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Functionally graded carbon nanotube and nafion/silica nanofibre for electrical actuation of carbon fibre reinforced shape memory polymer

Purpose – To study the synergistic effect of self-assembled carboxylic acid-functionalised carbon nanotube (CNT) and nafion/silica nanofibre nanopaper on the electro-activated shape memory effect (SME) and shape recovery behaviour of shape memory polymer (SMP) nanocomposite.

Design/methodology/approach – Carboxylic acid-functionalised CNT and nafion/silica nanofibre are first self-assembled onto carbon fibre by means of deposition and electrospinning approaches, respectively, to form functionally graded nanopaper. The combination of carbon fibre and CNT is introduced to enable the actuation of the shape memory effect in SMP by means of Joule heating at a low electric voltage of 3.0-5.0 V.

Findings – Nafion/silica nanofibre is employed to improve the shape recovery behaviour and performance of the SMP for enhanced heat transfer and electrical actuation effectiveness. Low electrical voltage actuation and high electrical actuation effectiveness of 32.5% in SMP has been achieved.

Research implications – A simple way for fabricating electro-activated SMP nanocomposites has been developed by using functionally graded CNT and nafion/silica nanofibre nanopaper.

Originality/value – The outcome of this study may help the fabrication of SMP composites with high electrical actuation effectiveness under low electrical voltage actuation.

Keywords Carbon nanotube, Electrical actuation, Nafion/silica nanofibre, Shape memory polymer

Paper type Research paper

Introduction

Shape memory polymer (SMP) is one of the more fascinating stimulus responsive materials which are able to respond to a particular stimulus by means of altering their physical and/or chemical properties (Mather *et al.*, 2009; Xie, 2011; Sun *et al.*, 2012). Research and development on SMPs have become a very hot topic in recent years due to their excellent

and exciting properties, from high elastic strain, light weight, tailorable transition temperature, to a wide range of mechanical and physical properties (Lendlein and Langer, 2002; Meng and Hu, 2009; Liu et al., 2009; Lu and Gou, 2012; Hu et al., 2012; Zhao et al., 2012; Lu et al., 2013a; Lu et al., 2014a). SMPs are featured with the classic shape memory effect (SME), which enables the temporary shape to be virtually held unless a suitable stimulus is applied to induce the shape recovery (Rousseau, 2008; Nguyen et al., 2008; Xie 2010; Huang et al., 2010). In SMPs, the dual-segment/domain system is essential for the SME to maintain the temporary shape and to response to the external stimulus (Lu and Huang, 2013a; Lu and Huang, 2013b; Lu et al., 2013b; Sun et al., 2014; Lu and Du, 2014). Upon heating to above the transition temperature of the SMPs, the transition segment becomes soft and can be deformed to a temporary shape. On the other hand, the elastic segment could effectively prevent the SMP from returning back to its original shape due to the stored mechanical energy, which is the driving energy for the SME (Lendlein and Kelch, 2002; Dietsch and Tong, 2007). Since discovery of SMPs, tremendous progresses in chemical synthesis, characterisation, structure design, modelling and simulation of shape recovery behaviour have promoted the fast development of SMP materials (Tobushi et al., 2001; Miaudet et al., 2007; Sun and Huang, 2010; Maitland et al., 2002; Gall et al., 2004; Paik et al., 2006; Lan et al., 2009; Liu et al., 2009). Another major achievement related to SMPs is the actuation approach. Stimuli for SMPs include direct heating, Joule heating, induction heating, light, moisture or solvent etc (Ma, et al., 2013; Bellin et al., 2006; Tobushi et al., 2008). In these actuation approaches, the electrical actuation of SMPs by the electrically resistive Joule heating is desirable for many practical and potential applications, especially where direct heating is not conveniently achieved (Lantada et al., 2010). Therefore, a great number of research works have been carried out to achieve the electrical actuation of SMPs (Lu et al., 2009; Lu et al., 2010; Luo and Mather, 2010; Le et al., 2011; Lu et al., 2011b; Lu et al., 2011a; Fejos et al., 2012; Lu, 2012a; Lu, 2012b; Fejos and Karger-Kocsis, 2013; Lu et al., 2013; Lu et al., 2013c; Lu et al., 2014b). In practice, the use of a variety of electrically conductive filler is essential for the electrically conductive SMP composites due to the fact that most pristine SMPs are inertness to electricity. However, previous works have focused on the applicability of the SMP composite to be induced by the electrically resistive Joule heating. Thus, a variety of electrically conductive particles, fibres, electromagnetic particles, carbonbased nanopaper and mat have been embedded into the SMP matrix. Here, the lower electrical resistivity, the lower electric voltage was applied on the SMP composites to enable the electrical actuation to be conveniently achieved. Currently, it is essential to improve the electrical actuation with a high effectiveness, such as high recovery speed with low electric voltage, high recovery performance, uniform temperature distribution, high conductivity of electrically resistive Joule heating, to explore the great potential application of electrically conductive SMP composite.

