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The effects of various carbon nanofillers on the thermal properties of paraffin for energy storage applications

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Abstract The addition of carbon nanofillers to phase change materials (PCMs) has received much attentions recently. The shape effects of carbon nanofillers on the thermal properties of paraffin were investigated in this work. Four types of nanocomposite PCMs were prepared by mixing paraffin with multi-wall carbon nanotubes (MWCNTs), graphitized MWCNTs, nanographene with different layer numbers (GNP-B, GNP-C). It shows that phase change enthalpy of nanocomposite PCM decreases gradually with the increasing of the loading of carbon nanofillers. Due to the nucleating action of carbon nanofillers, the supercooling degree of nanocomposite PCM slightly decreases. The microstructure of the nanofillers plays an important role in enhancing the thermal conductivity of paraffin. It is the flaky structure of GNP shows a more significant effect on enhancing the thermal conductivity of paraffin than the tubular structure of CNTs, which is confirmed by comparing the melting/freezing time of PCM. In the four kinds of carbon nanofillers, GNP-C shows the greatest thermal conductivity enhancement up to 52.4 % at the loading of 3 mass%.

Keywords Carbon nanofillers · Paraffin · Nanocomposite PCM · Thermal conductivity · Supercooling degree

Introduction

Thermal energy storage technology plays a critical role in storing the solar energy and recovering the waste heat [1]. Among the various energy storage materials, PCM is a hot research topic because it absorbs and releases a large quantity of heat at a constant temperature during the phase change process.

Paraffin, a typical organic PCM, has long been practiced in low-to-medium temperature thermal energy storage due to its high latent heat, low cost, stability, nontoxicity and resistivity to corrosion [2]. However, the inherent low thermal conductivity of paraffin [3] seriously affects the energy storage system performance. To enhance the heat transfer rate, lots of methods have been proposed, such as preparing the encapsulated PCM using polymers or inorganic materials as the shell and PCM as the core [4, 5], preparing the shape-stabilized PCM by inserting a metal structure or impregnating a porous material [6], and dispersing high thermal conductivity particles in PCM [7, 8]. Currently, carbon material has attracted considerable attention because of its superior thermal conductivity of 2000–6000 W m^{-1} K^{-1} [9]. Meanwhile, there are many kinds of carbon materials, such as carbon nanotubes, graphene nanoplatelets, carbon nanofibers. Warzoha and Fleischer [10] stated that carbon-based nanoparticles drastically reduced the phase change time of PCM compared with Al/paraffin and TiO_2/paraffin nanocomposites. Zhang and Zhao [11] studied the thermal properties of aqueous suspensions of microencapsulated phase change materials and MWCNTs. A maximum enhancement of 8 % in thermal conductivity was achieved for 1 mass% MWCNTs. Harish et al. [12] found that the inclusion of GNPs enhances the thermal conductivity of PCM by 230 % at a loading of 1 vol%. Li et al. [13] found the thermal

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conductivity of stearic acid was improved by 11.4 % and the phase change enthalpy was reduced by 6.7 % when adding 3.0 mass% MWCNT. As shown in the above results, the kind of carbon materials has great effects on the thermal property of nanocomposite PCM [14]. Thus, the comparison study of the addition of various carbon nanofillers is very necessary. Fan et al. [15] compared the effects of short and long MWCNTs, GNPs and carbon nanofibers on the thermal conductivity of nanocomposite PCM. It was found that GNPs had the greatest relative enhancement up to 164 % at the loading of 5 mass%. Karaipeklia et al. [16] stated that the thermal conductivity of expanded graphite/stearic acid was higher than that of carbon fiber/stearic acid. Shaikh et al. [17] reported that the types of carbon nanofillers (single-wall CNTs, MWCNTs and carbon nanofiber) had obvious effects on the latent heat of PCM. By molecular dynamics simulations, Babaei et al. [18] stated that the thermal conductivity enhancement of GNPs/octadecane was higher than that of CNTs/octadecane. Although many references reported the effect of adding various carbon nanostructures on the thermophysical properties of PCM [7, 19], it is limited to simultaneously compare the performance of the multi-carbon nanofillers on the supercooling degree and the thermal conductivity.

