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Tensile and creep behavior of microvascular based self-healing composites: Experimental study

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Abstract

A 3D microvascular network which was generated by 3D printing method was within the composite specimens. Healing efficiency of the composites was investigated using mechanical tensile and creep tests. The experimental results revealed that despite mechanical properties degradation in the specimens due to the presence of the embedded network, significant healing efficiency can be obtained at both room and elevated temperatures by the presented healing method. The maximum healing efficiency of 89% was obtained in tensile strength for the specimens, which were healed for 7 days. Moreover, creep behavior of the self-healing specimens showed promising results and the healing efficiency was 83% in creep rupture time of the composites at 90 °C.

Keywords: Self-healing, Creep, Microvascular network, 3D printing

1. Introduction

In recent years, various types of self-healing materials and structures have been developed to enhance the performance of the structures during their service life. Microencapsulation approach [1–3], hollow glass fibers [4] and [5], thermoplastic additives [6], and microvascular systems [7–9] are various approaches for creating self-healing behavior in the polymers and polymer composites. Moreover, shape memory alloys which were used as a reinforcement of composites [10] and [11], are recently utilized for the manufacturing of self-healing composites [12].

The microvascular self-healing approach is inspired by the biological organisms. In this method, a 3D or 2D network of vessels is utilized to distribute the healing agent within the structure. Various types of damages in any location of the structures can be healed by this healing method. The main advantage of the microvascular systems is the ability to distribute a large amount of healing agent uniformly within the structure. Aragon et al. [13] designed 2D and 3D microvascular networks based on the generic algorithm. They found that the flow efficiency was enhanced by adding diagonal micro-channels in the network. Various manufacturing methods for microvascular polymer composites were reviewed in [7]. The fabrication methods are categorized into the removable and nonremovable cores, micromachining, ink writing, electric discharge, and vaporization of sacrificial components (VaSC). Capolla et al. [8] investigated the tensile and damage behavior of vascular 3D woven glass/epoxy composites experimentally. They used VaSC technique for fabrication of the specimens and found that if there is no misalignment of reinforcing fibers, the reduction in the strength of the composites does not occur. Hartl et al. [14] quantified the negative effects of the microvascular networks on the mechanical properties of the composites. A hierarchical sensing- healing system based on the microvascular healing method was introduced by

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Sakurayama et al. [15]. They used compression after impact tests to evaluate the effectiveness of their system and achieved promising results. Kim et al. [16] fabricated a self-healing membrane using a network of capillary tubes filled with water-reactive healing agent. They reported that using this system lead to recover the water permeation and particle rejection performance of the damaged membrane to its undamaged conditions. Most of the previously published investigations studied the healing efficiency of the composites at room temperature and characterizing the mechanical behavior of the self-healing systems at the elevated temperatures needs more investigations.

The creep behavior of polymer composites is an important parameter, which should be considered in the design process of composite structures. Some research works were performed to investigate the creep behavior of glass fiber reinforced composites [17–22]. Experimental study on the creep behavior of polypropylene/glass composites was carried out by Hugo et al. [17]. They concluded that creep damage and creep rupture of the composites were controlled by the steady state creep mechanism of the matrix. Kouadri-Boudjelthia et al. [18] studied the creep behavior of glass/polyester composites experimentally. They used experimental data to develop a phenomenological model for describing the creep behavior of composites. More recently, Arruda et al. [19] performed a numerical modeling on the creep behavior of sandwich panels with GFRP face sheets and polyethylene terephthalate (PET) foam cores. They verified their model with the experimental results and analytical predictions and reported good agreements between the results.

So far, the 3D printed microvascular network has been less considered for investigations, in comparison with other healing methods. To the best author's knowledge, the creep behavior of microvascular based self-healing composites has not been investigated in the previously published papers In the present article, an experimental study is carried out on the tensile and

creep behavior of glass fibers reinforced epoxy composites, with embedded microvascular healing system. The 3D network of the microvascular system was fabricated using a 3D printer. Healing efficiencies of composite specimens were obtained at room and elevated temperatures. The fracture surfaces of the specimens are also investigated using scanning electron microscopy (SEM).

