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Effects of vacuum thermal cycling, ultraviolet radiation and atomic oxygen on the mechanical properties of carbon fiber/epoxy shape memory polymer composite

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ABSTRACT

The use of shape memory polymer composites as novel materials for next generation space deployable structures requires the study of the effect of space environment upon their properties. The resistance of carbon fiber/epoxy shape memory polymer composite to vacuum thermal cycling, ultraviolet radiation and atomic oxygen has been evaluated separately by using ground-based simulation facilities. The thermal cycling condition has a temperature range of −100 °C to +100 °C with cycle times of 0, 15, 30, and 45. The irradiation times of ultraviolet with a wavelength range of 250 nm–400 nm are 0, 80, 160, and 240 h. The atomic oxygen radiation has a translational energy of about 5 eV and a flux of about 2×10^{15} atoms/cm^2 at the sample position, and the irradiation times are 0, 33, 66, and 100 h. The shape memory polymer composite specimens are compared in terms of morphology, tensile modulus, breaking strength and elongation at break before and after irradiation. Results show that thermal cycling increases tensile modulus but decreases the breaking strength and the elongation at break of material after 45 cycles. The effect of ultraviolet radiation on mechanical properties of materials depends on the radiation dose received. Atomic oxygen can negatively but slightly affect the mechanical properties of the material.

1. Introduction

Shape memory polymer composite (SMPC) is a kind of smart material that can be programmed by external force and fixed in a temporary shape, then restore to its initial shape under certain type of stimulus (such as temperature [1,2], magnetism [3,4], electricity [5–7], solutions [8,9]). SMPC not only has the characteristics of lightweight, high specific modulus and specific strength as traditional fiber reinforced composites, but also has the characteristics of deformability, high recovery rate and recovery force. It has shown great potential as novel materials for next-generation space deployable structures [10,11]. Several SMPC-based structures have been developed, such as hinges, solar arrays, booms, and so forth [10,11].

The space environment is comprised of thermal cycling, high energy protons, high vacuum, ultraviolet (UV) radiation, atomic oxygen (AO) radiation, and so forth. Materials that are exposed to space environment without protection must be evaluated for their performance under space radiation, which can lead to material degradation, resulting in component or structural damage, system reliability reduction, and even shortening the spacecraft operation time [12–14]. Only these materials that can withstand the harsh space radiations during their planned service life could be selected for aerospace use. Before the space flight evaluation, ground radiation testing of materials is usually performed. Typical ground radiation tests include thermal cycling, UV radiation, AO radiation, and so forth.

Spacecrafts are commonly subjected to alternating temperature changes since they are exposed to continuous changes of the solar radiation [15–17]. Materials used for space application must be able to withstand extremes of high and low temperatures, as well as temperature changes between those extremes, which is so-called thermal

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cycling. Thermal cycling can directly affect the physical, thermal and mechanical properties of materials or components. Therefore, thermal cycling test has become a preset routine for materials targeted for space applications. Sinmazcelik et al. studied the effect of vacuum thermal cycling on unidirectional carbon fibre reinforced polyetherimide (PEI) matrix composites, finding the residual stress induced by thermal cycling lead to a decrease in interlaminar shear strength and flexural modulus at the interface between fiber and matrix [18]. Lvders et al. found that fiber-reinforced plastics under the cyclic thermal loading would undergo alternating stresses in the material at both macroscopic and microscopic scales and that laminate matrix cracking did not reduce the stiffness and strength of the cross-ply laminates [19]. Gassemide et al. employed the Taguchi technique to investigate the effects of fiber volume fraction, composite layup, temperature difference, and thermal cycle number on the mechanical properties of glass/epoxy composites during thermal cycling, and found that the fiber volume fraction and stacking sequence were the major factors [20]. Eslami-Farsani et al. studied the effects of thermal cycling on the hardness and impact resistance of phenolic-matrix composites, finding that the decrease in hardness was affected by the reinforcements and that the basalt fiber had better resistance compared to carbon fiber [21]. Luo et al. developed a shape memory composite based on shape memory epoxy resin, using flexible polyurethane (PU) foam as the foaming material, and carried out thermal cycling tests, which revealed that the shape fixation rate was essentially maintained at 99%. Rapid expansion of the shape memory polymer foam (SMPF) was achieved within 60 s by heat transfer through an electrical heating film [22].

