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Study of low earth orbit ultraviolet radiation and vacuum thermal cycling environment effects on epoxy-based shape memory polymer

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Abstract

Shape memory polymers have been studied as the matrix of lightweight self-deployable space structures for decades. The epoxy-based shape memory polymer, which is a potential material for aerospace application, has been tested about their resistance abilities to the ultraviolet radiation and vacuum thermal cycling by using ground-based simulation facilities. The wavelength of ultraviolet radiation is 250–400 nm with not less than five times the solar constant, and the irradiation times are 80, 160, and 240 h, respectively. The vacuum level, temperature range, and cycling times of vacuum thermal cycling are 10^{-5} Pa; −100°C to + 100°C; and 0 cycle and 15, 30, and 45 cycles, respectively. The shape memory polymer specimens are compared for mass loss ratio, chemical compositions, surface morphology, and mechanical properties before and after the ultraviolet radiation and thermal cycling. The experimental results indicate that the glass transition temperature (T_g), mass loss ratio, modulus, and yield and break strength increase after experiencing 240 h ultraviolet radiation or 45 thermal cycles, which shows great potential of epoxy-based shape memory polymer in aerospace structures and device applications.

Keywords

shape memory polymer, ultraviolet radiation, thermal cycling, material properties

1. Introduction

Shape memory polymers (SMPs) represent a relatively new class of shape memory materials, which also include shape memory alloys (SMAs) and shape memory ceramics. SMPs are a stimuli-responsive smart material with the ability to recover an applied deformation upon the application of an external stimulus (heat, electricity, magnetism, light, solution, moisture, etc.) (Behl and Lendlein, 2007; Leng et al., 2011). They are potential alternatives for space deployable structures because of their variable strength and stiffness, lightweight, large recovery and superior process abilities, and so forth (Behl and Lendlein, 2007; Lan et al., 2009; Leng et al., 2011; Li et al., 2016; Lin et al., 2006). The main drawbacks of SMPs are low modulus, low recovery force, and so on. Thus, the shape memory polymer composites (SMPCs) have been developed by adding particles or fibers to reinforce the material properties (Baluch et al., 2016; Behl and Lendlein, 2007, Hassanzadeh-Aghdam and Mahmoodi, 2018, 2019a, 2019b, Leng et al., 2011; Li et al., 2019; Liu et al., 2017; Mahmoodi et al., 2019).

Most spacecrafts (space shuttles, spaceships, satellite and space stations, etc.) travel in low earth orbit (LEO). SMP-based structures, which might be one of the components of spacecraft, will be influenced by various environmental factors, such as thermal cycle, space debris, atomic oxygen (AO), ultraviolet (UV) radiation, and plasma environment (ions and electrons), which

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might impose the normal operation and the lifetime on spacecrafts (Hossain and Ensinger, 2015; Kern et al., 1993; Paillous and Pailler, 1994; Peng et al., 2015; Tennyson, 1991). Among these space environmental factors, UV is one of the most active and predominant components of LEO environment, which will cause the materials of spacecraft to erode and degrade. For evaluating the degradation of materials applied in spacecraft, accelerated experiments using ground-based simulated facilities are often employed (Elleder and Borovansky, 2001; Liau and Tseng, 1998; Peng et al., 2015; Yan et al., 2015). Elleder and Borovansky (2001) claimed that UV could induce yellow auto fluorescence on the surface of specimens, and the common mechanism may be a complete bleaching and oxidative breakdown of melanin and melanin-like polymers resulting from an oxidative attack. The effect of different UV wavelengths and exposure time on polymers and their composites is different. Through comparing the mass loss, morphology, and strength before and after the UV radiation, Liau and Tseng (1998) investigated the influences of the thermal shock and the long-term UV radiation of various thicknesses of both the glass/epoxy and the graphite/epoxy composites. Yan et al. (2015) investigated the effect of UV radiation on fiber-reinforced polymer composites (exposure time: 500, 1000, and 1500 h) and found the tensile and flexural properties deteriorated after 1500 h since the UV radiation might cause degradations of the chemical composition and reduction of thermo-optical property of the polymer's surface (Gotoh and Kikuchi, 2005; Han and Kim, 2006; Kaczmarek et al., 2002; Li et al., 2000; Suchocka-Galas and Kowalonek, 2006). UV wave is useful approach for curing epoxy resin (Bouanga et al., 2011; Yu et al., 2009). Chantarapanich et al. found the elastic modulus increased 8.3%, 15.3%, 15.6%, and 17.1%, respectively, after postcuring for 2, 4, 6, and 8 h. Literature reveals the postcuring plays an important role in the improvement of mechanical properties, and the mechanical strength improved significantly corresponding to the increase of UV radiation postcuring time which ranged from 0 to 4 h. The mechanical properties of specimens after 4 h of UV postcuring were relatively constant (Chantarapanich et al., 2013). Whitten and Youssef (2015) reported the effective elastic modulus of polyurea rose from 0.55 to 0.58 GPa after around 3 weeks of UV radiation, whereas saw a decreasing trend to 0.49 GPa after 15 weeks of UV radiation.