In this study, we also aim at the electrical actuation with recovery performance and low energy loss. CNT and nafion/silica nanofibre was initially grafted onto the carbon fibre to form a functionally graded layer. Carbon fibre worked as the electric conductor to produce the electrically resistive Joule heat. CNT lay worked as the thermal conductor and was used to transfer the Joule heat to the underlying SMP matrix. On the other hand, the nafion/silica nanofibre worked as the thermal insulation to postpone the electrically resistive Joule heat exchanging between carbon fibre and the environment. Consequently, a series of experimental tests was carried out to characterise and analyse the electroactivated recovery performance and actuation effectiveness of SMP composite. The study was expected to provide an effective approach to significantly improve the actuation effectiveness of SMP nanocomposite induced by electrically resistive Joule heating.

Experimental

CNTs were received in powder form and had a diameter of 10-20 nm and a length of 1-15 μ m. CNTs were firstly dispersed into distilled water with a non-ionic surfactant C₁₄H₂₂O(C₂H₄O)_n. The surfactant had a hydrophilic polyethylene oxide group and a hydrocarbon hydrophobic group to aid the dispersion of CNTs in the distilled water. The CNT suspension was sonicated at 25°C for 20 minutes at an ultrasound power level of 300W. Two duty cycles were applied for the dispersion of CNTs. The CNF suspension was then filtered with a piece of hydrophilic polycarbonate membrane with the aid of a positive pressure of 0.6 MPa. After this, nafion solution and silica sols were mixed into a HCl (36.7%) solution as electrospun sols. The nafion/silica composite nanofibre (in a weight ratio of 40:60) was electrospun onto the opposite surface of the carbon fibre using a roller electrostatic spinning apparatus. In the process of electrospinning, the applied electric voltage between collector and electrode was kept at 30 kV. The electrode-to-collector distance was 15 cm, and the electrode rotation rate was 3 rpm. Following this, nafion/silica nanofibres were treated at 120°C for 2 hours to remove residue solvent.

An epoxy-based thermoset SMP resin was used to fabricate the composite via resin transfer moulding technique. Five carbon fibre mats incorporated with functionally graded CNT and nafion/silica nanofibre (at weights of 0 g, 0.08 g, 0.10 g, 0.12 g, 0.14 g, respectively) were prepared to reinforce the polymer matrix. After filling the mould, the mixture was cured at a 100°C and kept at that temperature for 5 hours before being ramped to 120°C at

20°C per 180 minutes. Finally, it was heated to 150°C at 30°C per 120 minutes to produce the final SMP composites.

Results and discussion

Morphology of nafion/silica nanofibre composite onto carbon fibre

Scanning electron microscopy (SEM) was used to characterise the morphology and structure of nation/silica nanofibre assembly, as shown in Figures 1, which provides a typical surface view of the nafion/silica nanofibre at scale of 20 µm. Hybrid nafion/silica nanofiber with a belt structure having a diameter ranging from 2 to 5 µm was observed. No large aggregate of nanofibre and a multi-scale porous structure was found. A network structure was formed by the mechanical interlocking between individual nanofibre. The composite nafion/silica nanofibre was uniformly coated on the entire surface of carbon fibre [shown in Figure 1(a)]. Therefore, the assembly of nation/silica nanofibre is expected to prevent the heat transfer and exchange from the underlying carbon fibre to the atmosphere due to their thermally resistive properties. In our previous report, hybrid nation/silica nanofibre with low thermal conductivity was found to have a good thermal stability to resist to 600°C (Yao et al., 2015). In the electropinning process, nafion nanofibres were rearranged and coated on surface of the silica nanofibre as a shell under a high electric voltage. Strong chemical bond formed between nation and silica nanofibres. The excellent thermal insulation is attributed to the porous structure and the strongly chemical bonding between sulphonic group and silanol group of the silica and nafion, respectively.