UV refers to the fraction of electromagnetic light having wavelengths between 10 and 400 nm. It has high energy and can cause ionization and photolysis of molecules, hardening (crosslinking) and weakening (chain scission) of polymers or polymeric composites due to the accumulation of the UV dose over time. Space UV is amongst the most active and conspicuous components of Low Earth Orbit (LEO) at altitudes of roughly 300 km–500 km. This would cause the corrosion and degradation of materials used on the exterior of spacecrafts [23,24]. Yan et al. studied the influence of UV irradiation on the fiber reinforced polymer composites and discovered that the flexural and tensile capabilities worsened after 1500 h because the irradiation may induce the chemical composition of the polymer's surface to degrade, as well as a decline in its thermo-optical characteristics [25]. Abdullah et al. figured out the influences of UV irradiation on the mechanical properties of the carbon fiber/polyetheretherketone (CF/PEEK) composite. The degradation properties and temperature behavior of the samples were evaluated [26]. Wu et al. found that the yellowing mechanisms is the result of aging-induced free radical oxidation reactions by measuring the yellowing index of epoxy and vinyl ester resins under thermal, UV and natural aging conditions [27]. Xu et al. studied shape memory polymer (SMP) with the matrix of syntactic foam and conducted a 90-days exposure to UV radiation, finding that UV exposure resulted in yellowing and cracking of the foam surface, increased modulus, and decreased mechanical strength and ductility [28]. Xie et al. showed that the UV radiation reduced the thermal stability of the cyanate-based SMP, but its mechanical properties remained almost unchanged [24]. Azzawi et al. did experimental study on the influence of UV radiation on the shape memory behavior and thermomechanical characteristics of the styrene-based SMP and its fiber reinforced composites, revealing that the UV radiation degraded the mechanical properties of materials [29].

AO, in LEO, is a type of free oxygen atom produced by photolysis of diatomic oxygen molecules exposed to solar UV radiation at wavelengths less than 243 nm [30]. AO is considered to be one of the main reasons for the degradation of surfaces exposed to the LEO environment, since the AO can easily break molecular chain and erode the surface of materials [31,32]. Many researchers have investigated the effects of AO on polymeric composites by exposing the materials directly to the space environment or by conducting ground simulations [31–34]. Andropova et al. found the hybrid nanoparticles in polyimides increased its AO erosion resistance since the filler prevented the penetration of AO into the inside of the material [34]. Jang et al. performed AO exposure experiments on carbon fiber-reinforced SMPCs (CF-SMPCs) and used the linear product of shift factors acquired from accelerated experiments to estimate the long-term mechanical behavior, finding the mechanical properties of materials almost unchanged [35]. Jayalath et al. tested cyanate ester based SMPs with different accumulating AO dosages, indicating that the chemical bonding was changed and the surface was eroded due to the bombardment of the AO. However, there is discernible difference in thermal stability or mechanical qualities [36]. Liu et al. used AO-irradiated polyimide/Al_2O_3 composites to explore the influence of AO radiation on the structural and tribological behavior of the material. It was discovered that AO irradiation caused oxidation and degradation of the polyimide molecular chains, resulting in an increase in O, a decrease in C, and changes in the chemical structure and morphology of the sample surface [37].

This article mainly studies the resistance ability of carbon fiber/epoxy SMPC under the condition of ground-simulated thermal cycling, UV and AO radiations of the LEO environment. Section 2 goes through the specifics of the material preparation and experimental settings. The results and discussion of the morphological and mechanical characteristics of SMPC before and after irradiation are presented in Section 3. Section 4 concludes that the carbon fiber/epoxy SMPC can be used in aerospace as long as their mechanical properties are within the required range during operation.

2. Material and experiments

2.1. Material and specimen preparation

The matrix used in this study is the epoxy-based SMP developed by the Jinsong Leng's group. It has been exposed to the ground simulated thermal cycling, UV and AO radiations, showing good radiation resistance. The glass transition temperature (T_g) of the matrix is 109 °C, determined by the temperature corresponding to the maximum value of the loss factor (Fig. 1). The reinforcement is the plain carbon fabric (CO6343B, TORAY).

The composite is carbon fabric reinforced epoxy-based SMPC with four layers in fiber orientation of 0°/90°. The carbon fiber/epoxy SMPC fabrication method combines hand and compression molding techniques (Fig. 2(a)). The manufacturing process is as follows: Firstly, the plain carbon fabric sheet is fixed by the fixture which is used for positioning the fabric in the middle of the mould. Then, the SMP resin is injected into the mould. The mould is placed in the drying cabinet at 120 °C for 2 h to make a prepreg. Then the mould is taken out of the drying cabinet, and the prepreg is demoulded. After that, four layers of

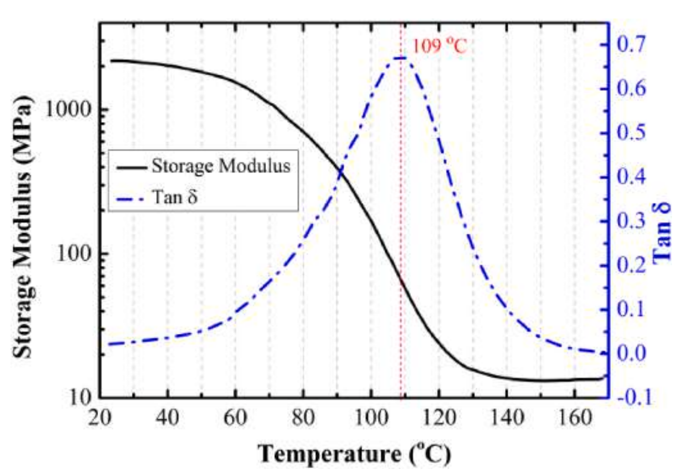

Fig. 1. Results of dynamic mechanical analysis of SMP.

