate the highest level of healing efficiency possible with this system. Cycom 823 is not designated a 'healing agent' and was chosen as being the best available commercial system to fulfill this role.

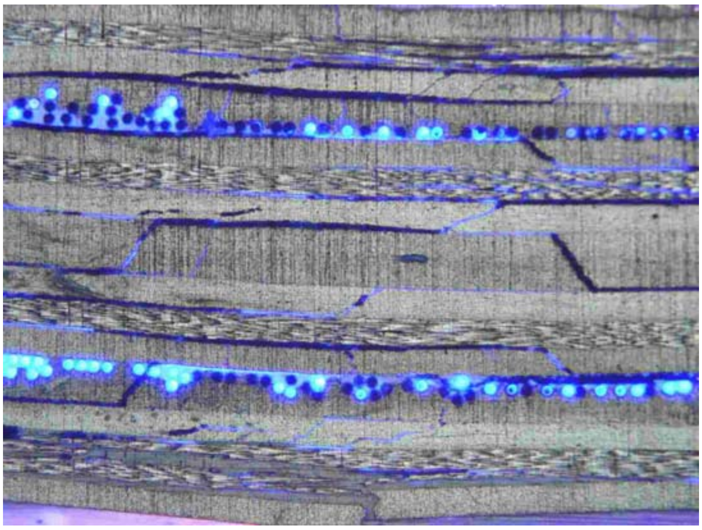


Figure 2: Cross-sectional view of a CFRP laminate with HGF at two 0º/-45º interfaces. A fluorescent dye has been mixed with the repair resin to enhance healing visibility.

After healing, test specimens were mounted on a Roell Amsler HCT25 electro-mechanical test machine with roller spacing determined by specimen dimensions and ASTM D6272-02 [34]. An Instron 8800 controller/data-logger was used to control the test machine and record data. Specimens were loaded to catastrophic failure and were monitored to ensure a consistent failure mode with optical microscopy being used to record detailed observations. Results were obtained from ten undamaged, five damaged and five healed specimens.

The results of the four point bend flexural testing are shown in Table 2. Comparisons are made between the performance of undamaged, damaged and healed specimens for the two HGF spatial densities alongside a baseline CFRP laminate with no HGF. The results show that at high HGF spatial density (70µm) a small reduction (8%) in flexural strength is incurred by their inclusion, attributed to the resin rich regions and disruptions in the laminate generated by HGF clumping. However, this configuration can also be seen to exhibit a degree of increased damage tolerance (i.e. higher residual strength after damage) compared to both the lower spatial density (200µm) HGF and the baseline laminate. This is primarily attributed to the energy absorbed in crushing and fracture as crack fronts propagate through HGF. The detrimental effects of HGF inclusion are outweighed by the strength recovery achieved upon healing which exhibits a recovery of 97% of a laminates undamaged strength. This suggests that any increase in healing resin storage volume provided by a higher HGF volume fraction is beneficial for the recovery of strength and combined with an increase in damage tolerance, somewhat mitigates the initial greater reduction in mechanical performance of the host structure.

Table 2: Effects of HGF and healing on flexural strength of CFRP

Specimen type		**Undamaged (CV%)**	**Damaged (CV%)**	**Healed (CV%)**
Plain CFRP	Flexural strength (MPa)	583.3 (2.3)	405.0 (16.2)	-
	% of undamaged baseline	*100%*	*69%*	-
HGF spaced @70µm	Flexural strength (MPa)	534.9 (2.4)	443.7 (10.7)	519.6 (5.5)
	% of undamaged baseline	*92%*	*76%*	*89%*
HGF spaced @200µm	Flexural strength (MPa)	568.8 (3.3)	401.0 (13.2)	466.6 (4.7)
	% of undamaged baseline	*98%*	*69%*	*80%*