Effect of nafion/silica nanofibre on the temperature distribution in carbon fibre mat

The nafion/silica nanofibre was employed to postpone the heat transfer, reduce the loss dissipation and therefore improve the electrical actuation effectiveness. Electric voltages of 3.0 V, 4.0 V and 5.0 V were applied on the carbon fibre mat grafted with 0.08 g CNT and 0.08 g nafion/silica nanofibre. The temperature distribution in both the CNT and the nafion/silica nanofibre surfaces is recorded as a function of heating time, as shown in Figure 2. It was found that the temperature distribution in CNT surface is higher than that in the nafion/silica nanofibre surface under the same electric voltage. The temperature distribution in the CNT surface reached 77.14°C, 135.14°C and 195.11°C within a heating time of 180 seconds with an electric voltage of 3.0 V, 4.0 V and 5.0 V, respectively. On the other hand, it reaches 69.74°C, 127.69°C and 186.79°C in the nafion/silica nanofibre surface. These experimental results are attributed to the thermally resistive nafion/silica nanofibre, of which the thermal conductivity is low and postpones the heat transfer from the carbon fibre mat to the environment.

Furthermore, the effect of weight concentration of nafion/silica nanofibre on the temperature distribution in carbon fibre mat has been studied and characterised. Figure 3(a) plots the relationship between the temperature and the heating time of the carbon fibre mat grafted with 0.08 g nafion/silica nanofibre and CNT (0.06 g, 0.08 g and 0.12 g, respectively). It could be seen that with an increase in the weight content of CNT, the temperature is gradually increased against the heating time under an electric voltage of 3.0 V. Experimental results also revealed that more electrically resistive heat had been transformed due to the increase in weight concentration of CNT, resulting from the temperature distribution having been raised. However, with the decrease in the weight concentration of CNT increased), the difference in the temperature distribution between CNT and nafion/silica nanofibre surfaces was significantly decreased. The effective energy ratio is determined by the difference in temperature distribution of CNT surface (T_{CNT}) and nafion/silica

nanofibre surface $(T_{N/S})$ as expressed as: $\frac{T_{CNT} - T_{N/S}}{T_{CNT}} \times 100\%$. Such a parameter is used

to characterise the amount of electrically resistive heat that is required to induce the SME in SMP, which has been employed to characterise the difference in the temperature distribution between two surfaces, as shown in Figure 3(b). These experimental results demonstrate that the nafion/silica nanofibre could be useful to postpone the heat transfer and reduce the loss dissipation. With a decrease in the weight concentration of nafion/silica nanofibre (where the increase in the weight concentration of CNT), the effective energy ratio is approximately decreased from 32.5% to 5%. The thermally resistive nafion/silica nanofibre plays a critical role in enhancing the effective energy ratio of the electrical actuation of SMP nanocomposite by means of electrically resistive heat.

Effect of CNT on the temperature distribution in carbon fibre mat

The nafion/silica nanofibre has been proven to have the potential of improving the electrical actuation effectiveness for the SMP nanocomposite by means of electrical actuation, according to its good heat resistant and low loss dissipation. The effect of CNT on temperature was also necessary to be investigated. In the measurement of temperature distribution, it has been collected from the nafion/silica nanofibre surface under an electric voltage of 2.0 V via an infrared camera. The temperature distribution in the nafion/silica nanofibre surface is monitored as a function of heating time, as revealed in Figure 4. Experimental results show that the temperature in the nafion/silica nanofibre surface is decreased from 66.30°C, 66.23°C, 53.92°C, 50.53°C to 47.36°C with an increase in the weight

content of CNT raised from 0.06 g, 0.08 g, 0.10 g, 0.12 g to 0.14 g, respectively. These experimental results could be contributed to the excellently electrical conductivity of the CNT. When the electric current is transformed into the electrically resistive heating in the carbon fibres, the electrically resistive energy is easier to transferring from the carbon fibres to the CNT part than the nafion/silica nanofibre due to their difference in the thermally conductive properties. Here, the amount of the electrically resistive energy is raised to transfer to the CNT part as an increase in the weight content of the CNT. Therefore, the amount of electrically resistive energy transferred to nafion/silica nanofibre part is decreased, resulting in the temperature distribution significantly being decreased.