In this paper, a series of nanocomposite PCM were prepared by paraffin homogeneously mixing with four kinds of carbon nanofillers, including MWCNTs, G-MWCNTs, GNP-C and GNP-B, respectively. The shape effects of carbon nanofillers on the thermal properties of paraffin were investigated, such as the phase change enthalpy, the supercooling degree, thermal conductivity and melting/freezing times.

Materials and methods

Materials

Paraffin (melting point: 58–60 °C) used in the experiment was supplied by Shanghai Huashen Recover Equipment Co., China. Four different types of carbon nanofillers (GNP-B, GNP-C, MWCNTs and G-MWCNTs) were used. GNP-B and GNP-C are nanographene with different layer numbers. MWCNTs are the multi-wall carbon nanotubes with a small diameter. G-MWCNTs are the graphitized multi-walled carbon nanotubes. The suppliers and specifications of these carbon nanofillers are listed in Table 1. All materials were used without any further purification.

Preparation of nanocomposite PCM

The preparation process of nanocomposite PCM was as follows. Firstly, to ensure the good compatibility between carbon nanofillers and paraffin, all carbon nanofillers were placed in the vacuum drying oven at 100 °C for 12 h. Secondly, the carbon nanofillers were slowly added to the molten paraffin with magnetic stirring for 15 min at 75 °C. Then, the suspensions were transferred into the ultrasonic vibrator for 1 h. The ultrasonic temperature was above 58 °C to ensure that the samples were kept at the molten state. The concentrations of the nanocomposite PCM were 0.2, 0.5, 1, 2 and 3 mass%, respectively.

Analysis methods

The microstructure of the carbon nanofillers and the nanocomposite PCM was examined using a scanning electron microscope (SEM, JSM-7500F, JEOL, Japan). A thin gold layer was coated on the surface of the nanocomposite PCM before SEM observation. However, it was no need for the highly conductive carbon nanofillers. Infrared spectra of solid PCM were obtained using a Fourier transform infrared spectrometer (FTIR, Nicolet 6700, USA) in the range of 4000–400 cm^{-1}. The thermal performances of PCM were measured using a differential scanning calorimeter (DSC-Q10, TA Instrument Inc., USA) under N_2 atmosphere. Samples were sealed in an Al crucible, weighing in the range of 4–8 mg. A complete DSC running was consisted of a heating and a cooling process between 25 and 75 °C with a heating/cooling rate at 5 °C min^{-1}. The phase change temperatures were estimated by the tangent at the point of greatest slope on the face portion of the peak of the DSC curve. The phase change enthalpies were determined by numerical

Table 1 Suppliers and specifications of the various carbon nanofillers

Materials	Suppliers	Dimensions	Purity/mass%
GNP-B	Beijing Qingdajiguang Technology Development Co. Ltd.	d^a: 100–200 nm, h: 0.335 nm, p: 3–5	>99
GNP-C	Chengdu Organic Chemicals Co. Ltd.	d: 5–10 µm, h: 4–20 nm, p: $<$ 30	>99.5
MWCNTs	Chengdu Organic Chemicals Co. Ltd.	l: $>$ 50 nm, d: 8–15 nm	>95
G-MWCNTs	Chengdu Organic Chemicals Co. Ltd.	l: $>$ 50 nm, d: 10–20 nm	>99.9

[a] d, h, p, l stand for out diameter, thickness, layer number and length, respectively

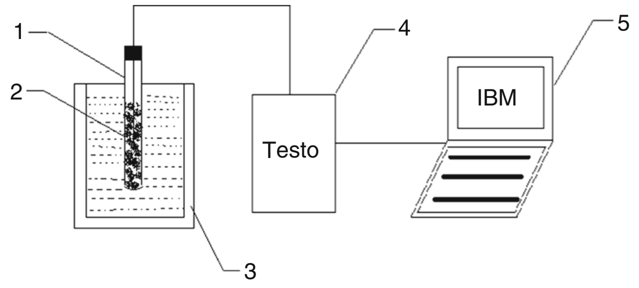

Fig. 1 Experimental system of the melting/freezing process. *1* glass tube, *2* thermocouple, *3* thermostatic water bath, *4* temperature data logger, *5* computer

integration of the area under the peaks. Thermal conductivity was measured by a Hot Disk thermal constants analyzer (Hot Disk Inc., Uppsala, Sweden).