4.3 Compression testing of CFRP self-healing laminates

CFRP panels (300mm X 300mm x 2.6mm) incorporating HGF (prepared as described in section B above), were infiltrated with a healing agent of pre-mixed, two-part epoxy resin (Cytec Cycom 823) using a vacuum assist capillary technique immediately prior to being subject to low velocity impact damage using an instrumented impact tester (Instron Dynatup). The impact boundary conditions were determined by guidelines proposed by Prichard and Hogg [35] which outline test conditions in close accordance with other published methods for impact and CAI. However, they propose modifications to allow smaller, thinner laminates (<3mm) to be tested. An impact energy level of 3J was selected by assessing the response of the laminate to a range of energy levels (1J negligible damage - 6J significant back face break out). A hemispherical tup of radius 20mm and impactor mass of 5.27kg struck laminates clamped between two 10mm steel plates with a circular impact window of 40mm diameter. Immediately after impact, laminates were subject to a healing cycle by being placed in an oven at 70ºC for 45 minutes to reduce healing resin viscosity (to 25cps) and facilitate infiltration into damage sites. This was followed by a cure schedule of 125ºC for 75 minutes.

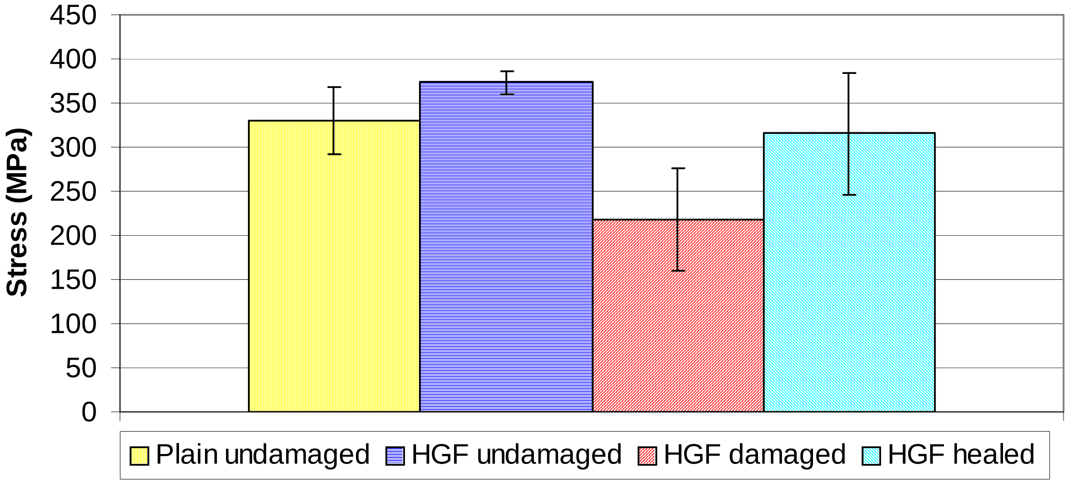


Figure 3: Results for CAI testing of CFRP self-healing laminates with HGF spaced at 70µm and 140µm on five 0°/±45° interfaces.

CAI testing was conducted using an Instron test frame with 250kN load cell. Five samples of each configuration were loaded into a ASTM D7137 test rig modified in accordance with Prichard and Hogg [35] to allow samples of 89mm x 55mm x 2.6mm to be tested. Samples were loaded at a rate of 0.4mm/min until failure. The results from CAI testing are shown in Figure 3 which indicates that under compressive loading there was no apparent reduction in failure stress due to the presence of HGF. The specimens damaged at an impact energy of 3 Joules consistently failed between 60% and 70% of the undamaged failure stress by a process of localized buckling at the mid-section. Coupons that were healed after 3 Joules impact were able to sustain loads up to and exceeding the undamaged failure stress.

5. CONCLUSIONS

This study has shown that a hollow fibre self-healing approach can be usefully employed in the repair and strength recovery of advanced composite structures. Hollow glass fibres containing a two-part epoxy healing resin can be manufactured and incorporated within conventional autoclave processing, indicating that this healing approach is applicable to existing composites manufacturing techniques. The specific placement of self-healing plies or individual fibres to match a critical damage threat has been shown to repair internal matrix cracking and delaminations throughout the thickness of a laminate when assessed in both flexural and compressive loading conditions. Such a system offers significant potential for restoring structural integrity to a composite component during service and prolonging residual life after a damage event.

ACKNOWLEDGMENTS

The authors would like to thank the UK Engineering & Physical Sciences Research Council for an Advanced Research Fellowship (GR/T03383/01) for I.P. Bond, and for research grants (GR/T17984/01 and GR/T03390/01) for R.S. Trask and G.J. Williams.

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