Electrically triggered shape recovery performance

The SMP nanocomposite specimen had a permanent flat shape (with a dimension of 60×60×15 mm³). After the test nanocomposite was heated above 120°C, it was bent into a "U" shape under an external force. A constant 4.0 V DC voltage was applied to the deformed specimen. Snapshots of the shape recovery sequence are shown in Figure 5. The test SMP nanocomposite specimen took 150 seconds to complete the shape recovery from the temporary deformation shape. It showed a small recovery ratio during the first 30 seconds. On the other hand, it exhibited faster recovery behaviour until 120 seconds and no noticeable deformation could be further found. Finally, the SMP nanocomposite regained its permanent shape and the recovery ratio is approximate to 92% compared with its permanent shape by means of the electric current. As can be seen from the images presented, the SMP nanocomposite was heated by the electrically resistive heat and the temperature in specimen was increased above the transition temperature of SMP matrix, resulting into a shape recovery. It should be noted that the increased electric current could raise the shape recovery speed of the tested specimen under the same electric voltage due to the high electric power based on the Ohmic rule.

To further characterise the synergistic effect of CNT and nafion/silica nanofibre on the electrical actuation behaviour of the SMP nanocomposite, a comparison in electrical actuation of SMP nanocomposite was carried out under an electric voltage of 3.0 V, 4.0 V and 5.0 V, respectively, as shown in Figure 6. Experimental results reveal that the SMP nanocomposite show a faster heating speed (in 50 s) under an electric voltage of 5.0 V, while that of SMP nanocomposite are 90 seconds and 100 seconds under the electric voltage of 4.0 V and 3.0 V, respectively. These experimental results are attributed to the Ohmic rule, that is the higher electric voltage, the higher electric power applied, resulting in the SMP nanocomposite being heated above its transition temperature and the SME driven by the

electrically resistive heat. Furthermore, the large difference in the electrical and thermal conductivities between carbon fibre and SMP matrix has been complimentary owing to the synergistic effect of CNT and nafion/silica nanofibre. The applied electric voltage could be increased to accelerate the recovery speed of the SMP nanocomposite induced by the electrically resistive heating. Otherwise, the interface between the carbon fibre and SMP matrix would be thermally damaged. Thus, the electrically induced shape recovery performance of the SMP nanocomposite has been significantly improved by the synergistic effect of CNT and nafion/silica nanofibre.

Conclusions

A series of experiments were conducted to study the synergistic effect of CNT and nafion/silica nanofibre on electrical actuation of carbon fibre reinforced SMP nanocomposite, of which the actuation was achieved by electrically resistive heating. CNTs were grafted onto the carbon fibre to help the heat transfer from carbon fibre to the SMP matrix. Nafion/silica nanofibre composite was electrospun onto opposite surface of carbon fibres to significantly improve the temperature distribution and shape recovery performance of the carbon fibre reinforced SMP nanocomposite. Furthermore, the effects of the weight content of CNT and nafion/silica nanofibre on the temperature distribution and shape recovery performance have been systematically investigated and discussed. A critical role of the nafion/silica nanofibre in improving the electro-activated shape recovery behaviour has been summarised.

Conflict of Interest

The authors declare no conflict of interest.

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Figure 1 Typical surface view of composite fibre mat (a) and the nafion/silica nanofibre electrospun (b)



Figure 2 Temperature distribution as a function of heating time of the CNT surface and nafion/silica nanofibre surface under the electric voltage of 3.0 V, 4.0 V and 5.0 V, respectively



Figure 3 (a) Temperature distribution as a function of heating time of the CNT surface and nafion/silica nanofibre surface with an increase in the weight concentration of CNT; (b) The evolution change in effective energy ratio as a function of heating time for the carbon fibre mat incorporated with 0.06 g, 0.08 g and 0.12 g CNT





Figure 4 Temperature distribution in the nafion/silica nanofibre surface as a function of heating time with an increase in the weight concentration of CNT, where the 2.0 V electric voltage was applied on the carbon fibre mat



Figure 5 Snapshot of Joule heating-induced shape recovery in SMP nanocomposite incorporated with 0.08 g CNTs under an electric voltage of 4.0 V



Figure 6 A comparison in shape recovery ratio of the SMP nanocomposites incorporated with 0.08 g CNT as a function of heating time under an electric voltage of 3.0 V, 4.0 V and 5.0 V