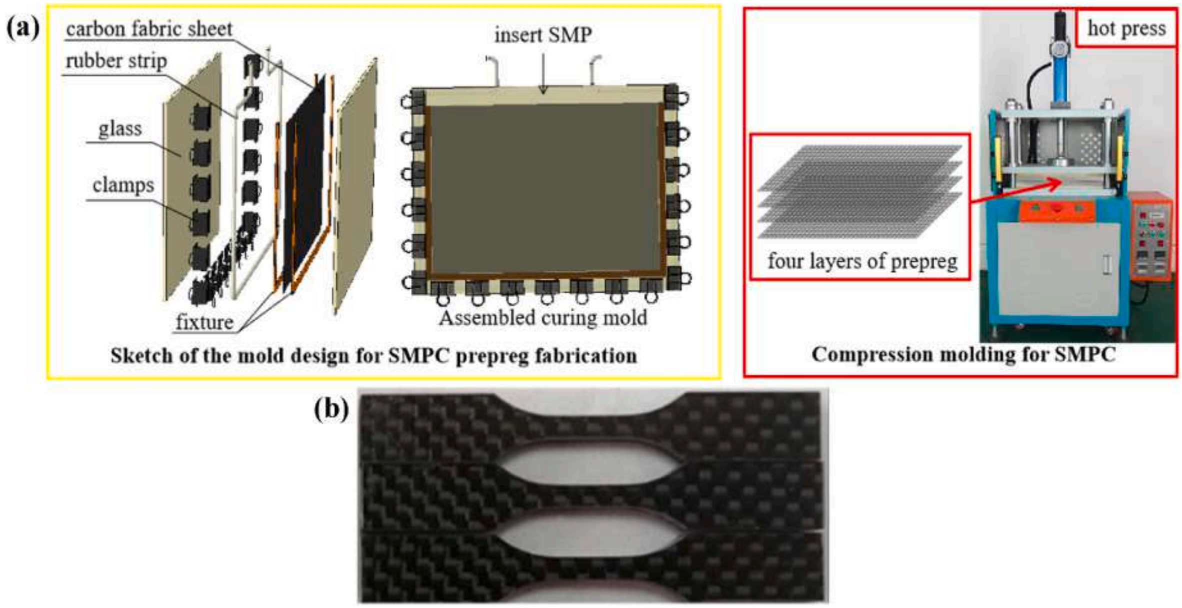

Fig. 2. Material and specimen preparation of the carbon fiber/epoxy SMPC. (a) The carbon fiber/epoxy SMPC fabrication method. (b) The experimental specimens of SMPC.

prepreg are put on a hot press for 5 h at temperatures around 150 °C and a pressure of roughly 0.3 MPa to cure the SMPC laminate. After demoulding, the SMPC is cut into the experimental specimens with a ply angle of 0°/90° according to the ASTM-D638, Type V (Fig. 2(b)).

2.2. Environmental exposure

Yanshan University's accelerated LEO space environment simulation facility, including a thermal vacuum chamber, UV lamp and AO beam, is used to simulate the LEO thermal cycling, UV and AO radiations. The photo of the simulation facility is shown in Fig. 3.

2.2.1. Thermal cycling

A LEO extreme temperature condition ranging from −100 °C to +100 °Cis simulated under a vacuum pressure of 10^{-5} Pa. The heating and cooling rates are 2 °C/min. Four groups of SMPC specimens are prepared to estimate the effect of the number of thermal cycles on mechanical properties of material. The first group is a blank control group without thermal cycling. After 15, 30, and 45 cycles, the second, third, and fourth groups are removed from the facility sequentially.

2.2.2. UV radiation

The UV radiation is carried out under a vacuum of 10^{-5} Pa and at a temperature of −10 °C. The deuterium lamp (Shanghai Ray Monde Co., Ltd., China) can emit light with a wavelength range of 250 nm–400 nm. The intensity of UV is at least five times that of the solar constant. Four groups of SMPC specimens are prepared with UV exposure times of 0, 80, 160, and 240 h.

2.2.3. AO radiation

The simulated condition for the AO radiation is a vacuum of 10^{-5} Pa with oxygen as the medium. At the specimen location, an AO beam is generated by a CO_2 laser with an advection energy of about 5 eV and a flux of roughly 2×10^{15} atoms/cm^2. Four groups of SMPC specimens are exposed for 0, 33, 66, and 100 h, respectively.

