Effects of short carbon fibres and nanoparticles on mechanical, thermal and shape memory properties of SMP hybrid nanocomposites

Jianming Guo^{a, b}, Zhenqing Wang^{a, *}, Liyong Tong^{b, *} and Wenyan Liang^a

^a College of Aerospace and Civil Engineering, Harbin Engineering University, Harbin, China

^b School of Aerospace, Mechanical and Mechatronic Engineering, The University of Sydney,
NSW 2006 Australia

* Corresponding author: Tel: +8613704519999 E-mail: wangzhenqing@hrbeu.edu.cn

* Corresponding author: Tel: +61 2 93516949 E-mail: liyong.tong@sydney.edu.au

Abstract:

In this study, carbon fiber/alumina/shape memory polymer (SMP) hybrid nanocomposites were developed with added short carbon fiber (7 wt%) and alumina nanoparticles (0.0–2.0 wt%) for improving the weak mechanical properties of TPI bulk. The shape memory properties and the effect of the weight fraction of alumina nanoparticle and chopped carbon fiber on the mechanical and thermal properties of the developed SMP hybrid nanocomposites were investigated by static tensile tests, mechanical cyclic tests and thermo-mechanical cyclic tests. The test results demonstrate that the SMP hybrid nanocomposites with chopped carbon fiber (7 wt%) and alumina nanoparticles (1 wt%) exhibit a more superior mechanical and thermal properties compared with other samples, and a good shape memory property was obtained in the developed nanocomposites.

Keywords: shape memory polymer; hybrid nanocomposites; thermo-mechanical property; shape memory effect

1. Introduction

Shape memory polymers (SMPs) are stimuli responsive materials, and they possess the capability of restoring their original shape from temporary one upon application of an external stimulus, such as, temperature, electric, light, chemical or magnetic fields [1-3]. During the past two decades, SMPs have attracted significant attention from academia and

industries [4-7] due to advantages, such as low density, low cost, high recovery ability, and superior processability [8, 9], when compared to shape memory alloys (SMAs) [10] and shape memory ceramics (SMCs) [11]. Some exciting applications, such as, self-folding machines [12], self-assembly robots [13], crawling robots [14] and shape memory hybrids [15], have been shown successfully in recent studies.

As one important branch of thermal sensitive SMP, Trans-1, 4-polyisoprene (TPI) and its copolymers have been used in biomedical engineering [16], e.g, backfill gutta-percha in dental clinic [17, 18]. Unlike other SMPs, TPI has several distinct features, such as, easily-tailored transition temperature (T_t) from 317 to 325K around room temperature [19], large deformation (with rupture strain $\geq 200\%$) recovery capacity [20, 21]. This feature of T_t and high rupture strain forms noticeable from the T_t of 328K, 358K and 485K and the rupture strain of 27%, 90% and 3% for shape memory epoxy [22], epoxy shape memory foams [23] and shape memory cyanate polymers [24], respectively. However, similar to other SMPs, the obvious shortcoming low mechanical strength of TPI also limits its applications in broader fields [25].

In order to improve the mechanical properties of SMPs, one simple method is to add high modulus inorganic or organic fillers [26]. The reinforcement fillers of SMPs may be classified into three types: continuous fibers [27, 28], short chopped fibers [29-31], and nanoparticles [32, 33]. It was shown that the maximum fracture stresses of SMP composites with added chopped fibers with 10, 20 and 30 % by weight can be improved by 9, 23 and 44% compared to SMP bulk at 298 K, while maintaining an acceptable shape memory effect (SME) [34]. In our previous study [35], the highest fracture stresses of the TPI composites reinforced by 7 wt% chopped carbon fibers are 30%, 19% and 16% higher than SMP bulk at 301 K (T_t-20 K), 321 K (T_t) and 341 K (T_t+20 K), respectively. However, the mechanical properties of TPI composites should be improved further for broader applications. As we

know, the mechanical properties of SMP nanocomposites showed significant improvement with the content of nanoparticles [32, 33], and the alumina nanoparticles own large surface area which is an important factor for notable improvement in mechanical properties of nano-alumina-filler composites. In this context, the SMP hybrid nanocomposites were developed with added short carbon fibers and alumina nanoparticles by using a new method in the present study.