Thermal performance

To investigate the effect of the addition of the carbon nanofillers on the heat storage/release performance, a latent thermal energy storage system was conducted, shown in Fig. 1. The glass tubes with an outer diameter of 30 mm and a wall thickness of 2 mm were immersed in a thermostatic water bath, which controlled the temperature in the melting and freezing processes. During melting, the heating temperature was kept constant at 75 °C, whereas freezing with the cooling temperature at 14 °C. The different kinds of PCM were enclosed in the glass tubes, respectively. K-type thermocouples were fixed at the center of the tubes to measure the temperature of PCM, which was collected by a Testo data logger at a time interval of 1 s.

Results and discussion

Microstructure of the carbon nanofillers and the nanocomposite PCM

The SEM images in Fig. 2 reveal the received carbon nanofillers. It is clearly seen that MWCNTs and G-MWCNTs present as tubular structures. The diameter of G-MWCNTs is bigger than that of MWCNTs, which is in agreement with those supplied by the sellers (see Table 1). The entangling phenomenon of MWCNTs is very seriously, which will affect its long stability dispersing in paraffin. GNP-B and GNP-C show flaky layers. According to the information of diameter and thickness in Fig. 2, GNP-C should have a significantly higher diameter-to-thickness ratio than GNP-B.

Figure 3 shows the images of the carbon nanofillers dispersing in paraffin. It is clear that the carbon nanofillers are well coated with multiple layers of paraffin, which will be detrimental to yield the percolating networks for thermal