2.3. Experiments

To investigate the effects of thermal cycling, UV radiation, and AO radiation on mechanical properties of the carbon fiber/epoxy SMPC, we carry out scanning electron microscopy (SEM) and tensile tests of SMPC

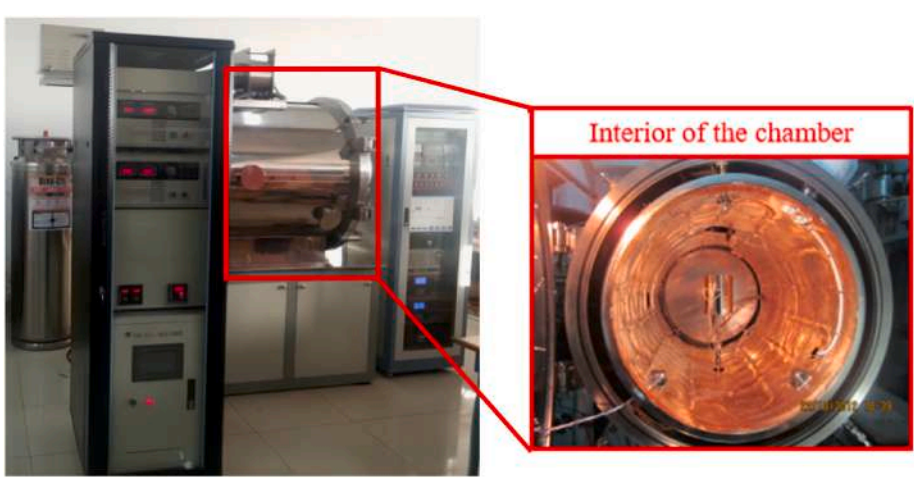

Fig. 3. The accelerated LEO space environment simulation facility.

before and after the irradiation. The SEM is performed to correlate the changes of SMPC in mechanical properties during irradiation with interfacial microstructure. The surface morphology of specimens before and after the irradiation is scanned using a scanning electron microscope with a 5 kV operating voltage (SUPRA35, Germany). Quasi-static tensile tests using a Zwick Z010 universal testing instrument (Zwick GmbH, Ulm, Germany) are carried out to determine changes in mechanical characteristics of SMPC after irradiation. The tensile modulus, breaking strength, and elongation are compared.

3. Results and discussions

3.1. Surface morphology

3.1.1. Effect of thermal cycling on SMPC morphology

The maximum temperature during the thermal cycling is 100 °C, which is lower than the curing temperature of SMPC. Thus, the morphology and surface color of SMPC specimens do not change significantly under different thermal cycles.

Fig. 4 shows the cross-sections of the fracture surfaces of tensile tested SMPC specimens without thermal cycling and after 45 thermal cycles. A, B, and C represent 90° laid-up carbon fibers, SMP matrix, and 0° laid-up carbon fibers, respectively. As can be seen in Fig. 4(a), the interface between fiber and matrix of the unexposed specimen is tightly bonded, and the cross-sectional areas of both the fibers and the matrix are relatively flat. After 45 thermal cycles, some micro voids appear on the surface of the matrix, some fibers are severely peeled off, indicating that the bonding properties of the interface were degraded. As the number of thermal cycles increases further and reaches a certain level, the mismatch of thermal expansion coefficients, which is the expansion of the matrix and the shrinkage of the carbon fibers, can lead to the debonding of the interface between the fiber and the matrix.

3.1.2. Effect of UV radiation on SMPC morphology

As shown in Fig. 5, surfaces of specimens exposed to different UV radiation times barely change, but the gloss of the surface is dimmed. Referring to our previous study, the dimming of SMPC is caused by the yellowing of matrix [23].

The morphology comparison of the SMPC before and after UV radiation is shown in Fig. 6. The surface of the specimen without UV radiation is relatively smooth. After 240 h of UV irradiation, the surface becomes rough since the energy of UV radiation induces the chemical aging of the material. Long-term UV irradiation of SMPC can also cause silver streaks to form on the surface. The appearance of silver streaks in a material does not always mean that it will fracture and degrade. The specimen will not fracture until the external force received exceeds a certain threshold. On the other hand, the production of silver streaks also contributes to a certain extent to the dull color of the material. Moreover, this erosion is limited to the exposed surface since it receives the highest radiation dose. The carbon fiber has good resistance to UV radiation, and it blocks the UV radiation from further damaging the resin encapsulated inside the material, which effectively improves the radiation resistance of the SMPC.

3.1.3. Effect of AO radiation on SMPC morphology

The duration of AO radiation affects the appearance and morphology of SMPC. Fig. 7 shows the color changes of SMPC specimens before and after AO radiation. The dimensions of specimens remain the same after 33, 66, and 100 h of AO exposure. However, the color of the surface gradually dims, and the surface becomes rougher as the exposure duration increases.

Our previous study on the effects of atomic oxygen on the epoxy-based SMP has shown that the SMP specimen exhibited significant erosion after AO irradiation [30]. And similarly, the SMPC surface morphology exhibits significant erosion.