The new method is based on a new feasible technology. Compared with traditional technology, the new technology makes chopped carbon fibers and nanoparticles add into TPI matrix more easily. Samples of the developed SMP hybrid nanocomposites were fabricated with TPI matrix, chopped carbon fibers (7 wt%) and alumina nanoparticles (0–2 wt%). The shape memory properties of the developed SMP hybrid nanocomposites were characterized according to the method of the fold-deploy shape memory test, and the effect of various weight fractions of alumina nanoparticles on thermo-mechanical properties were investigated by using static tensile tests, mechanical cyclic tests and thermo-mechanical cyclic tests.

2. Experimental

2.1 Materials

TPI matrix with $ML[3+4]100^{\circ}C=80$ was purchased from Wuhan Yuancheng Gongchuang Technology Co, Ltd. Chopped carbon fibers (T700SC-12k-50C) with an average length of 2mm was produced by Toray Industries, Inc. γ -alumina nanoparticles (Table 1) with an average diameter of 50nm were obtained from Shenzhen Jingcai Chemical co, Ltd. TPI was used as the matrix in the developed SMP hybrid nanocomposites, and the T_g of pure TPI is 321K. The associated materials and composition were as follows: 100 parts by weight of TPI, 5 parts by weight of zinc oxide, 15 parts by weight of light calcium carbonate, 0.5 parts by weight of stearic acid, 4 parts by weight of phthalate dioctyl (DOP), 1.5 parts by

weight of peptizer (SJ-103), 0.5 parts by weight of accelerator CZ, 1 parts by weight of antioxidant 4010NA, and 0.3 parts by weight of sulphur.

The chopped carbon fiber and alumina nanoparticles were treated as the fillers. Table 2 shows the proportion of the carbon fiber and alumina nanoparticle in the developed SMP hybrid nanocomposites. The pure SMP is referred to as Bulk, the sample with nominal carbon fiber weight fraction of 7% is referred to as Sample A, and the samples with short carbon fiber of 7wt% and alumina nanoparticles of 0.5wt%, 1.0wt%, 1.5wt%, and 2.0 wt% are referred to as B1, B2, B3 and B4, respectively.

Table 1 Properties of γ -alumina nanoparticles.

Parameters	Average diameter	Specific surface area	Al ₂ O ₃ content	Density	Appearance
γ -alumina	50 nm	130-150m ² /g	99.99%	0.1-0.2 g/cm ³	white powder

Table 2 Ingredient of the developed SMP hybrid nanocomposites

Sample name	Bulk	A	B1	B2	B3	B4
SMP weight fraction	100wt%	93.0wt%	92.5wt%	92.0wt%	91.5wt%	91.0wt%
carbon fiber weight fraction	0.0wt%	7.0wt%	7.0wt%	7.0wt%	7.0wt%	7.0wt%
alumina nanoparticle weight fraction	0.0wt%	0.0wt%	0.5wt%	1.0wt%	1.5wt%	2.0wt%

2.2 Sample preparation

In preparing samples, alumina nanoparticles and pure TPI were subjected to a pre-drying treatment by a vacuum oven (DZF-6210, Shanghai Yiheng Scientific Instrument Co, Ltd) at room temperature for 24 hours. Short chopped carbon fibers were processed by 60% HNO₃ aqueous solution for 90 minutes, distilled water until to neutral and a vacuum oven for 2 hours at 333 K. The weighed alumina nanoparticles were placed in a glass beaker pre-filled with acetone solution, the mass ratio of acetone to alumina nanoparticles is about 200:1. Fig 1 shows the process of dispersion of alumina nanoparticles and TPI. The alumina nanoparticles were dispersed by a magnetic stirrer (DF—II, Jintan Shunhua Instrument Co, Ltd) with 600 RPM and a ultrasonic dispersion analyser (JY98—III DN, Ningbo Xinzhi

Biological Technology Co, Ltd) with 2KHz for 2 hours. The pre-processed pure TPI was added into the acetone solution mixed with dispersed alumina nanoparticles slowly, and kept on dispersing by magnetic stirrer and ultrasonic dispersion analyser for 1 hour. Then the acetone solution mixed with alumina nanoparticles and TPI were dried by a vacuum oven at room temperature for 24 hours.