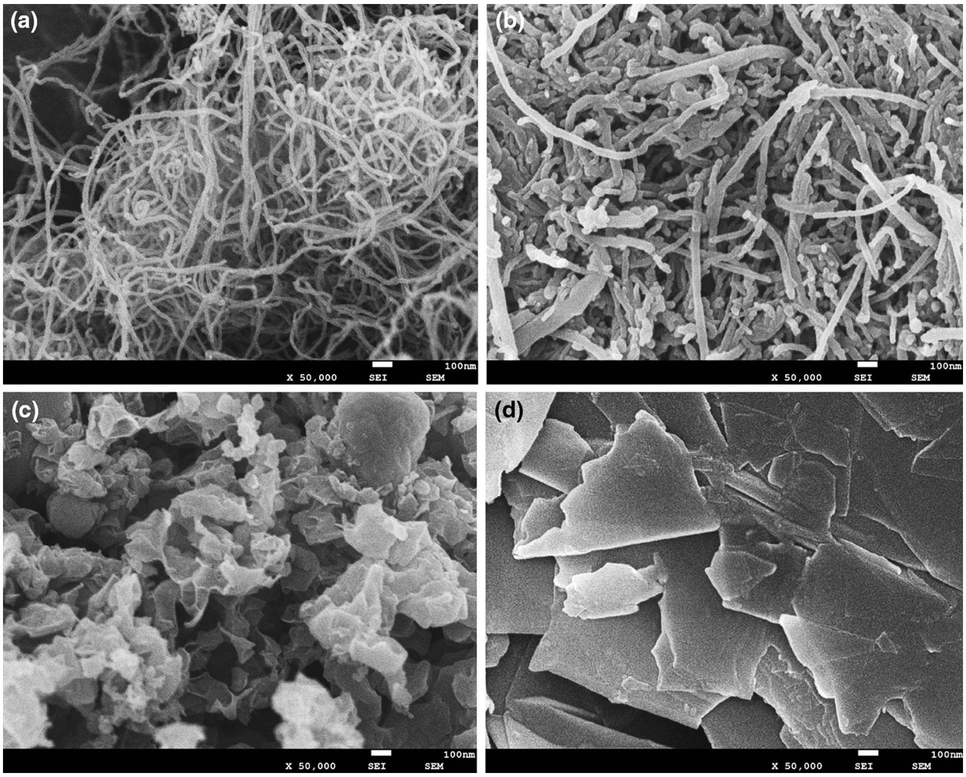

Fig. 2 SEM images of various carbon nanofillers **a** MWCNTs, **b** G-MWCNTs, **c** GNP-B, **d** GNP-C as received

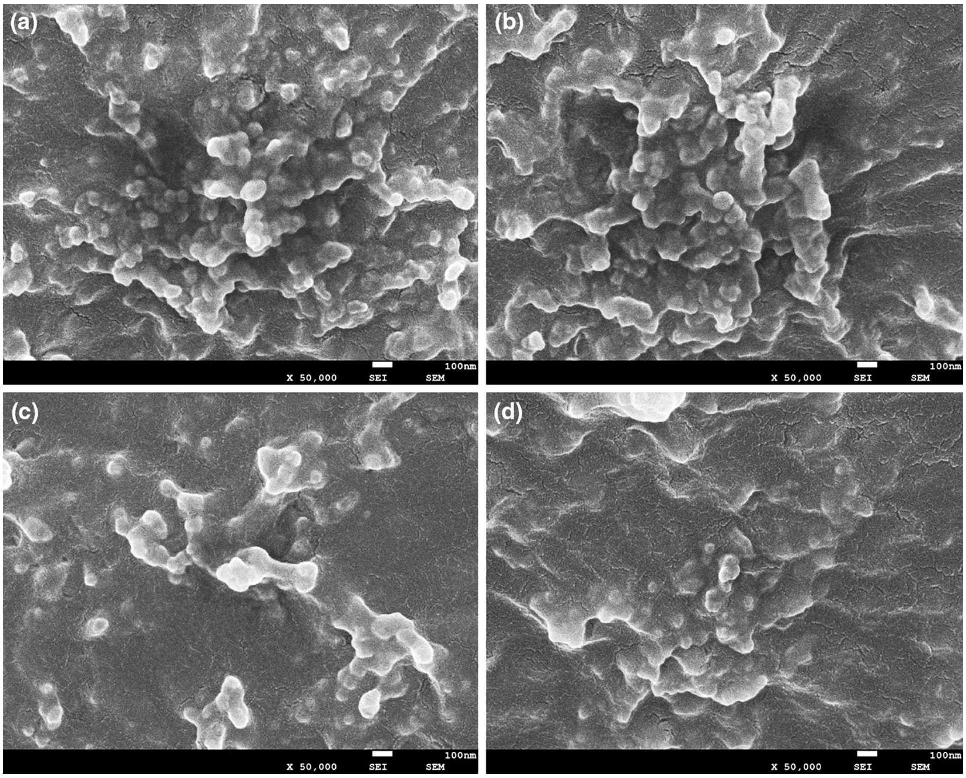

Fig. 3 SEM images of various carbon nanofillers **a** MWCNTs, **b** G-MWCNTs, **c** GNP-B, **d** GNP-C dispersed in paraffin

transport within the nanocomposites [20], especially for MWCNTs and G-MWCNTs. Moreover, the surface of the composites shows different images due to the difference of tubular and flaky structures of the carbon nanofillers.