2. Experiments

2.1. Materials

Unidirectional glass fiber reinforced epoxy composites were utilized for determination of the healing efficiencies. Epoxy resin KER 828 with the Epichlorohydrin hardener (Kumho P&B Chemicals Inc.) was used as the matrix. The resin to hardener ratio was 100:10 according to the manufacturer. Physical and mechanical properties of the resin are presented in Table 1.

E-glass fibers roving (Lintex, China) with the density of 2560 kg/m^3 was used as the reinforcement. The 3D network was fabricated using 3DM cast polymer (3D Materials, Inc.) with the density of 980 kg/m^3 and viscosity of 45 mPa.s .

Table 1. Physical and mechanical properties of KER 828 epoxy resin [23].

Property	Values
Density (kg/m^3)	1160
Viscosity (Pa.s)	12–14
Flash point ($^{\circ}\text{C}$)	> 150
Tensile strength (MPa)	69
Tensile modulus (GPa)	2.75

2.2. Specimen fabrication

Dog-bone tensile specimens were fabricated according to ASTM D638 [24]. To prepare the test specimens the 3D microvascular network was fabricated using a 3D printer instrument. Digital light processing (DLP) method was utilized to fabricate the microvascular network. This method is based on the Vat polymerization technique. In DLP method, the light source is developed digital light projector screen. The fabricated network consists of two distinct sub-networks for containing resin and hardener, separately. Figure 1 demonstrates the schematic of the generated network. Inner diameter and thickness of the tubes were 650 μm and 100 μm , respectively. Dimensions of the fabricated networks were 37 mm $\times$ 12.6 mm $\times$ 6.6 mm. The width and thickness of the tensile specimens were 13 mm and 7 mm, respectively.

