Damage development under fatigue loading in open hole composites with vasculesserving as self healing reservoirs

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Abstract

In the direction of self healing composite materials, several conceptual approaches have been developed for the implementation of self-healing functionality. The current study outlines the investigation for the damage development around the center hole of 6 mm in diameter vascularized composite specimens, under tension tension fatigue loading. Carbon and glass fiber reinforced polymer prepreg systems were compared regarding the damage progression. Interrupted fatigue tests were performed. Comparative results obtained among the different materials and discrepancies were observed regarding the number of the integrated vascules.

Ultrasonic C-Scan technique was applied at certain number of loading cycles during damage accumulation in order to determine at which level detectable damage started to develop and also at which level the damage reached the position of the vascules.

Once this process was completed, a premixed low viscosity epoxy healing system was manually injected through a syringe within the vascules. Infrared Thermography was applied to visualize the damaged area, the vascules and the injection process. After healing system cure cycle was completed, specimens were tested at the same conditions for determining how the healing process affects the composite in terms of the total cycles until failure.

1. Introduction

The interest about composite materials have grown rapidly since their introduction in a variety of application fields, one of which is the aerospace industry. However, besides their exponential growth, polymer composites are susceptible to damage in the form of micro cracking and delaminations generated mostly during their service. The damage accumulation degrades the mechanical performance of the material and leads often to catastrophic failure. In light of these issues, scientists inspired from biological systems and designed structural materials with a recovery mechanism triggered by the damage itself. Several approaches have successfully demonstrated the recovery of the mechanical properties of materials with self healing functionality. Norris et al. [1] described in detail how the presence of microvascular channels influences impact damage and subsequently the vascule-damage connectivity in a carbon fiber composite. Furthermore, crack-vascule interactions were investigated by Norris et al. [2], under Mode I, II loading in glass fiber composites. Regarding the fatigue loading conditions A.R, Hamilton et.al [3] studied the fatigue response of an epoxy matrix containing vascules. They concluded that the self healing response is most effective at slower propagating cracks. A step forward towards to quasi isotropic laminates based on fiber reinforced polymers (FRPs), O.J. Nixon-Pearsonet et.al [4] focused on the damage development in open hole

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composite specimens with no vascules. They claimed that failure damage propagates across the width of the hole and catastrophic failure occurs when the delamination propagates back to the grips at the -45/0° interface. Towards the same direction Zhang Yongbo et al. [5] reported the damage evolution in open hole carbon fiber reinforced specimens under fatigue conditions. The study showed that the cracks initiated at the edge of the hole and the free edge of the samples before the final fracture. A considerable amount of research has been carried out covering the fields of open hole fatigue testing in FRPs or the mechanical behavior of FRPs with vascules. The present study aims at combining new insights in the field of open hole quasi isotropic carbon fiber reinforced (CFRPs) and glass fiber reinforced (GFRPs) material systems, with vascules. Two different configurations were tested including either two vascules at the mid plane or two below and above of it (four in total). Tension tension fatigue tests were conducted for all concepts. Damage development was affected by the presence of the vascules. Ultrasonic C-Scan technique was used to visualize the damage progression, which varied due to the different material system (CFRP, GFRP) and the number of the integrated vascules (two or four) at the different layers. Infrared (IR) Thermography was used as an additional demonstration tool in order to visualize the vascules along the specimen, the damage development and to capture the process of the healing system injection.
The effect of healing efficiency on fatigue behavior in terms of fatigue lifetime and properties recovery are beyond the scope of this study and it will not be considered further.

2. Experimental section

2.1 Materials

Unidirectional carbon and glass low temperature epoxy prepreg system with an areal density of 200gr/m^2, supplied by Gurit, was used as the main reinforcement of the composites. The vascule forming material was stainless steel wires, commercially available at 0.5 mm diameter. The low viscosity resin-hardener system (HT2-HT2), supplied by R&G was selected as the healing system to be injected within the vascules.