FTIR analysis of nanocomposite PCM

The interaction between paraffin and carbon nanofillers was characterized using the FTIR analysis as shown in Fig. 4. Different concentrations of the same kind of nanocomposite PCM have the same FTIR curves. So, it displays only one curve for one kind of nanocomposite PCM in the figure. The FTIR spectra of the five samples are completely the same, and all the peaks belong to paraffin. The vibration spectra observed at 2915 and 2845 cm^{-1} are ascribed to the C–H stretching vibration of CH_3 and CH_2. The absorption bands obtained at 1455 and 719 cm^{-1} are primarily assigned to C–H bending vibration and in-plane deformation rocking vibration of paraffin molecular, respectively. Obviously, there are no new vibration bands forming in the nanocomposite PCM, compared to pure paraffin. It indicates that there is no change in the molecular structure of paraffin in the presence of carbon nanofillers [21], which enable the nanocomposite PCM to keep the original characteristics of paraffin, such as chemical stability.

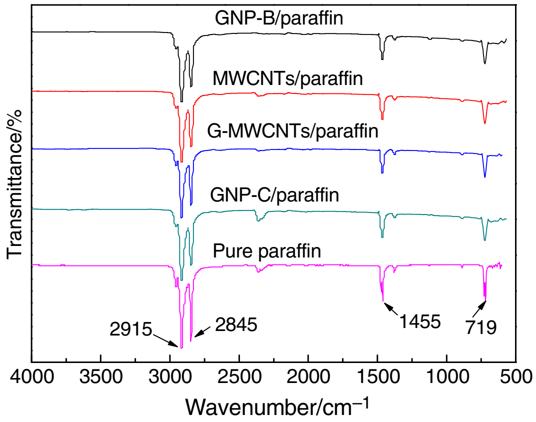

Fig. 4 FTIR spectra of pure paraffin and nanocomposite PCM

Phase change temperature and enthalpy

DSC analysis was conducted to investigate the influence of various carbon nanofillers on the thermal properties of nanocomposite PCM. Considering the similarity of the curves, the DSC curves of the different nanocomposites were only shown with 0.5 mass%, compared with pure paraffin in Fig. 5. It shows all kinds of nanocomposite PCM have similar heating/cooling cycles, which mean all PCM have very similar phase change properties. The main

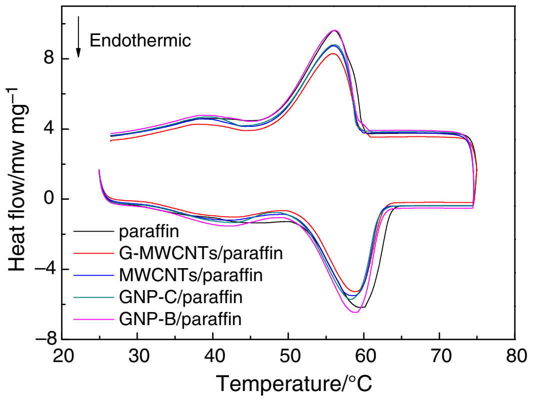

Fig. 5 DSC curves of nanocomposite PCM

peak corresponds to solid–liquid phase change in paraffin, whereas the minor peak to solid–solid phase transition [22].

The phase change temperatures of nanocomposite PCM are shown in Fig. 6. There is no clear relationship between the phase change temperature and the loading of the carbon nanofillers. Meanwhile, the type of the carbon nanofillers has slight influence on the phase change temperatures, which of different nanocomposite PCM all fluctuate around those of paraffin. The maximum changes on the melting and freezing temperatures are 0.74 and 1.11 °C, respectively.

The difference between the melting and freezing temperatures is taken as the supercooling degree, which is an important parameter that will impede the energy recovery of during the heat cycle [23]. As can be seen from Fig. 7, the decrease in the supercooling degree is observed in all various nanocomposite PCMs with different mass fractions. However, the relationship between the supercooling degree and the loading of the carbon nanofillers is not clear. The supercooling degree of nanocomposite PCM has a maximum decrease of 9.5 % for MWCNTs at 1 mass%, 14 % for G-MWCNTs at 0.5 mass%, 14.4 % for GNP-C at 3 mass% and 11.4 % for GNP-B at 1 mass%. The carbon nanofillers act as the nucleating agent providing the crystallized surface, which will be beneficial to reduce the supercooling degree. Park et al. [24] also stated the supercooling degree of paraffin@polyurea PCM nanocapsules was decreased by incorporating Fe_3O_4 nanoparticles.