Spacecrafts move in and out of the earth's shadow alternately, leading to a large temperature change of the exposed parts of spacecrafts. For some long-term durability spacecrafts, the thermal cycling is one of the important factors which need to be carefully considered since the temperature change may generate detrimental effects on materials' chemical, optical, and mechanical properties and so on (Gao et al., 2005; Ghasemi and Moradi, 2017; Grossman and Gouzman, 2003; Hancox, 1998; Lafarie-Frenot and Rouquie, 2004; Pagano et al., 1998; Rouquie et al., 2005; Russell-Stevens et al., 2005; Shin et al., 2000, 2001, Yu et al., 2011). Ghasemi and Moradi (2017) investigated the effects of the different conditions of thermal cycling process on the polymer matrix composite degradation, providing predictions of mass loss as a function of environmental exposures and material properties. Gao et al. (2005) investigated the mass loss ratio, modulus, strengths, and expansion/contraction distortions in longitudinal and transverse directions of the unidirectional M40J/AG-80 composites after different thermal cycles. Shin et al. (2000, 2001) found the stiffness and strength of epoxy composite exponential proportionally decreases due to the increase of thermal cycles, and the changes of stiffness and strength in transverse direction had a quicker loss compared to other properties. Yu et al. (2011) studied the physical and mechanical properties of the carbon/bismaleimide (BMI) composites before and after thermal cycling; although the thermal stability and cross-linking degree of the matrix increased after cycling, the general flexural strength was still impacted by the competing between the fiber–matrix debonding and the cross-linking degree.

Any material developed for aerospace field should go through the routine standard tests to confirm whether it is suitable for aerospace environment before the actual application. Because of the experimental time limitation in the laboratory, the accelerated experiment is always used to verify the material. The objective of this article is to study the impacts of the UV radiation and the thermal cycling on the epoxy-based SMP through ground-based simulated systems, respectively. Section 2 presents the material preparation and experimental setups. Section 3 details the results and discussions of the mass, morphological, and mechanical properties before and after the radiations. Section 4 presents the conclusion, indicating the epoxy-based SMP has a great potential in aerospace applications.

2. Material and experiments

2.1. Material preparation

The epoxy-based SMP in this study is synthesized by Jinsong Leng et al. (2009). The specimens for UV radiation and thermal cycling are fabricated and tested, respectively. Specimens' dimension for the dynamic mechanical analysis (DMA) tests is 19 mm × 3 mm × 2 mm, while for the tensile test, the mass loss ratio test and micro-morphology test follow the standard ASTM-D638, Type IV. There are 12 specimens for each type of cutting, divided into 4 groups of 3 specimens each. One group is the control specimens, which has not been exposed to any radiations, and the other three experimental groups are exposed to different doses of radiations. These specimens are labeled and stored in