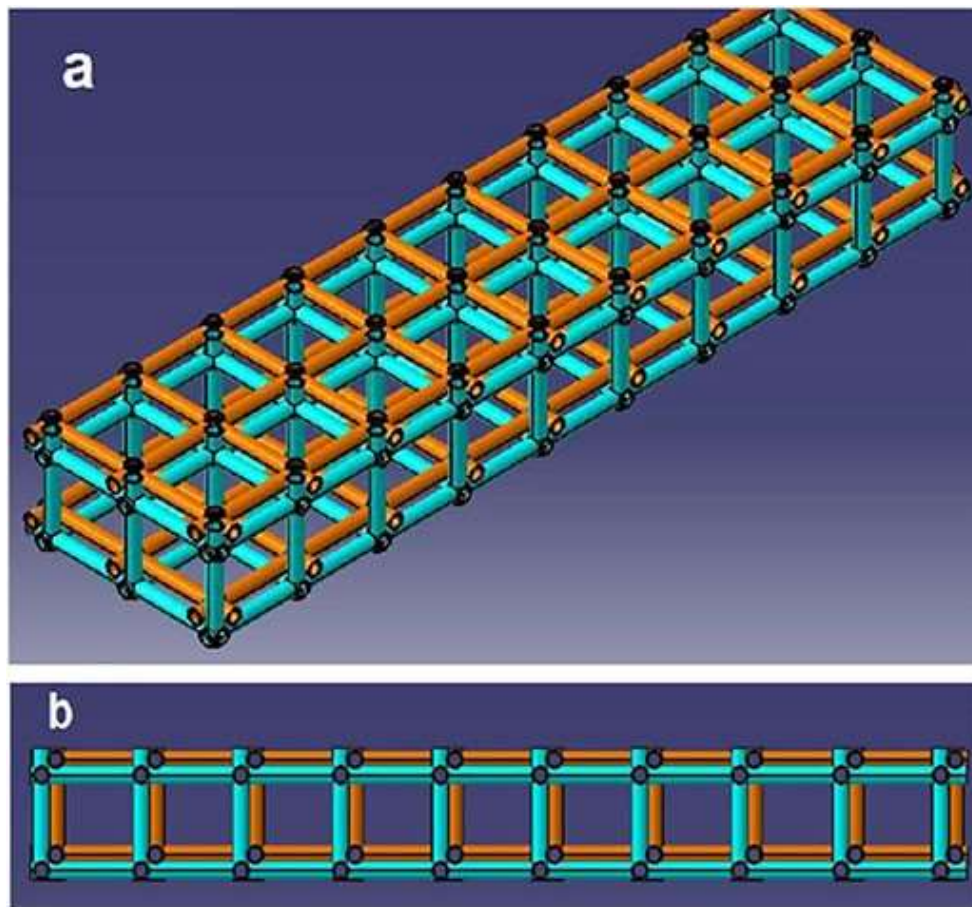


Figure 1. Schematic of the microvascular network, (a) isometric and (b) side view.

Specimen preparation was performed using a previously fabricated silicone mold. To fabricate the specimens, the microvascular network was filled with resin and hardener. Then the glass fiber roving was pre-impregnated with the resin and embedded in the silicon mold in the longitudinal direction of the tensile specimen as can be seen in Figure 2.

Finally, the epoxy resin which is the same epoxy type with the healing agent was poured into the mold. Curing of the specimens was completed after 48 hours at room temperature. Figure 3 shows the prepared specimens for tensile and creep mechanical tests.



Figure 2. Embedding microvascular network and glass roving within the specimens.



Figure 3. Prepared specimens for tensile and creep tests.

2.3. Mechanical tests

The prepared specimens were subjected to the tensile test according to ASTM D638 using Hounsfield universal testing machine (Model H25KS, UK). The crosshead speed was 2 mm/min during the test. Creep tests were also performed according to ASTM D2990 [25]. In this test method, the specimens were subjected to constant stress (12.3 MPa) at elevated temperatures. Prior to mechanical tests, initial damage was applied to some of the specimens for evaluating the healing efficiency. In the present experiments, the initial damage was created by the drop weight impact instrument. The specimens were subjected to 2.9 J impact energy to create internal cracks. Details of the test specimens are presented in Table 2. The specimens are categorized into four types:

- Control specimens: Specimens without embedded 3D network and without initial damage.
- Intact specimens: Specimens with embedded 3D network and without initial impact damage.
- Damaged specimens: Specimens with the empty 3D network, subjected to initial impact damage, and then subjected to mechanical tests instantly, with no healing treatment.

- Damaged and healed specimens: Specimens with the embedded 3D network, subjected to initial impact damage, healed for 3 and 7 days and then subjected to mechanical tests.

Table 2. Test matrix of the present experiments.

Specimen ID	Mechanical test	Embedded 3D network	Initial damage	Healing time	Test temperature (°C)
T-U-0	Tensile	No	No	—	25
T3D-U-0		Yes	No	—	25
T3D-D-0		Yes	Yes	—	25
T3D-D-3		Yes	Yes	3 Days	25
T3D-D-7		Yes	Yes	7 Days	25
C3D-U-0	Creep	Yes	No	—	90
C3D-D-0		Yes	Yes	—	90
C3D-D-7		Yes	Yes	7 Days	90

It is noteworthy to mention that according to the resin data sheet, the curing process of the utilized epoxy resin is completed in 7 days and the mechanical properties of the resin reach their maximum values. In this regards, 7 days was selected as the final time for self-healing process.

The healing efficiencies of the specimens for tensile and creep tests can be calculated according to Eqs. (1) and (2).

$$\eta_{Tensile} = \frac{S_{healed}}{S_{undamaged}} \quad (1)$$

$$\eta_{Creep} = \frac{T_{healed}}{T_{undamaged}} \quad (2)$$

where η is the healing efficiency and S and T represent the tensile strength and creep rupture time, respectively.