The phase change enthalpy is also one of important parameters to estimate a good performance PCM. In Fig. 8, the addition of carbon nanofillers into paraffin all resulted in lower melting and freezing enthalpies [25], which decrease gradually with the increasing of the loading of carbon nanofillers. Among the four kinds of carbon nanofillers, MWCNTs-based PCMs show the highest phase change enthalpies at the same mass fraction. At the highest loading of 3 mass% for MWCNTs, G-MWCNTs, GNP-C and GNP-B, the melting enthalpies are lowered by 7.3, 13, 12 and 12.8 % compared with pure paraffin for 196.2 J g^{-1}, respectively. Similarly, the freezing enthalpies are lowered by 11.2, 19.9, 17.8 and 11.4 %. These enthalpy values are still suitable for latent heat storage applications [26]. Since

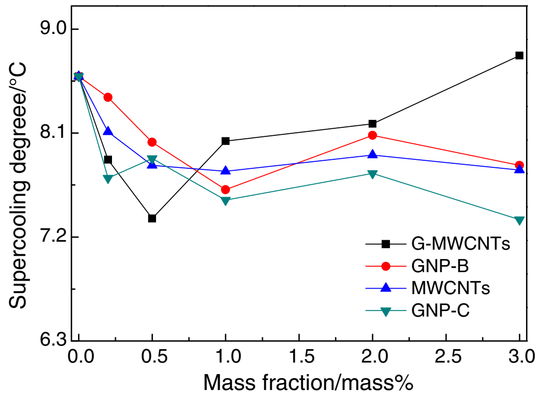

Fig. 7 Comparison of the supercooling degree of paraffin-based nanocomposite PCM

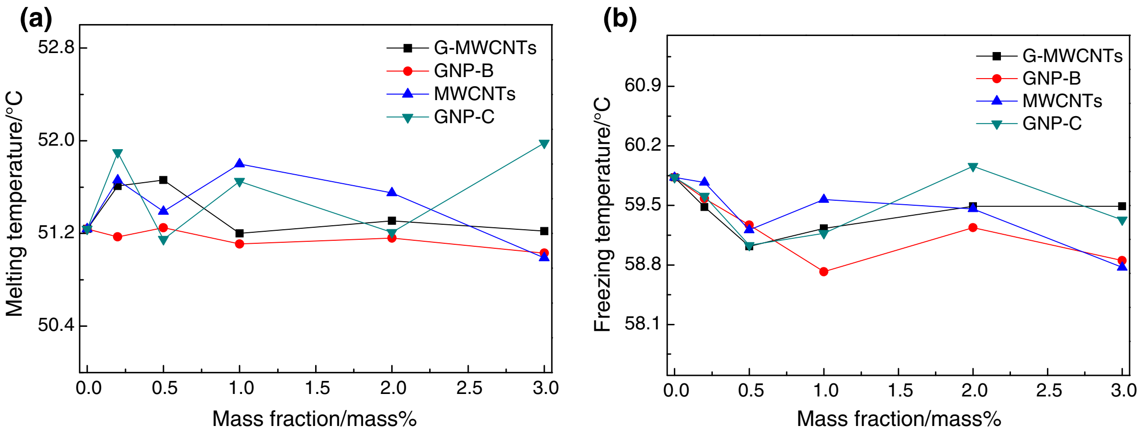

Fig. 6 Comparison of the phase change temperatures of paraffin-based nanocomposite PCM. **a** Melting, **b** freezing