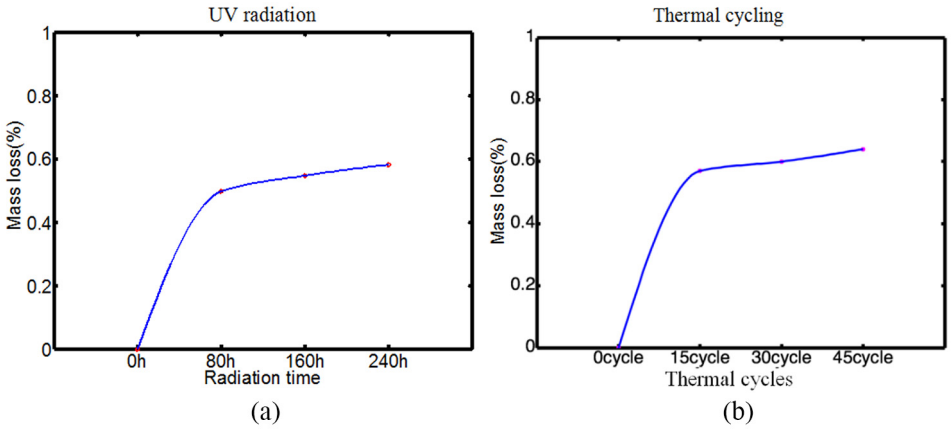

Figure 1. Mass loss ratio of epoxy-based SMP before and after UV radiation/thermal cycling: (a) UV radiation and (b) thermal cycling.

vacuum bags at room temperature before and after UV radiation/thermal cycling.

2.2. Environmental exposure

2.2.1. UV radiation. The specimens prepared for UV radiation are tested in a ground-based UV radiation simulated facility in Yanshan University. UV light is generated by using the low-pressure deuterium lamp produced by Shanghai Ray Monde Co., Ltd., China. The exposure is conducted at the vacuum level of 10^{-5} Pa. The wavelength is 250–400 nm with not less than five times the solar constant. The three experimental groups are exposed for 80, 160, and 240 h, respectively.

2.2.2. Thermal cycling. The specimens for thermal cycling are tested in the thermal vacuum chamber of a ground-based LEO space environment simulation facility in Yanshan University. The vacuum pressure is kept at 10^{-5} Pa throughout the whole test. The simulated LEO temperature condition is −100°C to + 100°C with the heating rate and cooling rate of 2°C/min. The duration for one cycle is ˜135 min. Experimental groups are taken out after 15, 30, and 45 cycles, respectively.

2.3. Experiments

The mass change of every specimen before and after UV radiation/thermal cycling is measured by a BP211D balance (Sartorius, Germany) with a precision of 0.01 mg. The Fourier transform infrared (FTIR) spectrum experiment is carried out on reflection mode from 4000 to 400 cm^{-1} by an FTIR spectrometer (Nicolet, Germany). The surface morphology is observed by a scanning electron microscope (SUPRA35, Germany) with an operating voltage of 5 kV. The dynamic properties are conducted on a DMA 242C analyzer (NETZSCH Instruments, Germany); the test mode is tension with a temperature range from + 10°C to + 170°C and a heating rate of 3°C/min. For estimating the influences of UV radiation/thermal cycling on the static mechanical properties of the epoxy-based SMP, tensile experiment is conducted on a Zwick Z010 universal testing instrument (Zwick GmbH, Ulm, Germany) which is equipped with a thermal chamber from the same company.

3. Results and discussions

3.1. Effect of UV radiation/thermal cycling on SMP mass loss

The mass loss ratio of the epoxy-based SMP specimens before and after UV radiation/thermal cycling is shown in Figure 1, respectively. The calculating equation is as following

$$\Delta m = \frac{m_p - m_a}{m_p} \times 100\% \quad (1)$$

Here, m_p represents the specimen mass before radiation, m_a denotes the specimen mass after the experiment, and Δm is the whole mass loss.

Figure 1(a) illustrates the mass loss ratio increases with the exposure time of UV radiation. After the initially 80 h of UV radiation, the mass loss ratio of SMP is above 0.4%. But with the time further increasing, the growing of mass loss ratio slows down. It can be observed from Figure 1(a) that the mass lose ratio in the first 80 h is higher than that after 80 h. Because the wavelength of UV light used in this test is in the range of 250–400 nm, the energy of the light is 1040–600 kJ/mol. Generally, the bond energy of a covalent bond is 160–600 kJ/mol, which means that the UV light at this wavelength range can destroy any chemical bond. Because there is epoxy chloropropane in SMP matrix, and the bonding energy of the C–Cl bond is relatively low and it is relatively reactive, the chemical bond can be easily ruptured by UV radiation to chlorine or hydrogen chloride. Besides, the unreacted hydroxyl