3. Results and discussion

3.1. Degradation of tensile strength

To investigate the effect of the 3D microvascular network on the mechanical properties of glass fibers/epoxy composites, tensile tests were performed on the specimens with and without embedded networks. The obtained stress–strain diagrams for the specimens are demonstrated in Figure 4. Failure of the specimens after tensile tests is also shown in Figure 5. According to the results, 38% reduction in the tensile strength of the composites was observed due to the presence of the network. Considering the dimensions of the embedded network and the composite specimens, reveals that the network acts as a stress concentration zone and reduces the tensile strength of the composite. Moreover, generation of the resin rich zones is another destructive parameter that affects the strength of the specimens. The degradation in mechanical properties of specimens was reported in many of the experimental studies in the field of microvascular or hollow fiber healing systems [14, 26–28]. Huang et al. [26] examined the compressive strength of the microvascular self-healing composites and reported 13–70% reduction in the compressive strength of the specimens depending on the microvascular channel dimensions. However, by increasing glass fibers volume fraction in the composite or by using the materials with higher stiffness and strength for 3D network fabrication, the negative effect can be reduced. In this regards, Hartl et al. investigated the effects of channel orientation, spacing and cross-sectional area on the mechanical properties of the composites [14].

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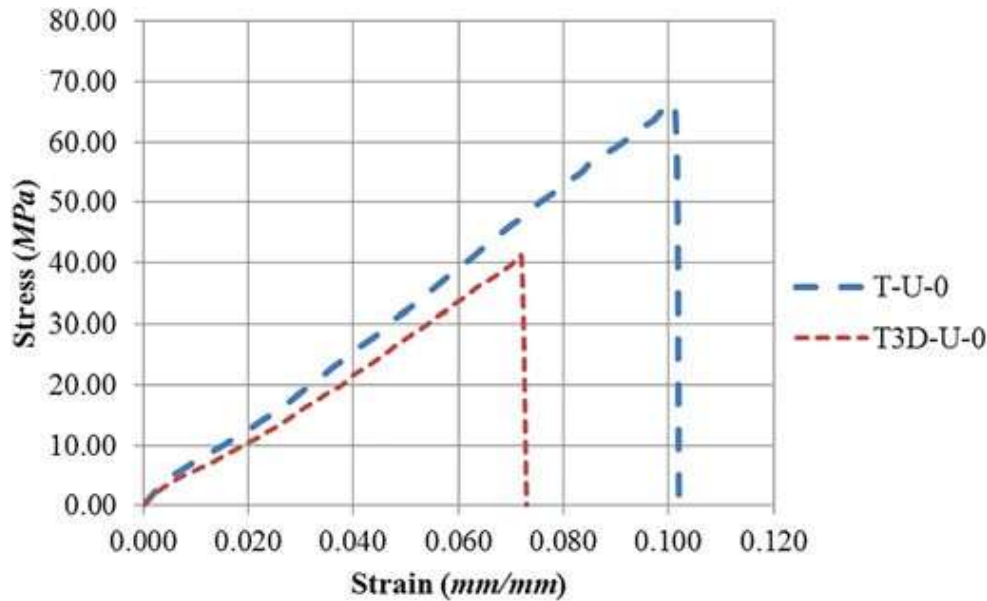


Figure 4. Stress–strain diagram for the specimens with and without embedded 3D microvascular network.



Figure 5. Tensile specimens with and without embedded network after failure.

3.2. Healing efficiency for tensile test

The test specimens were initially subjected to low-velocity impact for creation the internal damage in the composites. Figure 6, shows the impact area and the generated cracks within the specimen which are filled by the healing agent.



Figure 6. Filling the cracks with the released healing agent from the embedded network.

Stress–strain diagram for the damaged specimens and the specimens with 3 and 7 days healing treatments are demonstrated in Figure 7. As can be seen in Figure 7, significant variation in the tensile strength and also strain at failure is obvious for different types of the specimens. Healing efficiency of the composites can be obtained as the ratio between the strength of the healed specimens and that of the control specimen. The strength values and healing efficiencies are summarized in Table 3.