Fig.1. The dispersion process of alumina nanoparticles and SMP.

Manual agitation and mechanical stirring used an electrical hand blender (QJ-200 Tianjin Honour Instrument Co, Ltd) are used to mix the processed TPI, chopped carbon fibers, alumina nanoparticles and additives in a glass beaker: manual agitation was carried out firstly for 5 minutes and mechanical stirring was gone on for 5 minutes, the cycle was performed for three times. A planetary ball mill (QM-3SP4, NJU Instrument Plant) was used to mix the ingredients sequentially and knead the ingredients together with 580 RPM for 2 hours at 310 ± 5 K. Afterwards, the kneaded ingredients were vulcanized using an electric hot compacting press (XTM-103, Shenzhen Xintaiming Machinery Co, Ltd) with a pressure of 15 MPa and a temperature of 423K for 30 minutes. Then it was cooled down to room temperature. At last, a CNC laser cutting machine (HSLC-1410, Boye Equipment Co, Ltd)

was used to prepare the dumbbell samples (3-mm thickness, 25-mm length and 16-mm width) according to the ASTM D412, 'Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers-Tension'.

2.3 Mechanical and thermal property tests

In order to investigate the mechanical and thermal properties of the developed SMP hybrid nanocomposites, static tensile tests, mechanical cyclic tests and thermo-mechanical cyclic tests were designed. An Instron Universal Testing Instrument (Type 4501) and a temperature-controlled chamber were used to carry out the tests.

2.3.1 Static tensile test

The objective of the static tensile test is to investigate the most suitable mass fraction of alumina nanoparticles for the developed SMP hybrid nanocomposites. The fracture stress, fracture strain and the Young's modulus were used to describe the effects of various mass fractions of alumina nanoparticles under various temperatures. The experimental conditions of static tensile tests were constant tensile speed and various temperatures. The tensile speed was 20mm/min and the temperatures of the chamber were set at 301 K (T_r-20 K), 321 K (T_r) and 341 K (T_r+20 K), respectively. The dumbbell samples were stretched by a universal testing instrument until they fail completely and the ratio of the elongation measured by the crosshead displacement to the gage length was recorded as the axial strain.

2.3.2 Mechanical cyclic test

In order to investigate the influences of cyclic number and alumina nanoparticles mass fraction on the cyclic behaviours of the developed SMP hybrid nanocomposites, The mechanical cyclic tests were performed and the engineering stress and strain were used to represent the mechanical cyclic tensile properties of the SMP hybrid nanocomposites. The mechanical cyclic tests were conducted at room temperature. The constant strain was set to

100% by limiting the crosshead displacement. The number of cycles was set to 5 and the loading rate was 20 mm/min.

2.3.3 Thermo-mechanical cyclic test

Thermo-mechanical cyclic tests were carried out with the aim of exploring the effects of alumina nanoparticles mass fraction on the mechanical properties of the developed SMP hybrid nanocomposites under various temperatures. In a similar method to the mechanical cyclic tests, thermo-mechanical properties of the developed nanocomposites were described by engineering stress and strain. The crosshead speed of universal testing instrument was set to 20mm/min in constant strain cyclic test at 301 K ($T_i - 20$ K), 321 K (T_i) and 341 K ($T_i + 20$ K), respectively. The upper limit strain was 100% and the number of cycles was 5.

2.4 Shape memory properties test

In the present study, a new method called ‘fold-deploy shape memory test’ [36] was adopted to test the shape memory properties of the developed SMP nanocomposites.

