



Preparation and thermal properties of short carbon fibers/erythritol phase change materials



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ABSTRACT

The thermal properties of the short carbon fibers (SCFs) filled erythritol phase change composites (PCCs) were investigated experimentally. The samples were prepared with different mass loadings of two kinds of SCFs, 1%, 2%, 4%, 7% and 10%. The melting points and phase change enthalpies were measured by differential scanning calorimeter (DSC). The effects of SCFs on the melting points are relatively small but the enthalpies were reduced with the loadings of SCFs. The greatest loss of enthalpies is 11.3% for composites filled with 10% SCFs. The thermal conductivities increased with the loadings of SCFs but not linearly. The highest thermal conductivity is 3.92 W/(m·K) for the composites with 10% longer SCFs, which was enhanced by 407.8% compared to pure erythritol (0.77 W/(m·K)). Composites filled with longer SCFs possess higher thermal conductivity and the mechanisms were discussed. A simple setup was made to test the temperature-regulated property of these materials. These include pure erythritol and phase change composites with different loading of SCFs. The PCCs have shown good application potential and the longer SCFs can lead to the better performance of PCCs.

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1. Introduction

Recently, phase change materials (PCMs) have received great interests in many fields [1,2] and they are regarded as the key in the thermal energy storage [3–5], cooling applications [6–8] and building energy management [9,10]. Most of the PCMs possess many advantages in the meantime, such as high phase change enthalpy, negligible volume change during phase changing process and chemically stability [11,12]. The common and major shortcoming that limits their application is the low thermal conductivity. This results in slow heat transfer rate during the charging and discharging process. For most PCMs, like paraffin wax [11,13], stearic acid [14], erythritol [12,15,16] and palmitic acid (PA) [17], their thermal conductivities are undesirable for application. Erythritol possesses the highest thermal conductivity among them which is only about 0.7 W/(m·K), while the thermal conductivities of the others are in the range of 0.15–0.3 W/(m·K). The most common method to increase thermal conductivity is by adding highly thermal conductive fillers. To avoid ambiguity in the text, the abbreviation ‘PCMs’ refers to the phase change materials without fillers, and ‘PCCs’ refers to the composites with fillers.

Various materials have been tried and studied as fillers, such as metal particles [12], metal foams or porous materials [15,18–20] and different kinds of carbon materials. Carbon materials possess some great advantages over the traditional metal materials. The thermal conductivities are higher, which are of the 1000 W/(m·K) order. Their densities are usually no more than 2.26 g/cm³, which is only a fraction of metals’. Moreover, high chemical stability to avoid reaction with PCMs is another superiority of carbon materials. Several kinds of carbon materials have been tried, such as expanded graphite (EG), carbon nanotubes (CNTs), multi-walled carbon nanotubes (MWNTs), carbon nanofibers (CNFs) and graphene or graphene nan platelets (GNPs). However, the most common carbon fibers have been little studied, which is cheaper but also widely used in various composites [21–23].

The loadings of fillers are the most studied influence factor for thermal conductivity. Besides, the geometry of fillers has also been proved to affect the thermal conductivities of PCCs, but a common conclusion has not been reached yet [24–27]. For the widely-used nanomaterials, they are usually agglomerated because of their small size and this phenomenon can be observed in the micrographs [17,24,28]. It will inevitably lead to errors when they are used to study the geometric effects because the size and shape of nanomaterials may change when embedded in the PCMs matrix.

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In comparison, carbon fibers are much larger and more rigid, so they can keep their original geometry in composites.

One of the common application of PCMs is temperature-regulation of various kinds of devices. Many devices such as the wearable computers, cellphones and precision instruments [8,29] can only operate within a limited temperature range. However, their temperature will rise at work because of the excessive heat. This reduces the performance of columns and even damage them. The PCMs can be used as a passive cooling technique means to remove these excessive heat and keep them in the suitable temperature range. Rajapakshe [30] has simulated the temperature behaviors of different parts of a motor when two kinds of PCM (KNO_3 and NaNO_3) are applied. The results showed that both kinds of PCMs can help reduce the temperature rise in the motor components.

In this study, two kinds of short carbon fibers (SCFs) with different lengths were chosen as the fillers in this work. The diameter is around 9 μm and the nominal aspect ratios of the two kinds of SCFs were 5 and 25. Erythritol was used as the PCM matrix in this study, and its melting point is around 116 $^\circ\text{C}$. The original geometry will not change when embedded into the matrix because of their size and rigidity, which provide a good chance to study the effects of the fillers' geometry on the thermal conductivities of PCCs. A simple setup was made to simulate heat duration of a motor and the temperature-regulated performances of different phase change materials were compared. The results are helpful to maximize the performance of PCCs by choosing the proper geometry and loading of fillers.

2. Materials and experiments

2.1. Materials and preparation

The thermal conductivity of the two kinds of SCFs is 900 W/(m·K) and it is comparable to that of nanofillers. The thermal conductivity of erythritol was measured by ourselves.