3.2. Mechanical properties

3.2.1. Effect of thermal cycling on the mechanical properties of SMPC

The mechanical properties of SMPC undergo the alternating temperature from −100 °C to +100 °C are shown in Fig. 8. The data points represent the average of at least three specimens with a standard deviation. The tensile modulus rises from 8.1 GPa (0 cycles) to 9.0 GPa, 8.7 GPa, and 9.5 GPa after 15, 30, and 45 thermal cycles. The breaking strength decreases by 0.04% (15 cycles), 4.15% (30 cycles), 4.56% (45 cycles), and the elongation at break decreases by 10.5% (15 cycles), 3.3% (30 cycles), and 11.1% (45 cycles) compared to the value at 0 cycle.

The tensile modulus shows an non-monotonic increasing trend with the increase of the number of thermal cycles The increase in modulus is due to the post curing of SMPC. Since the high temperature is not severe enough to break the chemical bonds but increase the crosslinking degree and shortening the segment lengths between adjacent cross-linking spots [38]. The breaking strength and elongation at break show an overall decreasing trend with the increase of the thermal cycle number. The decrease of breaking strength and elongation might be caused by the cyclic thermal stress generated by the cyclic temperature loading. As the temperature increases, the SMP matrix expands and the carbon fiber shrinks, and vice versa. The mismatch of the coefficient of thermal expansion between the fiber and the matrix leads to a relatively internal stress in the composite. The internal stress weakens the fiber-matrix interfacial bonding and initiates microcracking inside the material, which reduces the breaking strength and the elongation at break of the material. On the other hand, the interfacial debonding and

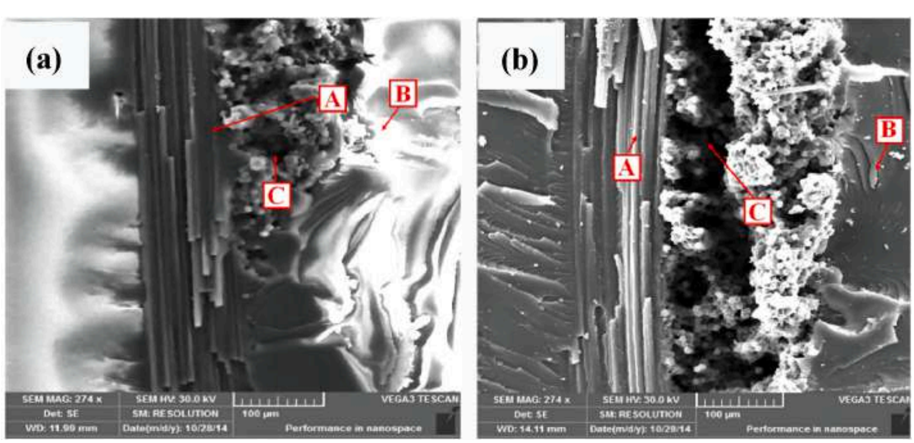

Fig. 4. Morphology of SMPC before and after thermal cycling. (a) 0 thermal cycles. (b) 45 thermal cycles.

Fig. 5. SMPC color change before and after UV radiation. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

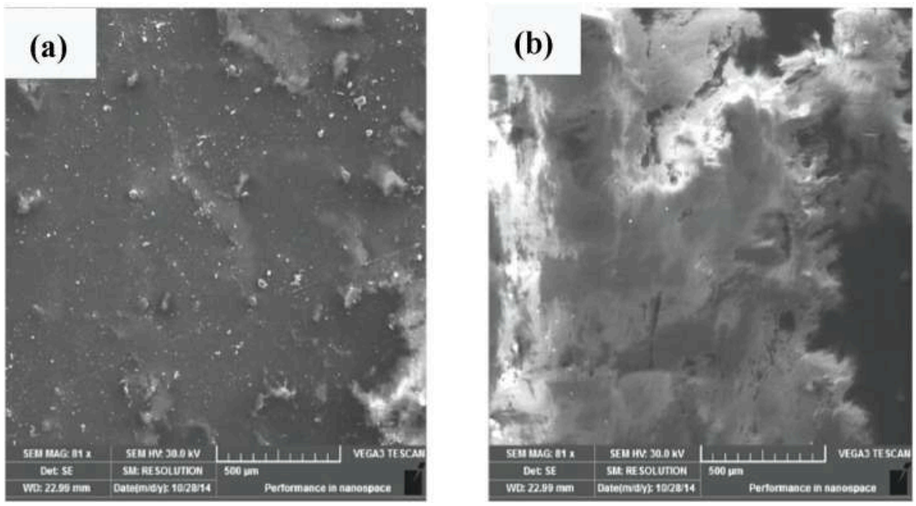

Fig. 6. Morphology of the SMPC before and after UV radiation. (a) 0 h. (b) 240 h.