2.2 Manufacturing process

Glass/epoxy and carbon/epoxy quasi-isotropic plates (284 mm x 240 mm x[4 mm for GFRP and 2.4 mm for CFRP]) were manufactured (8 plies $[45°/90°/-45°/0°]_S$) and sealed on an aluminium tool plate under a vacuum of 660-710 mmHg. Curing was undertaken according to the manufacturer's recommendations (100°C for 100 minutes under a continuous vacuum) in a program controlled oven. Stainless steel wires (0.5 mm in diameter) pre-coated with a polytetrafluoroethylene (PTFE) release agent were placed in pre-cut 0.5 mm channels. Vascular specimens containing two vascules were embedded at a pitch of 20 mm centered on the specimen centerline in the 0° plies located on the midplane. Vascular specimens containing four vascules were embedded at a pitch of 20 mm centered on the specimen centerline in the -45°/90° interface located either side of the midplane. Stainless steel wires were manually pulled out from the cured composite laminate. Table 1 summarizes the under investigation cases.

Table 1. Summary of the case studies

Composite Material	Specimen Configuration
CFRP (Case 1)	Reference with no vascules Two vascules at mid plane Two vascules above and below mid plane
GFRP (Case 2)	Reference with no vascules Two vascules at mid plane Two vascules above and below mid plane

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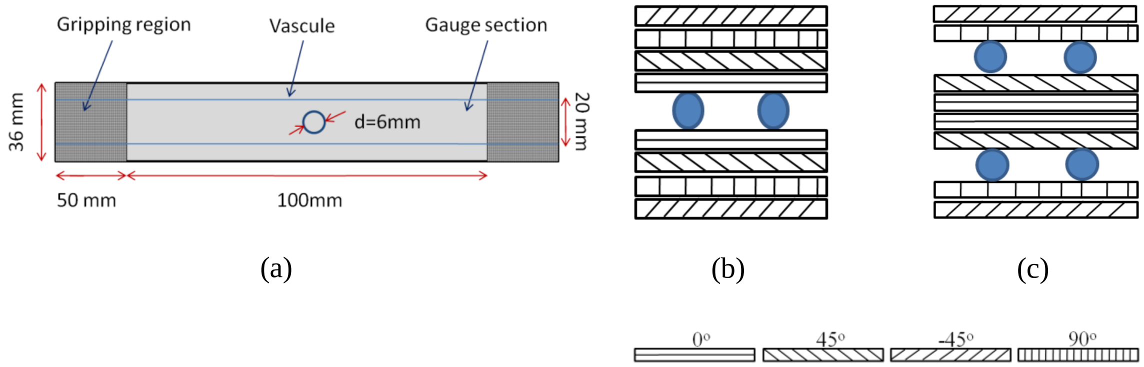
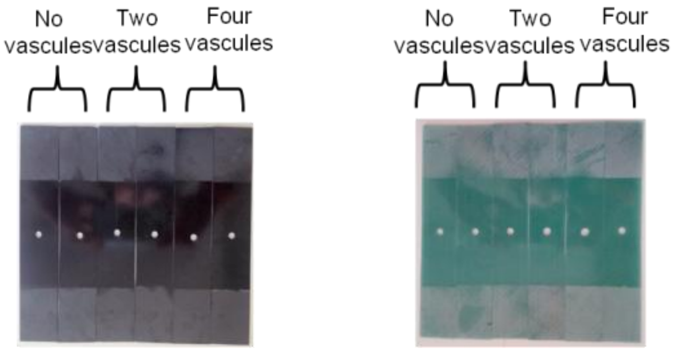
Cured composite plates were cut into open hole tension (OHT) coupon specimens (according to ASTM D5766) using a water-cooled diamond grit saw (200 mm x 36 mm x [4 mm for GFRP and 2.4 mm for CFRP]). A centrally located 6mm hole was drilled using a composite drill bit prior to testing. Figure 1 present in details the specimen characteristics while on Figure 2 the final specimen configuration can be observed.



(a) (b) (c)

Figure 1. a) OHT specimen geometry, Cross section schematic for each case study b) two vascules, c) four vascules



(a) (b)

Figure 2. Representative samples a) CFRP, b) GFRP

2.3. C-Scan

Ultrasonic C-Scan technique was employed for the quality control of the manufactured composite plates and the visualization of the damage development. The equipment consists of a MISTRAS Group AD-IPR 1210-PCI card and a VUB2000 tank. The transducer was a Krautkramer single element probe at 5 MHz, non-focal. The color bar presents the signal response from the weakest (green), at areas where damage occurs, to the strongest (red) which indicate no major defects within the material. The quality of the produced composites, as shown in Figure 3, were considered acceptable in order to continue the experimental campaign.