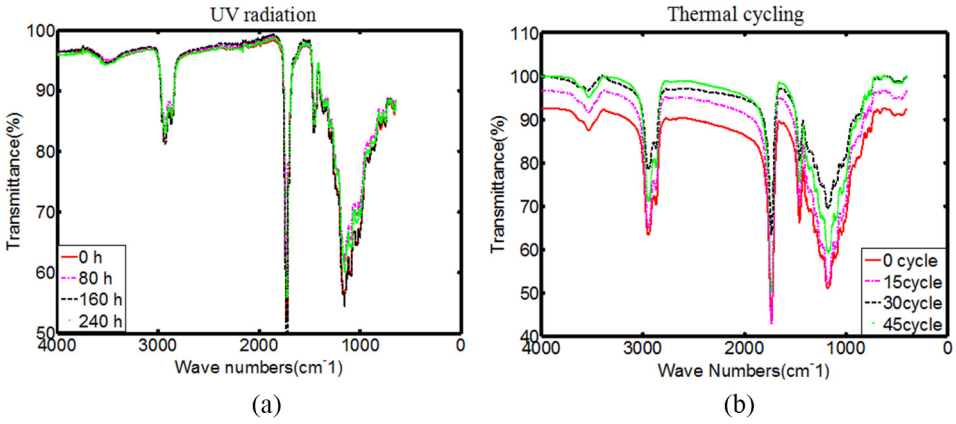

Figure 2. FTIR spectra of epoxy-based SMP before and after UV radiation/thermal cycling: (a) UV radiation and (b) thermal cycling.

Figure 3. The color of epoxy-based SMP before and after UV radiation.

functional groups in the backbone might be volatized by absorbing UV light with wavelength of 200 nm. These chemical bonds are relatively reactive and can be easily prolapsed, causing higher mass lose ratio in the first 80 h. When the specimen has been irradiated by UV light more than 80 h, the removable group in the specimen surface can be exhausted. The C–O bond and C = O bond in SMP can be degraded in the process of UV irradiation. Besides, the irradiation of UV light can make a part of group into the excited state, and the energy can be released as heat. During the process, small molecule gases such as CO, CO_2, and H_2 can be released. The amount of degradable chemical bond and small molecule gases is small; thus, in the later 160 h, the mass loss ratio is relatively small. Therefore, the mass loss of SMP comes from two parts: one is the prolapse of the lateral group and functional group of SMP by higher energy UV radiation and the other is the release of small molecule gases such as CO, CO_2, and H_2 from the degradation of the chemical bond

Figure 1(b) illustrates the mass loss ratio of the SMP under different thermal cycles. The mass loss ratio for 15 cycles is significant, ˜0.57%. It then maintains at a stable value and rises slowing, rising by 0.07% at 45 cycles compared to 15 cycles. The mass loss of SMP can be contributed to the following reasons: the remained organic solvent in the curing process and the small molecular segment inside the material are volatilized during the thermal cycling and the gas adsorbed on the surface or dissolved in the material will be detached and released; in addition, during the production and storage of epoxy-shaped memory polymers, moisture is inevitably absorbed, and this part of the moisture will volatilize with the circulation of the hot vacuum.

The mass loss is significant at lower radiation doses and almost steady at higher radiation doses. Because the release of the organic solvent, gas, moisture, and so on is significant at the beginning, it is almost complete under low radiation dose conditions; the mass loss under high radiation dose condition contributes to the degradable chemical bond, but the amount of these are small, so the mass loss is slight. The total mass loss is an important feature to measure whether a material is suitable for use in a space environment. Generally, aerospace materials require the total mass loss in vacuum environment should not be exceeding 1.00%. The epoxy-based SMP subjected to the radiation dose in this study can withstand this requirement.