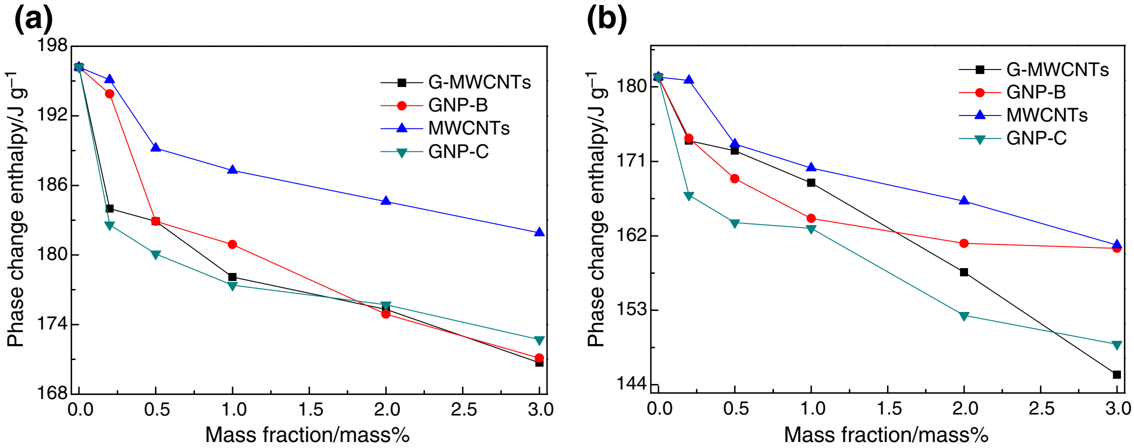

Fig. 8 Comparison of the phase change enthalpies of paraffin-based nanocomposite PCM. **a** Melting, **b** freezing

the carbon nanofillers do not undergo phase change during the test temperature of 25–75 °C, the melting and freezing enthalpies of the nanocomposite PCM are expected to be somewhat lower than those of pure paraffin. However, the main reason probably may be the realignment of molecules of the matrix PCM in the presence of carbon nanofillers [27, 28].

Thermal conductivity enhancement

The heat transfer rate is highly dependent on the thermal conductivity of materials. As show in Fig. 9, adding carbon nanofillers to paraffin results in a higher thermal conductivity. However, the thermal conductivity enhancement ratio is closely related to the shape of the carbon nanofillers. The enhanced ratio of thermal conductivity of CNTs/paraffin is lower than that of GNP/paraffin, which is due to their different microstructures. GNP-B and GNP-C both are a two-dimensional planar structure. G-MWCNTs and MWCNTs are a three-dimensional structure. As reported by Yu et al. [29], the planar structure is beneficial to reduce the phonon scattering at the interfaces between nanofillers and paraffin. In addition, CNTs tend to aggregate into bundles and settle down when dispersed in paraffin, which makes it difficult to realize their full potential to improve the thermal conductivity of nanocomposite [30]. The comparisons with different carbon nanofillers indicate the superiority of GNP-C as a conductive nanofiller for organic PCM. The addition of GNP-C at 3 mass% increases the thermal conductivity of paraffin by 52.4 %.

Due to the high thermal conductivity of carbon nanofillers, one might expect a more dramatic improvement in thermal conductivity of nanocomposite PCM with the addition of carbon nanofillers. However, due to the poor phonon coupling at the paraffin-filler interfaces and the oxidation of carbon nanofillers in the preparation process [31], the thermal conductivity of the nanocomposite PCM is lower than the expected value. The similar results were also reported by Fan et al. [15]. He showed that the thermal conductivity enhancement in paraffin containing S-MWCNTs reached about 30 % at 5 mass%.

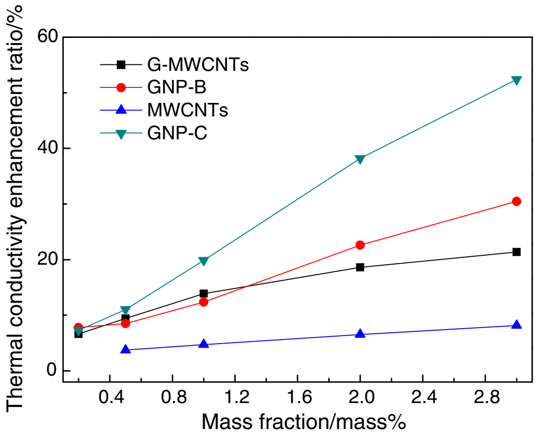

Fig. 9 Comparison of the thermal conductivity enhancement of paraffin-based nanocomposite PCM