Fig. 7. Color changes of SMPC before and after AO radiation. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

microcracking would allow the stress to be redistributed, which in turn alleviates the internal stress in the composite. However, the redistribution of stress is random, which leads to an unpredictable relationship between the internal stress and the interfacial debonding and microcracking. This is reflected in the non-monotonic growth in the mechanical parameters against the numbers of thermal cycles. Besides, one of the reasons for SMPC's elongation drop is the matrix's elongation reduction. Our previous study of the effects of thermal cycling on SMP show a decrease in elongation, we deduce that it might be caused by defects generated by outgassing [23].

3.2.2. *Effect of UV radiation on the mechanical properties of SMPC*

Fig. 9 shows the tensile modulus, breaking strength, and elongation at break of SMPC with different UV exposure times. The tensile modulus increases slightly with increasing irradiation time. The increases in tensile modulus are 1.7% (80 h), 2.1% (160 h), and 4.0% (240 h) compared to the specimens without UV exposure. With the gradual accumulation of UV radiation, the breaking strength first decreases and then increases. Breaking strengths are 121.7 MPa, 116.9 MPa, 138.0 MPa, and 142.0 MPa corresponding to UV exposure times of 0, 80, 160 and 240 h, respectively. The elongation at break has the same tendency as the breaking strength. The elongation at break decreases by about 4% after 80 h of UV radiation and increases by 11% and 13% for specimens irradiated for 160 and 240 h.

The tensile modulus increases monotonically with the increase of exposure time, which is mainly due to the post curing of the material. The small rate of change in tensile modulus at the initial stage of radiation, which is 80 h in this study, can be explained by the fact that the elevated temperature mainly accumulates on the surface of the specimen and hardly transfers to the interior of the SMPC. As the exposure duration lengthens, the UV irradiation inevitably raises the interior temperature of the material, and the post curing reaction occurs to a relatively large extent, which leads to an increase in the crosslinking density of SMPC at the later stage, reflected in a larger rate of change in modulus at an exposure time of 240 h. The breaking strength and elongation at break decrease after 80 h of UV irradiation because the matrix is eroded on the surface. The reason for the defects being confined to the surface of SMPC is due to the limited penetration depth of UV radiation. The defects on the irradiated surface weaken the material properties. However, mechanical properties of the SMPC increase at the exposure times of 160 and 240 h. This can be explained by the fact that with the accumulation of UV dose, the effect of increasing

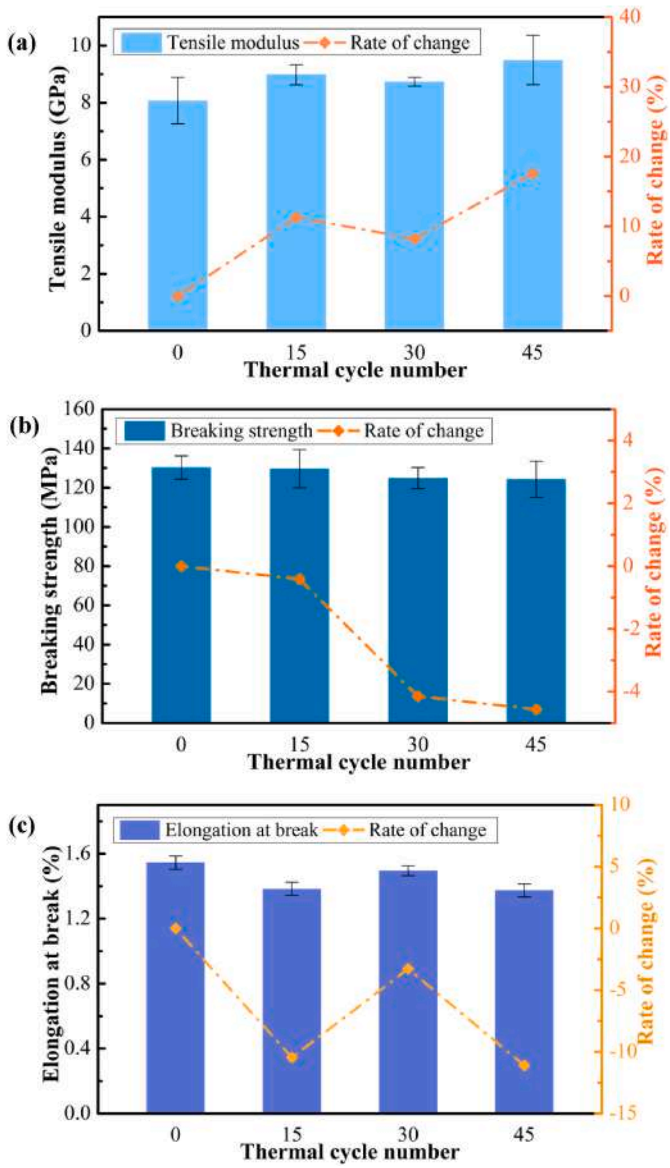

Fig. 8. Mechanical properties of SMPC before and after thermal cycling. (a) Tensile modulus. (a) Breaking strength. (c) Elongation at break.