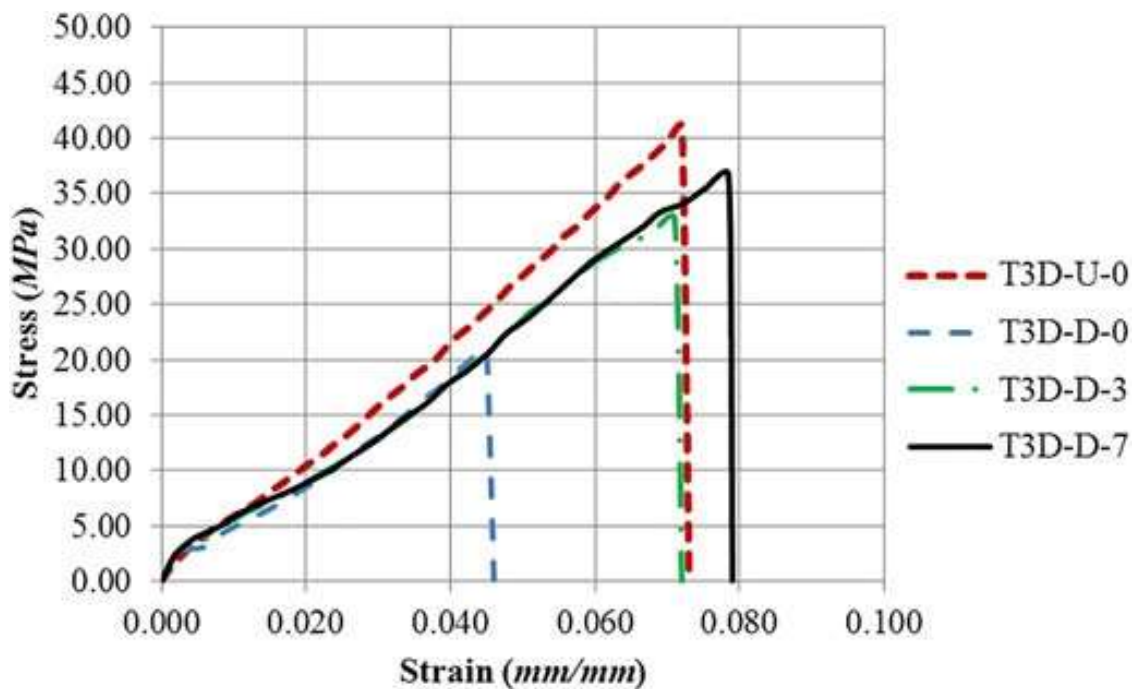


Figure 7. Stress–strain diagrams for the self-healing specimens.

Table 3. Healing efficiency for different types of the specimens.

Specimen	Tensile strength (MPa)	Healing efficiency (%)
T3D-U-0	41	—
T3D-D-0	21	—
T3D-D-3	32.7	80
T3D-D-7	36.7	89

According to the results, the healing efficiency of the microvascular healing system in the present study reaches 80% and 89% for 3 and 7 days healed specimens, respectively. The results showed that the curing of the healing agent was completed after 7 days and the maximum healing efficiency was achieved, but in 3 days healed specimens, the specimens didn't show their maximum strength. The obtained healing efficiency (89%) is higher than other healing methods of the composites, for example healing efficiency for tensile strength in molecular Ion-mediated healing [29] (75%) and microencapsulation [30] (19%) due to a large amount of healing agent. A comparison between the obtained results with the previously published papers on vascular system as was reviewed by Cohades et al. [31] (healing efficiencies were between 40% and 96%) shows that the obtained healing efficiency lies in a high range of efficiencies. Some other healing methods (for example thermally reversible resins) are able to achieve 100% healing efficiency but their low mechanical properties and their need to external force and pressure for healing limited their applications.

For studying the surface properties of the specimens, SEM was used on the fracture surfaces of the composites after mechanical tests. Figure 8, shows the fracture surface of the specimens with and without healing system after tensile failure. According to Figure 8a, the

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glass fibers were entirely pulled out from the epoxy resin during failure and the interfacial failure between the fibers and the matrix is obvious in the specimens without healing system. On the other hand, in the specimens, which were healed for 7 days, a strong bonding between the fibers and the surrounding matrix was created due to the presence of the healing agent (Figure 8b). Moreover, investigating the fracture surface of the healed specimens, revealed that the presence of the cured healing agent, makes the fracture surface of the specimens rough, in comparison with the smooth fracture surface of the pure epoxy resin (Figure 9). The generation of the micro-cracks within the specimens and healing of the generated cracks, lead to a rough surface in the fracture surface of the specimens, while in the pure epoxy, the cracks were not filled by the healing agent and therefore the fracture surface is smooth.