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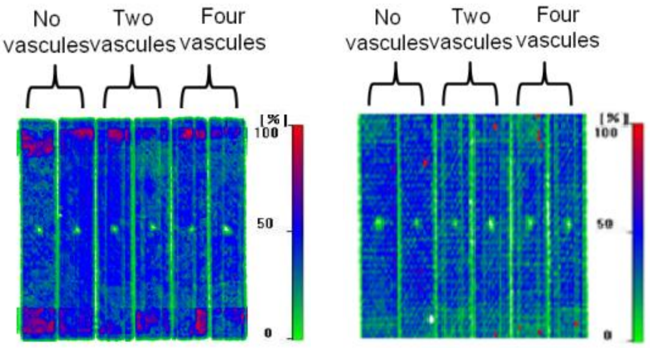

(a) (b)

Figure 3. C-Scan visualization ofrepresentativesamples a) CFRP, b) GFRP

2.4. Infrared Thermography

Thermographic inspection was carried out using an IR FLIR system (SC660) with a 640x480 IR pixel detector and thermal sensitivity ≤45mK. A halogen lamp was used to apply a step heating (long pulse) to the specimen, while its surface temperature was monitored as a function of time (Fig. 4a). Indicative captured images are presented below. In Figure 4b, the evolution of damage can be observed as occurs from the hole to the free edges. On the second case (Fig. 4c), the damage development in an early stage along with the vascules can be both visualized. The healing system while being injected after damage has occurred, is detected in Figure 4d. The area of damage is significantly different as the heat absorption is greater however the injection process is barely visible. This can be attributable to the direct temperature equilibration of the injected material with the composite and thus temperature differentiations cannot be captured.

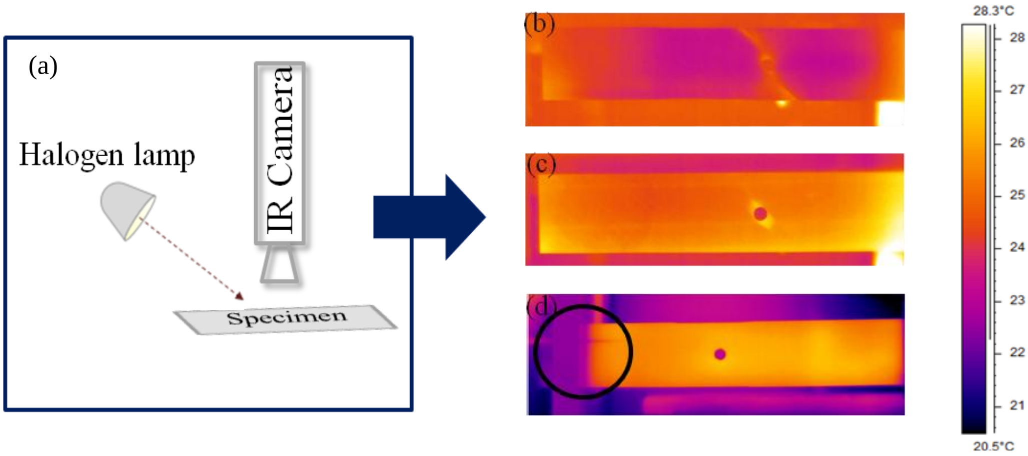

Figure 4. a) IR setup, b) CFRP sample with two vascules, c) four vascules and d) GFRP with four vascules

2.5. Mechanical tests

Fatigue tests were conducted on a Kinston 8802 servo-hydraulic test machine with hydraulic grips (Fig. 5a). A grip pressure of 40 bar was applied to ensure there was no slippage at the grips. All tests were carried out at standard laboratory conditions (21±2°C, 50±5% relative humidity).

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Previous work on quasi-static tension tests (cross head velocity 2 mm/min) determined the nominal quasi-static failure loads in the case of open hole samples, which were 25KN and 15KN for CFRPs and GFRPs respectively. Tension-tension fatigue tests were conducted for both cases at a maximum load of 10KN, applied an R ratio equals to 0.1 and a frequency of 5Hz (Fig. 5a).