3.2. FTIR spectra

The FTIR spectrum results of the epoxy-based SMP which experienced UV radiation/thermal cycles are illustrated in Figure 2. Based on the previous investigation of Leng et al. (2009), the major chemical bonds of the epoxy-based SMP material are carbonyl, carbon–hydrogen, methylene, and carbon–oxygen bond. The characteristic absorption bands of the above ingredients can be all observed in Figure 2. The main component' location and the shape in the FTIR spectrum characteristic absorption bands of the material before and after UV radiation/thermal cycling have rarely

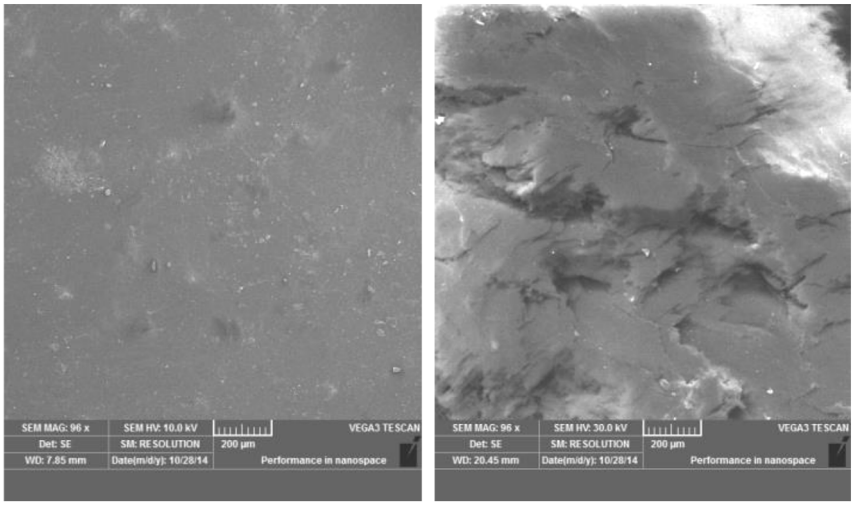

Figure 4. Micro-morphology of epoxy-based SMP before and after UV radiation.

changed. It can be speculated that the main chemical bonds and functional groups have not changed after experiencing a period of 240 h and vacuum level of 10^{-5} Pa of UV radiation or vacuum thermal cycle for 45 cycles.

3.3. Effect of UV radiation on SMP morphology

Figure 3 shows the macro visual images of epoxy-based SMP specimens at different UV radiation doses: 0, 80, 160, and 240 h. Before the radiation, the surfaces of SMP are transparent. After continuous UV radiation, SMP specimens undergo discoloration. The yellowness on the exposed surface of the SMP specimen increases with the duration of exposure. It is believed that the yellowness on exposed surfaces of the SMP specimens attributes to the following reasons: first, the epoxy functional groups may be oxidized and the carbonyl has been formed and second, the amine-type accelerator in epoxy SMP can be yellowed by the UV light. While there are other researchers explaining that the polymer itself is impure and the additives possess chromophoric groups, after UV radiation, the polymer will undergo chemical modification by absorption of photons (Xu et al., 2011).

Figure 4 presents the surface morphology of the epoxy-based SMP specimens before and after UV radiation. The surface is smooth and uniform before UV radiation, while crazing is observed on the side of specimen that was facing the UV radiation after the UV radiation. This illustrates that most of the UV radiation energy has been absorbed by the surface of the material. And this will result in the material aging and increase the surface roughness. But the crazing is limited to only few micrometers in depth; the surface is just slightly accidented in Figure 4, indicating that the UV radiation does not erode SMP severely and further mechanical properties should be tested.

The surface color and morphology of the SMP specimens under different thermal cycles have not shown significantly change since the highest temperature during thermal cycling is 100°C, which may not lead to yellowing of epoxy-based SMP. Therefore, the surface color and the micro-morphology are not posted in this article.

3.4. Dynamic mechanical analysis

The effects of UV radiation/thermal cycling on the dynamic properties of the epoxy-based SMP, including storage modulus, and the damping parameter (tanδ) are analyzed by conducting the DMA. The T_g, which can be used to characterize the cross-linking degree of the epoxy-based SMP, is defined by the temperature corresponding to the damping parameter peak. Figure 5 illustrates the curves of storage modulus and the damping parameter as a function of temperature for these SMPs exposed to different doses of UV radiation and thermal cycles, respectively.