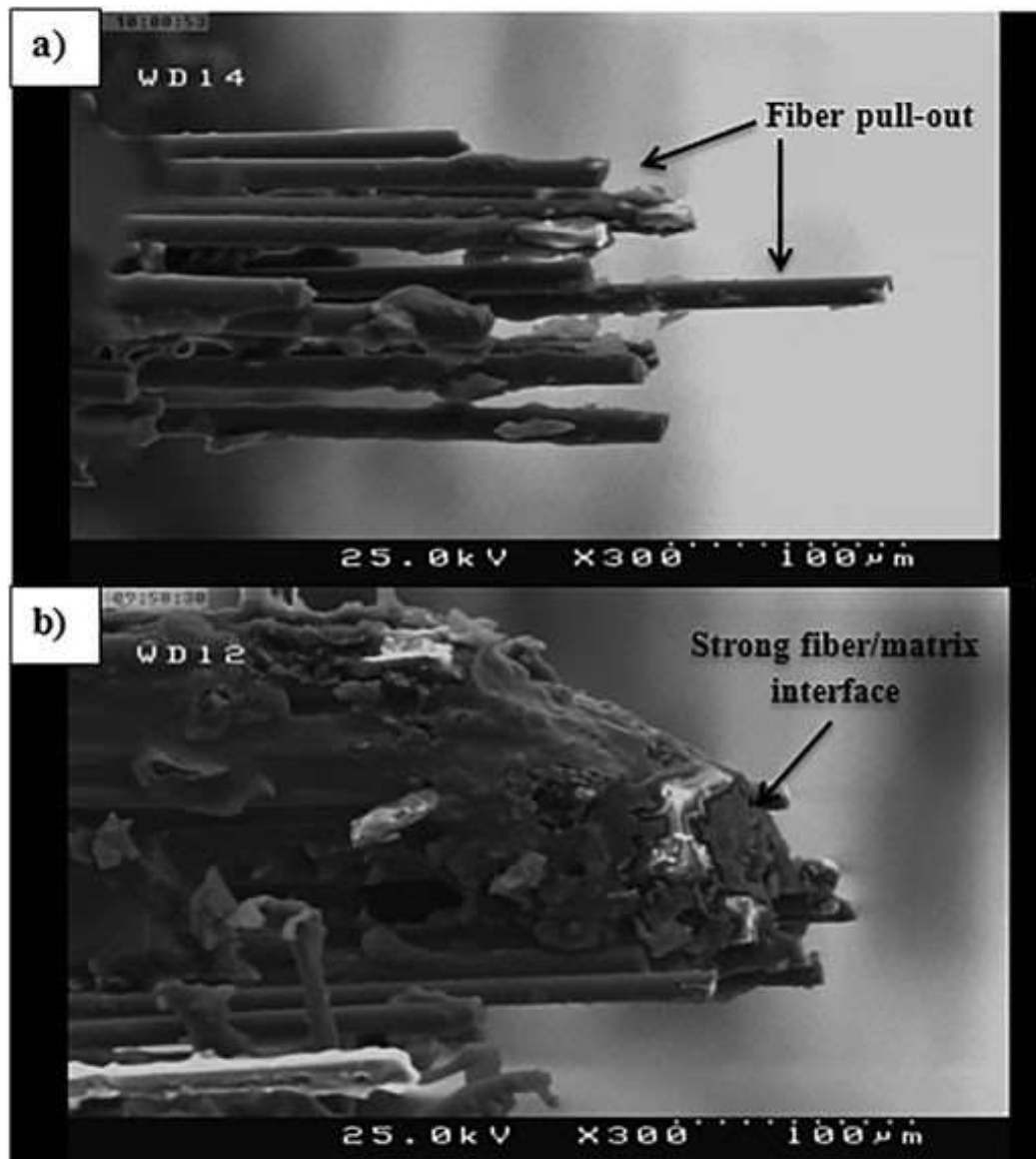


Figure 8. Fracture surface of the specimens, (a) without and (b) with healing system.