Reference samples were interrupted at various stages during fatigue loading in order to detect the damage progression until completely failure. The inspection tool used for the identification of damage evolution was C-Scan. In the case of vascularized specimens the injection of the healing system was applied at the stage where damage growth reached the vascules position. A premixed low viscosity resin system (healing agent) was then manually injected through a syringe within the vascules. The healing system was injected premixed to eliminate the possibility of not contacting Part A and Part B after vascules eruption.Vascules ends were remained open to the atmospheric pressure to promote the healing agent to flow either through the channel or into the damage zone (Fig. 5b). The reason for not including the healing system within the material during manufacturing and before testing is to avoid its polymerization before damage reaches vascules. During injection process the healing system was observed flowing at the surface cracks, confirming its diffusion (Fig. 5c). C-Scan results are presented in Table 2 and Table 3 for each case study. Due to the large anount of the results, representative figures of a single specimen are selected. The trend of the damage development is the same for each subcategory.

(a) (b) (c)

Figure 5: CFRP samples a) fatigue loading, b) manually injection of the healing agent, c) healing agent blots flow through cracks

3. Results and discussion

Typical damage evolution under constant amplitude tension-tension fatigue loading at CFRP and GFRP specimens was observed by Ultrasonic C-Scan technique as shown in Table 2 and Table 3 respectively. Loading conditions were interrupted in order to inspect the damage development for each case at certain loading cycles.

Reference samples with no vascules were tested up to fracture. Vascularized specimens were loaded until damage reached the position of the vascules. After the injection of the healing system, the recommended cure cycle was followed according to the material supplier which was 24 hours at room temperature. C-Scan method was applied again in order to determine, as highlighted, how the healing agent affect the damaged area. After this process was completed, specimens were tested again at the same conditions.

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Table 2. C-Scan figures for CFRP samples

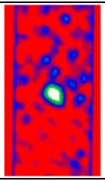
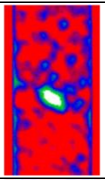
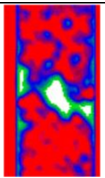
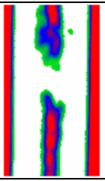
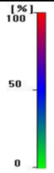
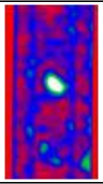
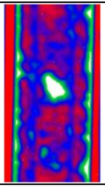
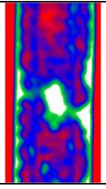
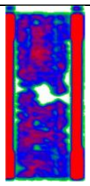
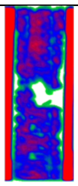
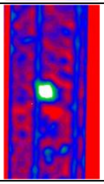
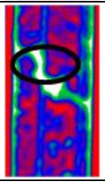
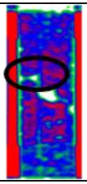
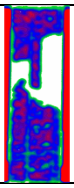
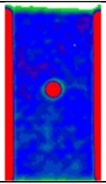
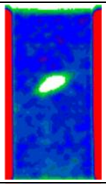
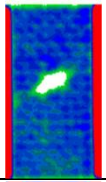
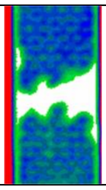
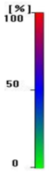
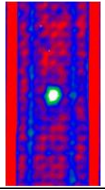
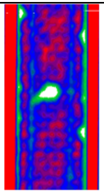
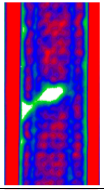
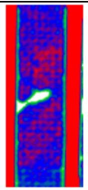
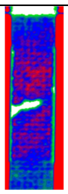
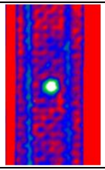
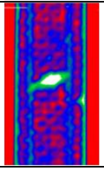
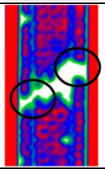
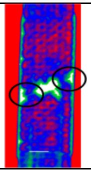
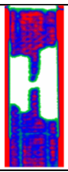
Case Study 1	0 Cycles	25.000 Cycles	40.000 Cycles	125.000 Cycles	After Healing	5.000 Additional Cycles	
Reference					-	-	
2 vascules							
4 vascules			-	-			