Changes in T_g of the SMP subjected to different doses of UV radiation are shown in Figure 5(a). As the UV radiation time increased to 80 h, the T_g increases from 104°C to 107°C, with an increase of about 2.9%. Afterwards, the T_g tends to be a constant, and there is rare increment as the time of UV irradiation increases from 80 to 240 h. This can be contributed to the UV energy accumulation which increases the surface temperature of the SMP specimen. At high temperature, the cross-linking density increased by opening ring reaction of epoxy chloropropane and esterification cross-linking reaction of the hydroxyl group on the main chain and the acid anhydride in curing agent. It

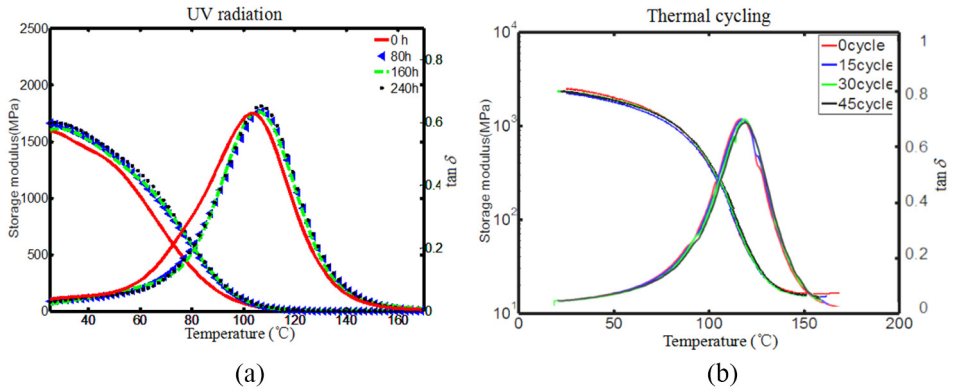

Figure 5. Elastic modulus and tanδ of epoxy-based SMP before and after UV radiation/thermal cycling: (a) UV radiation and (b) thermal cycling.

can be observed that the storage modulus increases slightly as compared to the specimen which has not been exposed to UV radiation. Also, because of long-term UV radiation, the temperature on the surface of material increases, which may cause postcure reaction and acid hydroxyl cross-link esterification, and the cross-linking density of resin increases.

The T_g of SMP specimens in the control group of UV and thermal cycling is 104°C (UV) and 116°C (thermal cycling), respectively, since they are not the same batch. As it can be observed from Figure 5(b), after thermal cycles, the T_g increases from 116°C to 119°C after 45 cycles. Therefore, it can be asserted that the thermal cycle can cause a postcure reaction and increase the level of cross-linking of the epoxy-based SMP to some extent. The DMA experimental results also suggest that dynamic mechanical properties almost remain the same after vacuum thermal cycling. As illustrated in Figure 5(b), the SMP storage moduli change little as thermal cycle increased from 0 to 15, 30, and then 45 cycles.

The increases of T_g after UV radiation/thermal cycling can be contributed to the temperature increase of specimens. The increasing temperature leads to the postcure reaction of the material. Further, the postcure effect leads to the increase of epoxy SMP cross-linking density. For thermoset epoxy-based SMP, the higher the cross-linking is, the shorter the length between cross-linking points is. Based on the free volume theory, the free volume of the chain segment decreases as the cross-linking increases. Due to the reduced free volume, higher temperatures are required to cause movement of the segments. At the macroscopic level, the T_g increases.

3.5. Static mechanical properties

The tensile tests of SMP before and after UV radiation/thermal cycling are performed. The elastic modulus, yield strength, break strength, and elongation of SMP as a function of UV radiation time and number of thermal cycles are compared in Figures 6 and 7, respectively.

As shown in Figure 6, the modulus and yield and break strengths increase with time of UV exposure. The growths of elastic modulus are significant (41% after 80 h of UV radiation, 36% after 160 h, and 35% after 240 h). The yield strengths increase by 46%, 43%, and 40%, and the break strengths rise by 66%, 50%, and 51% after exposing for 80, 160, and 240 h compared to the control specimens. All these mechanical properties increase at first and then slow down. The elongation decreases all the way, dropping by 65%, 53%, and 55% after 80, 160, and 240 h of exposure, respectively.