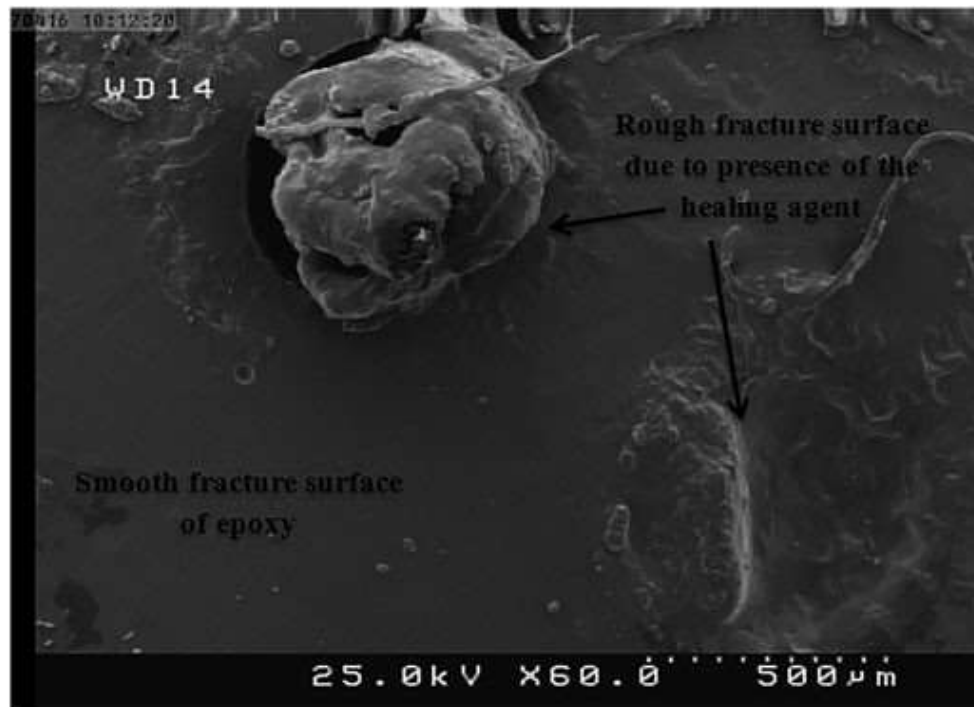


Figure 9. Fracture surface of the epoxy matrix in the healed specimens.

3.3. Healing efficiency for creep test

In addition to tensile tests, the healing efficiency was also investigated for the creep behavior of the specimens. In this regards, the specimens were subjected to initial damage using drop weight test and their creep behavior was examined experimentally. To investigate the creep behavior of the composites, the specimens were subjected to 12.3 MPa constant tensile stress at 90 C. Among the suggested temperatures in ASTM D2990, 90 C was selected for providing suitable rupture time (about 42 min for each test). Moreover, higher temperatures may lead to damage on the microvascular network. Strain-time diagrams for the undamaged, damaged and healed specimens are presented in Figure 10. According to the results, the initial strains of the specimens are different. This means that the presence of the initial damage, changes the modulus of elasticity of the specimens, therefore, the initial strains corresponding to constant applied stress are different. Moreover, damaged specimens showed higher strain

rates in comparison with undamaged ones. The creep rupture of the specimens occurred at different times. Figure 11 demonstrates the failure of the specimens subjected to creep tests. Failure of the specimens first occurred in the polymeric matrix, and after that in the embedded glass fibers. The failure time for the undamaged and damaged specimens was 42 min and 23 min, respectively. A summary of the creep results is presented in Table 4.