Fig 2 shows the schematic diagram of fold-deploy shape memory test. Firstly, the developed samples were heated to 356 K which was higher than T_i , and they were bent into a ‘U’ shape with diameter of 4 mm by external forces. The bending angle was recorded as $\theta_{\max}$ (Fig 2.A). Secondly, the samples are cooled down to 298 K which was lower than T_i with the application of external force. External force was removed after 5 minutes and a marginal recovery occurred, at the same time, the bending angle was recorded as θ_{fixed} (Fig 2.B). Lastly, the samples were heated to T_i (T_i is from 301 K to 353 K each 5 K except each 2.5 K from 311 K to 326 K), the bending angle (θ_i) (Fig 2.C-F) and recovery time (t) were recorded respectively.

$$\text{Shape retention ratio: } R_f = \frac{\theta_{\text{fixed}}}{\theta_{\max}} \times 100\% \quad (1)$$

$$\text{Shape recovery ratio: } R_r = \frac{\theta_{\max} - \theta_i}{\theta_{\max}} \times 100\% \quad (2)$$

$$\text{Shape recovery rate: } V_r = \frac{\pi(\theta_{fixed} - \theta_i)}{180t} \quad (3)$$

3. Results and discussion

3.1 Static tensile properties

Fig. 3 shows the fracture stresses of the samples at temperatures of 301 K ($T_t - 20$ K), 321 K (T_t) and 341 K ($T_t + 20$ K). The maximum fracture stresses were obtained in sample B2 and they showed an increase of 49.4%, 29.2%, 82.6% for Bulk, 15.3%, 8.4%, 10.6% for sample A at 301 K, 321 K and 341 K, respectively. It can also be seen in Fig 4 that the maximum Young's modulus were observed in sample B2. They achieved an increment of 414.8% and 215.9% in comparison with Bulk and sample A at 301 K, 639.1%, 361.7% at 321 K and 353.3%, 172.0% at 341 K, respectively. The reason for the improvement of the mechanical properties of the developed SMP hybrid nanocomposites are considered as follows: a) pits and functional groups were created on the surfaces of carbon fibers when they were treated by HNO₃ aqueous solution, the TPI matrix was bonded to the roughened surfaces easily owed to the pits and the functional groups [37]; b) both distribution [38] and geometry [39] are the most important factors for notable improvement in mechanical properties of nano-alumina-filler composites, and a reasonably good distribution of γ -alumina nanoparticles with large surface area of 130 m²/g (Table 1) was realized by appropriate dispersing process using magnetic stirrer and ultrasonic dispersion analyser in the present study; c) the 'nanoparticle bridge' enhanced the bonding forces between TPI matrix and carbon fiber [40] due to functional groups on the surface of carbon fibers [37] and hydroxide groups existing on the surface of alumina nanoparticles[41]. From Fig 3 and Fig4, one found that both the fracture stresses and Y-modulus decreased in samples B3 and B4. This is attributed to the weak zones (filler – filler cohesion) [42] resulted from the agglomeration of alumina nanoparticles [43] in the SMP hybrid nanocomposites.

Fig. 5 shows the SEM images of the fractured surfaces of the SMP hybrid nanocomposites after the static tensile tests. The cracks were observed around carbon fibers in all samples, and they were caused by debonding between SMP matrix and fibers, this is because the carbon fibers didn't use surfactants [44]. It indicates that failure of the combined mode of carbon fiber and SMP matrix is the major reason of the final destruction of the SMP hybrid nanocomposites in the static tensile tests.

Fig.5. SEM images of the fracture surfaces after static tensile tests.

Fig. 6 shows the fracture strains of all samples at temperatures of 301 K (T_t-20 K), 321 K (T_t) and 341 K (T_t+20 K). The maximum fracture strains were obtained in Bulk, and the minimum fracture strains were observed in sample B4 at all testing temperatures. This decreasing tendency of fracture strains in all samples were due to the composition structures among SMP matrix, carbon fibers and alumina nanoparticles limited the movement of SMP macromolecular chains when the samples were loaded.