Thermal performance of nanocomposite PCM

The enhancement of thermal conductivity was verified by comparing the melting and freezing times of the nanocomposite PCM with those of paraffin. The thermal performance experiment was only gone on with G-MWCNTs/paraffin and GNP-C/paraffin with 0.5 mass%, which represented the tubular and flaky structures of the carbon nanofillers, respectively. Temperature curves for the phase change process of PCM are shown in Fig. 10. During the melting process, pure paraffin needs 1485 s to reach to the bath temperature, while

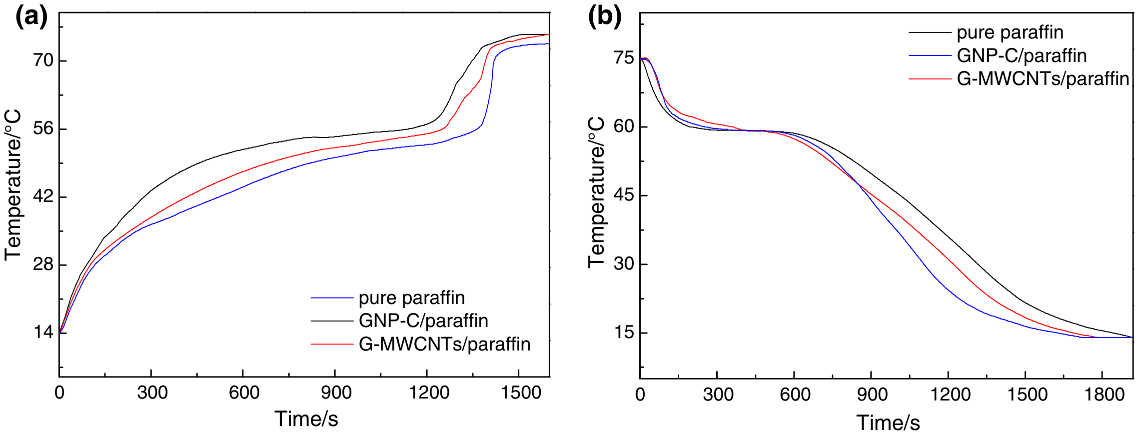

Fig. 10 Temperature curves of PCM during **a** melting process and **b** freezing process

the time is 1384 and 1416 s for GNP-C/paraffin and G-MWCNTs/paraffin. During the freezing process, the time is 1918, 1721 and 1780 s for pure paraffin, GNP-C/paraffin and G-MWCNTs/paraffin to reach 14 °C, respectively. This result verifies the higher thermal conductivity and the more intensive heat transfer process of nanocomposites PCM compared with pure paraffin. Meanwhile, it is obvious that GNP-C/paraffin has a higher heat transfer rate than G-MWCNTs/paraffin, which is in agreement with the reports for comparing the melting times of expanded graphite/stearic acid and carbon fiber/stearic acid by Karaipeklia et al. [16].

Conclusions

In this paper, the thermal properties of paraffin-based nanocomposite PCM with four kinds of carbon nanofillers have been investigated. The results show that the phase change enthalpy and the supercooling degree decrease due to the addition of carbon nanofillers. Their variations have no significant relationship with the kinds of the carbon nanofillers. The thermal conductivity enhancement ratios of paraffin increase with the loading of nanofillers. The microstructure of the nanofillers plays an important role in enhancing the thermal conductivity. In the four types of carbon nanofillers, GNP-C exhibits to have a greatest relative enhancement up to 52.4 % at the concentration of 3 mass%, due to its two-dimensional planar structure. The comparison of the melting/freezing times of PCM also confirms that the heat transfer rate of the flaky structure of GNP-C nanofillers is higher than that of the tubular structure of G-MWCNTs nanofillers.

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