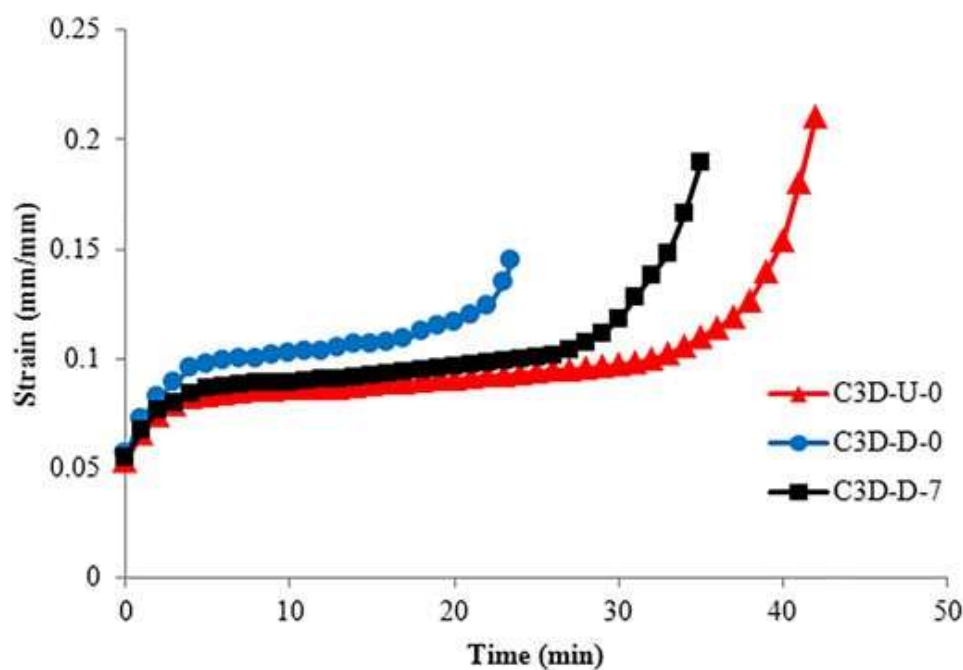


Figure 10. Strain-time diagrams for the control and self-healing specimens according to creep test results.



Figure 11. Self-healing specimen after creep test.

Table 4. The obtained results from creep tests on different types of the specimens.

Specimens	Initial strain (mm/mm)	Strain rate ($\Delta\epsilon/\Delta t$)	Strain at failure (mm/mm)	Failure time (min)
C3D-U-0	0.054	0.0007	0.210	42
C3D-D-0	0.057	0.0009	0.139	23.5
C3D-D7	0.055	0.0008	0.189	35

The healing efficiency of the composites, with respect to the failure time, under constant stress and temperature is calculated to be 83% for 7 days healed specimens. Reinforcing the damaged matrix and improving the interfacial shear strength between the glass fibers and matrix (which was investigated in Section 3.2) are the main reasons for this healing efficiency. According to Table 4, initial damage caused 34% reduction in strain at failure for the damaged specimens. Healing the specimens for 7 days, improves the failure strain of the damaged composites by 35%. The obtained results indicate that the presented healing process is an efficient method for enhancement the mechanical properties of the damaged specimens at elevated temperatures. The healing efficiencies for the tensile and creep behavior of the specimens are presented in Table 5.

Table 5. Healing efficiencies for the specimens at ambient and elevated temperature.

Test type	Temperature (°C)	Test parameter	Healing efficiency (%)
Tensile	25	Tensile strength	89
Creep	90	Failure time	83

4. Conclusion

Healing efficiency of the glass fibers/epoxy self-healing composites with the embedded microvascular network was experimentally investigated. Tensile and creep behavior of the composites were examined and the healing efficiencies were calculated. The results indicate that the microvascular self-healing method shows significant healing efficiency in both tensile and creep behaviors, due to large healing agent capacity. The healing efficiency of 89% was obtained for tensile strength at 25 °C. Moreover, 83% healing efficiency was achieved for the creep rupture time at 90 °C. Investigations on the fracture surface of the specimens using SEM on the fracture surfaces demonstrated the effect of the released healing agent on the improvement of interfacial shear strength between the glass fibers and the epoxy matrix. Besides the promising healing efficiencies, according to the obtained results from the control specimen, there is relatively high degradation in the tensile strength of the composites due to stress concentration of the embedded 3D network.

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