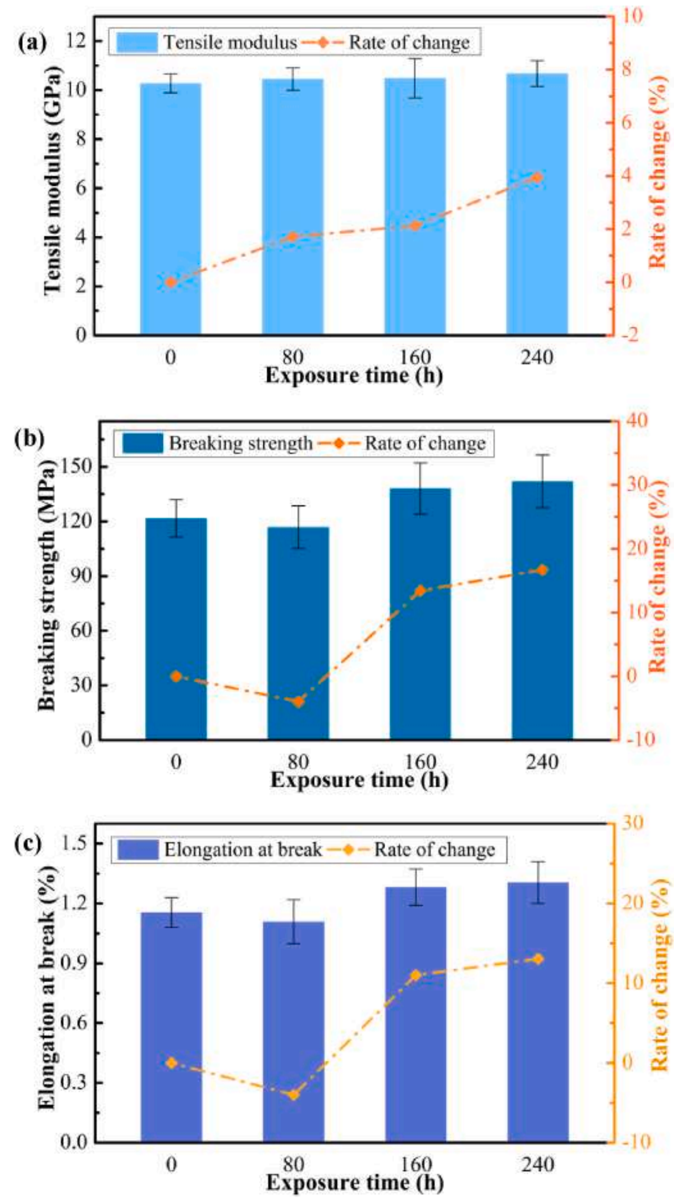

Fig. 9. Mechanical properties of the SMPC before and after UV radiation. (a) Tensile modulus. (b) Breaking strength. (c) Elongation at break.

crosslinking on the enhancement of mechanical properties is higher than the effect of surface erosion on the reduction of mechanical properties. Thus, the breaking strength, and elongation at break increase significantly under 160 h of UV radiation. However, the main load-bearing part of SMPC is the carbon fiber, so when the irradiation reaches a certain level, the post curing and degradation of the matrix have little effect on material properties of SMPC, which is a good explanation of the rare improvement of mechanical properties at 240 h of UV exposure compared to 160 h.

3.2.3. *Effect of AO radiation on the mechanical properties of SMPC*

The tensile modulus, breaking strength and elongation at break of SMPC with AO exposure times of 0, 33, 66, and 100 h are shown in Fig. 10. Compared to the unexposed SMPC, the tensile modulus decreases by 8.0%, 8.1%, and 12.3%; the breaking strength reduces by 4.7%, 8.5%, and 15.8%; and the elongation at break decreases by 1.6%, 4.6% and 17.8% after being exposed by 33, 66, and 100 h.

The AO radiation could affect the mechanical properties of SMPC more profoundly. This is because the chemical bonds of SMPC can be dissociated by the high-energy AO, such as the unreacted hydroxyl groups and ester groups containing C, H, O, and so forth. Oxidation reactions produce volatile oxides, which can be deposited nearby and continue to trigger local oxidation, increasing the proportion of deteriorated parts as exposure duration increases. In comparison with our previous study of the effects of AO radiation on the epoxy-based SMP, we can conclude that the degradation of SMPC is much lower than that of SMP [30]. This is because the exposed carbon fiber on the damaged surface can prevent the internal material from being directly exposed to the radiation. The degradation of SMPC is mainly caused by the strong oxidation of AO that destroys the interface of matrix and fiber, resulting in the generation of microcracks and defects. The microcracks contribute to the large decline in the breaking strength and elongation at break.