The results of thermal cycling show the same increase tendency of yield strength and break strength as that of UV radiation, while the increase percentages are much lower. From Figure 7, it can be seen that the yield strength rises from 49.7 to 51, 54, and 53.5 MPa as thermal cycles increase to 15, 30, and 45 cycles, respectively. The break strength increases by 2.7%, 9.6%, and 14.4% corresponding to different thermal cycles. The elastic modulus almost remains the same with 2.6% decrease after 15 cycles, 4.4% increase after 30 cycles, and 7.0% increase after 45 cycles. In addition, the figures of elongation see a drop of 5.9%, 2.6%, and 26.6%, respectively.

Throughout the UV radiation/thermal cycling experiment, the specimens experience high temperature, which leads to postcure of polymer. All these postcure reactions increase the cross-linking density of SMP and shorten the segment lengths between those adjacent cross-linking points and eventually improve the mechanical properties. Because these reactions increase the amount of chemical bond, the more the cross-linking point amount in the main chain, the more rigid the molecular chain, thus the higher strength and modulus of material. Besides, postcure reduces internal stress caused by curing reaction, enhancing the cracking resistance capacity which raises the yield strength and break strength of SMP. But the elastic modulus, yield

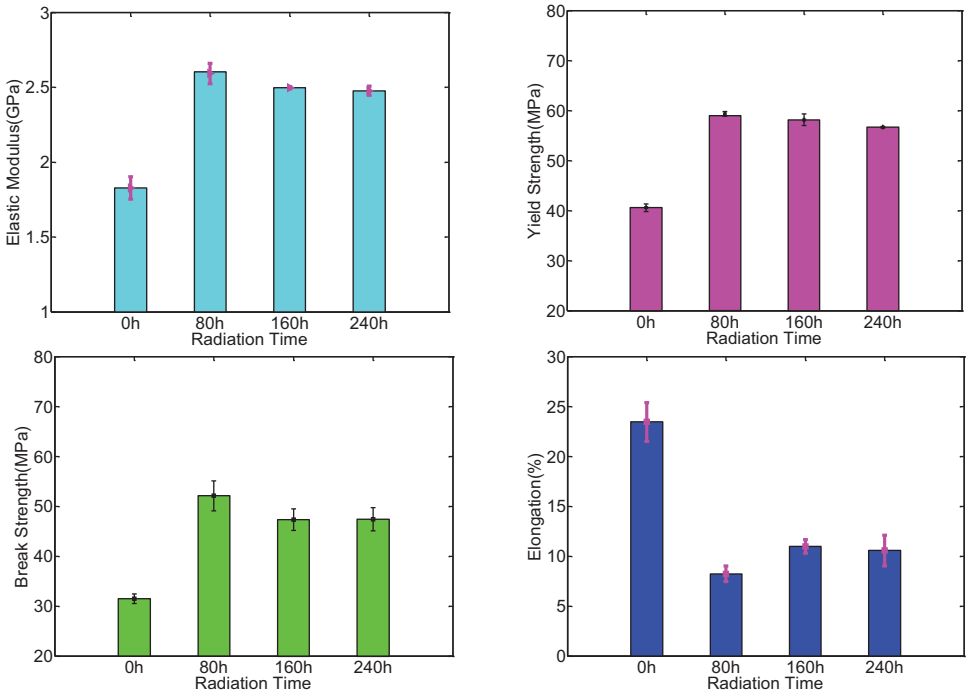

Figure 6. Mechanical properties of epoxy-based SMP before and after UV radiation.