The preparation process is similar to that described in [15,16]. Erythritol was melted at 125 $^\circ\text{C}$, then poured into beaker. The beaker was put on the magnetic stirring apparatus (equipped with heat system) for 20 min. Each mixture was then agitated at 350 r/min with a stirrer and the temperature was kept at 125 $^\circ\text{C}$ during the whole process. Next, the melting composites were picked out by a dropper and then poured into a cylindrical mold with diameter of 12.7 mm. Finally, the products were taken out from the molds, dried and polished. Samples with different mass loadings were prepared, 1%, 2%, 4%, 7% and 10%. Aspect ratios of the two kinds of SCFs are different (one is 5 and the other is 25), these two kinds of SCFs are referred as C5 and C25 for short. For convenience, the SCFs/erythritol composites are named in the following way, XCY. X stands for the loading (%) of fillers, Y labels the nominal aspect ratio of SCFs and C means carbon fibers for short. So 1C5 stands for composites with 1% SCFs whose nominal aspect ratio is 5.

2.2. Experiments

2.2.1. Measurements and characterization

The morphologies of the raw materials and composites were obtained using SEM (FEI Quantum 2000). All of samples were coated with gold to improve their imaging. Creating a conductive layer of gold on the sample inhibits charging, reduces thermal damage and improves the secondary electron signal required for topographic examination in the SEM. Melting points and phase change enthalpies were measured by DSC (METTLER TOLEDO TGA/SDTA 851). The testing temperature was from 80 to 170 $^\circ\text{C}$ at 5 $^\circ\text{C}/\text{min}$. The thermal conductivity was measured in an indirect

way in 25 $^\circ\text{C}$. First the thermal diffusivity was obtained using the laser flash method [11] by flash heat conductivity meter (NETZSCH LFA 447). Then the thermal conductivity k was calculated by the following equation:

$$k = a \cdot \rho \cdot c_p \quad (1)$$

where a is the thermal diffusivity, ρ is the density, and c_p is the specific heat.

To determine the crystal structures of these two kinds of SCFs, the degree of graphitization of SCFs was calculated based on the XRD results. The test was performed by Epyrean, Panalytical Company, at a scan speed of 4 $^\circ/\text{min}$ from 20 $^\circ$ to 60 $^\circ$. In order to study the effects of SCFs on the chemical structure of erythritol, FTIR (SPECTRUM ONE B, SHIMADZU) spectrum experiment was conducted, and the testing wavenumber was from 800 to 4000 cm^{-1} .

2.2.2. Testing setup of temperature-regulated performance

The temperature-regulated property of the PCCs were tested. The schematic of the setup was illustrated in Fig. 1. A hollow aluminum column was used to imitate a small motor. The inner and the outer radiuses are 6 mm and 22.5 mm, and it is 50 mm high. There is a heater inside the column, and the heating power is 80 W and the radius is 6 mm. Outsidess, there is graphite sheath with inner radius 27.5 mm and it is 5 mm thin. The interspace between the column and sheath was filled with PCCs and the amount is 39.3 ml. A thermocouple was used to measure the temperature in a spot 2 mm from the inner wall of the column.

The setup was put on a polyfluortetraethylene base. The whole setup was placed in a plastic sheath to eliminate the effect of air convection. The test range is from room temperature to 200 $^\circ\text{C}$.

3. Results and discussion

3.1. Characterization of materials

3.1.1. Morphologies of SCFs and PCCs

The diameters and lengths of SCFs can be determined from the SEM images, as is shown in Fig. 2. The diameters were 9 μm but the lengths were not strictly uniform. The lengths distribution was presented in Fig. 3. The statistic information like the maximum length (l_{max}), and minimum length (l_{min}), and the median length (l_{median}) were calculated based on the distribution. The real aspect ratio p of SCFs were redefined as the ratio of the median length and their diameter. All these are presented in Table 1.

The lengths of both kinds of fibers were distributed in an interval. The maximum length is several times of the median length while the minimum is only a fraction of that. From the results, the lengths distribution of C5 is more concentrated than the C25.

The SEM images of the two kinds of PCCs with various loadings were presented in Fig. 4. SCFs can be easily distinguished from the matrix in both kinds of PCCs. No agglomeration nor entanglement of SCFs were observed in the images. The SCFs distributed quite homogeneously in the matrix as a result of stirring processing during preparation. The grain boundaries of matrix can be detected in the micrographs. It seems the size of grain was related to the SCFs loading. The larger the loading of SCFs is, the smaller the grain is. The composites with higher loadings can transfer heat quickly, so the under cooling inside is larger in the solidification process. This resulted in more formation of crystal nucleus at the beginning and smaller grains at the end of solidification. There are also some pores in the composites. The pores tend to occur in the interface between SCFs and erythritol matrix and intersection of multiple grains. These defects can affect the heat transfer process in PCCs [31].

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