Since the erosion of AO radiation is a cumulative process, the radiation dose ought to be considered when evaluating the resistance ability of SMPC. It can be seen that the mechanical properties of SMPC have not significantly decreased until the AO radiation time reaches 100 h. The tensile modulus and breaking strength of SMPC with AO radiation for 33 h and 66 h are above 9.3 GPa and 110 MPa, respectively. In addition,

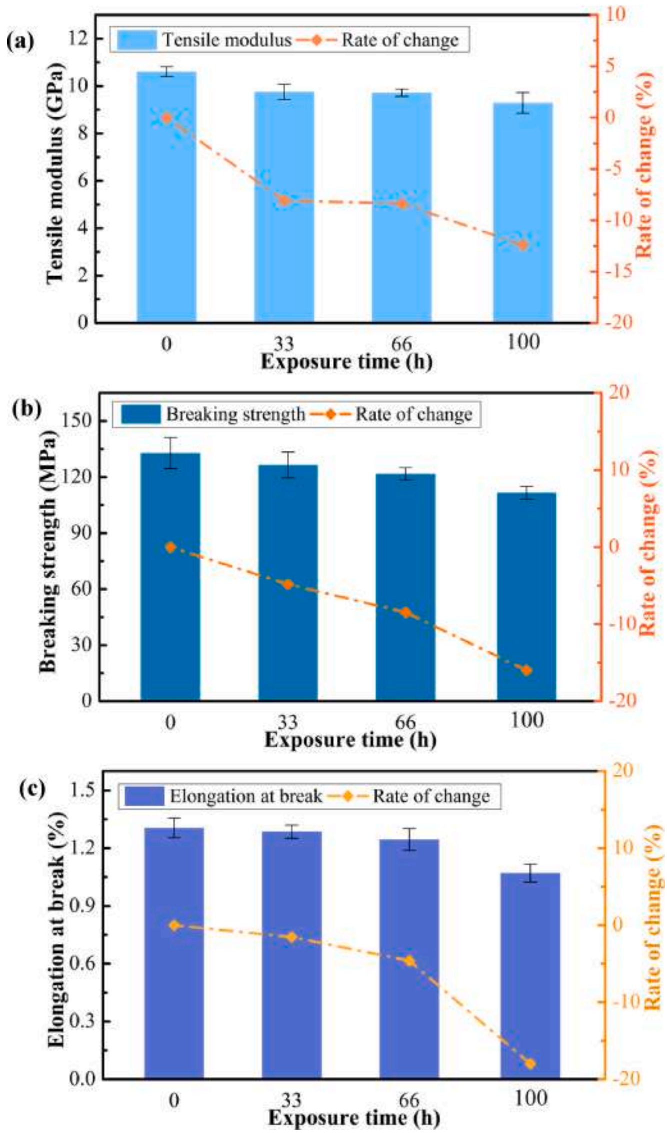

Fig. 10. Mechanical properties of the SMPC before and after AO radiation. (a) Tensile modulus. (b) Breaking strength. (c) Elongation at break.

the elongations at break decreases by less than 10% when the AO exposure time is less than or equal to 66 h, indicating that the SMPC has relatively good resistance to AO attack for exposure times less than 66 h.

4. Conclusion

The mechanical properties of carbon fiber/epoxy SMPC exposed to thermal cycling, UV, and AO radiation are characterized by using an accelerated LEO space environment simulation facility. The morphology, tensile modulus, breaking strength, and elongation at break of SMPC before and after irradiation are compared.

(1) After temperature cycling from −100 °C to +100 °C, the SMPC's surface color and shape are almost unchanged. The tensile modulus increases because of the phenomenon of post curing of the matrix. The breaking strength and the elongation at break decrease by 4.56% and 11.1% after 45 cycles, respectively. These reductions are caused by the fiber-matrix interfacial debonding and microcracking of the SMPC.
(2) The surface of the SMPC is dull and rough after UV radiation. The tensile modulus increases slightly because of the post curing reaction. The breaking strength and the elongation at break first decrease and then increase with the accumulation of UV radiation. The decrease of breaking strength and elongation at break at 80 h of exposure is due to the matrix erosion. But with higher radiation doses, the effect of surface erosion is weaker than the enhancement of the increased crosslinking, so the mechanical properties of SMPC increase significantly at the exposure times of 160 and 240 h.
(3) After being irradiated by AO, the SMPC surface becomes darker and rougher. The tensile modulus, breaking strength and elongation at break monotonically decrease with the increasing of AO exposure time. The reason for the decrease in mechanical properties is that the high-energy impact of AO generates the oxidation reaction of the matrix, resulting in the erosion and degradation of SMPC. After 66 h of AO radiation, reduction rates of mechanical properties are less than 10% compared to the unexposed SMPC, the tensile modulus and breaking strength remain at 9.3 GPa and 112 MPa, respectively.

Inferring from the experimental data, we acknowledge that space radiation can negatively affect the mechanical properties of the SMPC. The SMPC, on the other hand, has greater radiation resistance than the pure SMP, owing to the mechanical enhancement and strong erosion resistance of carbon fiber. Since the effect of space radiation is a cumulative process, the SMPC can be utilized in aerospace as long as its mechanical properties remain within the required range during operation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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