Table 3. C-Scan figures for GFRP samples

Case Study 2	0 Cycles	25.000 Cycles	40.000 Cycles	175.000 Cycles	After Healing	5.000 Additional Cycles	
Reference					-	-	
2 vascules				-			
4 vascules				-			

As it was observed, for both CFRP and GFRP materials systems, the damage is developed from the hole to the free edges of the samples, passing through the vascules. Significant ultrasonic signal diffractions for the samples with two and four vascules bult into the material structure. The healing system being injected at the two vascules did not heal the damaged area as shown in Table 2 and Table 3. This is probably attributed not only to the insufficient healing quantity but also to the vascules position and to the healing system diffusion ability through the layers. The presence of the vascules between the 0° layers, where there in no stiffness mismatch between the adjacent layers, and in

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practice no delamination damage is promoted, makes the healing process almost innefective. On the contrary in the case of four vascules system, placed above and below the mid plane, between the adjacent plies of 90° and 45°, delamination has been developed and the injected premixed healing system significantly reduced the area of the damage. The healing agent is deliberately selected with a low viscosity in order to facilitate the injection process and the capillary flow, however due to its polymerization rate the achieved diffusion is considerably confined.

The case scenario including four vascules proved to be more efficient regarding the decrease of the damaged region. It has to be mentioned however that the damage development up to vascules position occurred at lower loading cycles (25.000) than in the case of the material with two embedded vascules (40.000). It is conceivable that the size of the vascules affect the structural integrity of the composite, thus comparatively the damage accumulation is observed at a smaller number of fatigue cycles. Comparing the damage developed in the 2 different vascularized groups (the one with 2 and the second with the 4 vascules) at the same number of fatigue cycles , it is obvious that the material with four vascules appears more extensive damaged area than that with two vascules.

All vascularized specimens were tested again at the same conditions, after the healing process was completed and the inspection was performed in denser stages than the initial.

The development of damage in an area larger than the initial one which was cured by the healing process, constitutes the benchmark for the termination of the test.

3. Conclusions

A preliminary study concerning the damage development in the case of open hole composite specimens with vascules as healing reservoirs, was performed. Different reinforcements (CFRP and GFRP) and three specimen categories (no vascules, two and four vascules) were investigated on how each case affected the damage growth under fatigue loading conditions. Ultrasonic C-Scan technique was employed for the inspection of the fatigue damage evolution. No major differences were observed regarding the damage development for glass and carbon reinforcement.

Two vascules set up within the composite did not sufficiently heal the damaged area . Four vascules configuration successfully managed to reduce the damaged area as shown by C-Scan plots.

Furthermore, comparing two over four vascules within the material, it can be deduced according to C-Scan results that composites containing two vascules developed the same damage zone in more loading cycles compared against the ones containing four vascules. The aim when developing composites with a self healing functionality is the ability for properties recovery but without compromising the overall mechanical performance, which is not the case in the present study.

Optimizing vascular system design, selecting the appropriate low viscosity healing agent and determining exactly the contribution of the healing system in properties recovery are further opportunities for improvement.

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References

[1] C.J. Norris, I.P. Bond and R.S. Trask. The Role Of Embedded Bioinspired Vasculature On Damage Formation In Self-Healing Carbon Fiber Reinforced Composites. *Composites Part A*, 42: 639-648, 2011

[2] C.J. Norris, I.P. Bond and R.S. Trask. Interactions between propagating cracks and Bioinspired self-healing vascules embedded in glass fiber reinforced composites. *Composites Science and Technology*, 71:847-853, 2011

Excerpt from ISBN 978-3-00-053387-7

[3] A.R. Hamilton, N.R. Sottos and S.R. White. Mitigation of fatigue damage in self healing vascular materials. *Polymer*, 53:5575-5581, 2012

[4] O.J. Nixon-Pearson, S.R. Hallet, P.J.Whithers and J. Rouse. Damage development in open hole composite specimens in fatigue. Part 1: Experimental investigation. *Composite Structures*, 106:882-829, 2013

[5] Z. Yongbo, F. Huimin, W. Zhihua, M. Xiaobing. Open hole fatigue characteristic and probabilistic model for fatigue life prediction of CCF300/QY8911 and T300/QY8911 composite laminates. *Journal of Composite Materials*, 0(0):1–10, 2014

Excerpt from ISBN 978-3-00-053387-7