3.2 Mechanical cyclic properties

Fig.7 depicts the maximum stresses of the samples under a constant strain cyclic loading. The maximum stresses were observed in sample B2 and they are 86.8%, 16.5% and 8.9% higher than Bulk, samples A and B4, respectively, in the first cycle. The increase-then-decrease development in mechanical cyclic properties was mainly caused by the increment of composite structures and the weak zones in the SMP hybrid nanocomposites. Fig.8 depicts that the residual strain decreases rapidly when the SMP matrix is reinforce by fillers. It is considered that this phenomenon is due to the excellent restorability of the composite

structures. In Fig 7 and Fig 8, both the maximum stresses and the residual strains tend to be constant with the increment of the number of cycles. This is expressed by the training effect of the SMP bulk reported and confirmed in [45, 46].

3.3 Thermo-mechanical cyclic properties

Fig.9. shows the stable stresses of samples in the case of 5 cycles under a constant strain cyclic loading at 341 K, 321 K and 301 K. The maximum stable stresses obtained in sample B2 which yields an increment of 83.1%, 270.6%, 141.1% for Bulk, 9.7%, 24.8%, 62.3% for A and 6.2%, 24.8%, 15.6% for sample B4 at 301 K, 321 K and 341 K, respectively. This result was mainly owing to the increment of composite structures and the weak zones in the SMP hybrid nanocomposites. Fig 10 shows that the minimum stable residual strains were shown in sample B2 and they are 11.8%, 16.4% and 15.6% lower than Bulk at 341 K, 321 K and 301 K, respectively. This indicates, irrespective of the temperature change, fillers play an important role on the residual strain in SMP composites.

3.4 Shape memory properties

The process of shape memory property tests is shown in Fig 11, and it was conducted according to ‘fold-deploy shape memory test’ in Fig 2. The test results show that the θ_{fixed} was equal to θ_{max} which indicates that the shape retention ratio is 100% and the SMP hybrid nanocomposites can fix to the desired shape completely.

Fig 12 depicts shape recovery ratios of samples from 301 K to 353 K. In the initial phase, the shape recovery ratios approaches zero, and when the temperature rises to T_i , the shape recovery ratio increases sharply. Finally, the shape recovery ratio increases slowly and approaches asymptotes when temperature exceeds the T_i . The shape recovery ratios of all samples began to increase sharply from T_i which is agreement with rules of thermodynamics of SMP. The shape recovery ratios of Bulk were higher than that of other samples above T_i , however, shape recovery ratios for all samples were above 90%. This clearly shows that the shape memory properties of the SMP hybrid nanocomposites in the context of shape recovery ratio are excellent.

Fig.11. The experimental process of shape memory properties tests.

Fig.12. The shape recovery ratios of samples under various temperatures.

Fig 13 shows shape recovery rates of samples under various temperatures. The shape recovery rates of all samples began to increase sharply from T_t and they increased with the increment of temperatures all the time. The shape recovery rate of Bulk was the highest, and that of samples A to B4 decreased in a small range. The test result is considered as follows: a) the inclusion interaction of fillers in shape memory polymer [47]; b) much energy was

needed when highly cross-linked structures achieve shape recovery [36]. More time was inevitable for the samples to recover the shape in the case that the temperature was fixed in the tests.

4. Conclusion

In the present study, the SMP hybrid composites with carbon fiber (7 wt%) and alumina nanoparticles (0–2 wt%) are developed. The shape memory properties and effect of fillers on mechanical and thermal properties of the developed SMP hybrid composites are investigated by experimental tests. The results obtained are summarized as follows:

1. The highest fracture stresses and the maximum Young's modulus are observed in sample B2 with 7 wt% chopped carbon fibers and 1 wt% alumina nanoparticles. The maximum fracture strains decreases with an increase in alumina nanoparticles weight fraction.
2. The maximum stresses and the residual strains tend to be constant with an increment in the number of cycles. All of the maximum stresses, maximum stable stresses, minimum residual strains and minimum stable residual strains are observed in sample B2 with

carbon fiber (7 wt%) and alumina nanoparticles (1 wt%).

3. The shape recovery rate decreases with an increment of alumina nanoparticle weight fraction. The shape memory properties of the SMP hybrid composites in both shape retention ratio and shape recovery ratio are excellent.

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