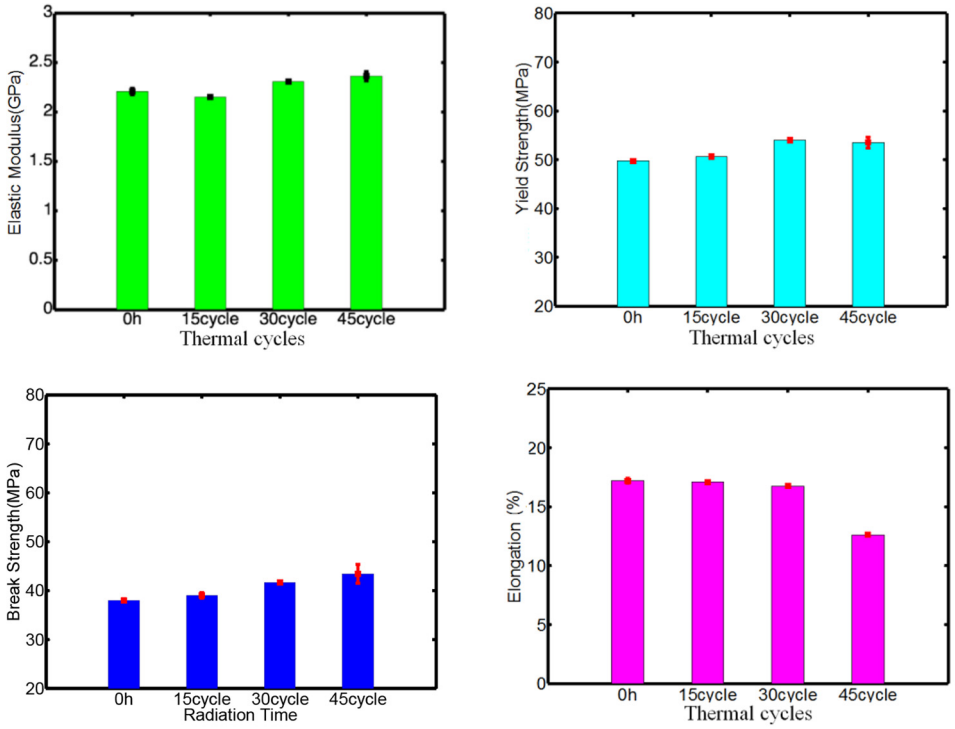

Figure 7. Mechanical properties of epoxy-based SMP before and after thermal cycling.

strength, and break strength decline slightly at 160 and 240 h compared to the values at 80 h. This may be caused by the following reason: with the UV radiation time increasing, the postcure reaction has been accomplished gradually; but with excessive radiation, the random chain scission and depolymerization emerges, resulting in the mechanical property decrease. Figures 6 and 7 show that the elongations decrease with the radiation dose increase. Based on free volume theory, the increasing of the cross-linking density will shorten

the distance between cross-linking points, decreasing the free volume of chain segment, resulting in the shrink of space which allowed the molecular chain relative movement. Besides, when the material is at glass state, the free volume is at frozen state, and this further decreases the ability of the molecular chain relative movement. Moreover, the mass loss results above indicate SMP specimens can outgas chemicals under UV radiation/thermal cycle. This will accelerate the emergence of flaws. And these flaws are sensitive to tensile load. As a result, SMP specimens are more vulnerable to external load and will be failure or fracture at lower load, which results in the decrease of elongation.

4. Conclusion

In this study, the mass loss ratio, FTIR spectra, morphology, and mechanical properties of the epoxy-based SMP material before and after UV radiation/thermal cycling are investigated by experiments on ground. The mass loss ratios are significant at the lowest radiation dose (80 h for UV, 15 cycles for thermal cycling), then trend steady at the other radiation doses. The maximum total mass loss ratios are all below 1%. The shape of the characteristic absorption bands and main chemical components' location before and after UV radiation/thermal cycling are almost the same at the FTIR spectrum experiment. The surface color and morphology of the SMP specimens under different thermal cycles show no significant change. But after the UV radiation, the surface color of SMP is slightly yellowed and crazing is observed on the side facing the UV radiation. This can be explained by the following reasons: the detachment of the lateral group and functional group by high-energy UV light radiation and the release of small molecule gases by UV aging. The T_g, modulus, and yield and break strengths of epoxy-based SMP all increase after radiation since the long-term UV radiation/thermal cycling leads to the postcure reaction, increasing the cross-linking density of the SMP and eventually improving the mechanical properties. While the elongations decrease with the radiation dose increase, because the increasing of the cross-linking density will shorten the distance between cross-linking points, the space which allowed the molecular chain relative movement is shrinked. The results indicate the epoxy-based SMP has relatively good resistances to UV/thermal cycling radiation dose mentioned in this study. However, the impact of any radiation is a cumulative process, and further experiments should be conducted if the SMP is subjected to more radiation dose.

Declaration of conflicting interests

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