

Research paper

Graphene size-dependent phase change behaviors of in situ reduced graphene oxide/polyurethane-based solid-solid phase change composites

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HIGHLIGHTS

- The crystallization of PEG in system get a lot enhancement.
- The phase change performance of the system is GO size-dependent.
- The interaction among crosslinking network plays an important role.

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ABSTRACT

In this study, the size effects of graphene oxide (GO) sheets on the phase change behaviors of crosslinking polyurethane system have been investigated. The experimental results show that phase change performance of the system is connected closely to the heterogeneous nucleation, physical confinements from GO and interactions in the system, which are inseparable from the specific surface area and the contents of oxygen functional groups depending on the size. This work has a theoretical significance to design thermal energy storage materials with desired properties.

1. Introduction

Phase change materials (PCMs) provide state-of-the-art thermal energy storage through phase transitions, which shows great potential to relieve the energy crisis and improve the energy efficiency as well as reducing the discharge of pollutants for their wide applications in solar energy harvesting, smart textiles, buildings, surplus heat management, etc. [1–7].

Nowadays, polyethylene glycol (PEG) has drawn tremendous interests to researchers due to its incomparable advantages including nontoxic, noncorrosive, good biocompatibility, chemical and thermal stability as well as low-cost and, etc. [8,9]. Nevertheless, the leakage when melting remains an obstacle to the practical applications. Hence, numerous methods have been developed to fabricate solid-solid PCMs (SSPCMs) like microencapsulation, vacuum impregnation, chemical grafting or blocking [10–14]. Undoubtedly, all these supporting matrices containing organic or inorganic shells, porous substrates, polymer skeleton with higher melting points, will affect the phase change behaviors of PEG due to the interaction among the components, which

may have great theoretical significance in practical applications. Regrettably, researches, devoted to the effects of the supporting matrices on the phase change behaviors of PCMs, are much limited to date.

Generally speaking, interest in fabrication polyurethane-based SSPCMs (PUSSPCMs) has become increasingly widespread due to superior merits like no additional containers, no gas or liquid generation and easily being processed, etc. [15,16]. However, the crystallization of PEG parts are easily influenced by the hard segment composed by isocyanate and chain extenders, which may impose chemical bonding and physical interactions to the growth of PEG crystals [17,18]. In like manner, the existence of nano-particles with or without active functional groups is also crucial to the final phase change performance of PUSSPCMs. Pielichowska and his co-workers found that a small amount of graphite nano-platelet could improve the crystallization of PUSSPCMs [19]. Wang et al. fabricated a series of sunlight-driven PUSSPCMs with higher latent heat through incorporation of single-walled carbon nanotubes [20] but lower latent heat with modified graphene [21]. Compared with the most common physical mixed composites, the effects of fillers with active functional groups on the

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final properties of the composites which are obtained one-pot are paid little attention to. In our previous study, nano-filler with abundant oxygen-containing functional groups like halloysite nanotubes and graphene oxide (GO) could participate in the reaction of the system and give rise to the difference of the final properties of the products [22–24]. Interestingly, the products prepared in similarly one-pot but at different reaction temperature exhibit completely different phase change properties, which were mainly attributed to the different surface functionalization of GO [23,24].

For decades, graphene and GO have aroused broader interest for their unique mechanical, electrical as well as optical properties and great promise in vast fields [25,26]. Therefore, graphene and GO are widely used to enhance the mechanical strength and impart excellent electrical or optical properties to the polymer matrices [27]. The properties of composites are largely dictated to the lateral size of graphene and GO sheets [28]. However, there has been blank in investigating the size effect of graphene or GO sheets on the phase change behaviors of PUSSPCMs.

In this work, we studied the size effect of GO on the phase change behaviors of the reduced GO/PUSSPCMs synthesized according our previous work [24]. The final phase transition properties of the system shows great size-dependence for the various specific surface area and contents of oxygen functional groups of GO with different size, which determine the differences in heterogeneous nucleation density and the interactions among system. The system with smaller GO sheets exhibits better phase change properties than that with the larger one. This work suggests that the phase change properties of PUSSPCMs could be tuned by the size of GO sheets.

2. Experimental

2.1. Materials

Graphite flakes (80, 300 and 1200 mesh), potassium permanganate and polyethylene glycol (PEG, Mn = 4000), were purchased from Sinopharm Chemical Reagent Co., Ltd., Beijing, China. Sulfuric acid (H₂SO₄, 98%), hydrochloric acid (HCl, 37%), phosphoric acid (H₃PO₄, ≥ 85%), hydrogen peroxide (H₂O₂, 30%) and *N,N*-Dimethylformamide (DMF), were obtained from Beijing Chemical Works, China. Hexamethylene Diisocyanate Biuret (named HDIB, N75, with NCO % = 15.6%, which was gotten by “di-*n*-butylamine-toluene” method and the structure is given in Fig. S1 in Supporting Information) was purchased from Bayer (Germany). Before use, PEG was dried under vacuum at 110 °C for 3 h and DMF was distilled under vacuum after refluxed and stirred overnight. Other reagents were used as received.

Supplementary data associated with this article can be found, in the online version, at <https://doi.org/10.1016/j.cplett.2018.08.033>.

2.2. Preparation of GO sheets with different size

The GO powder, prepared from graphite powder by a modified Hummer's method [29,30] (the details are given in Supporting Information), was named 80-GO, 300-GO and 1200-GO, respectively, according to the size of precursor. The dried GO powder was dispersed in distilled DMF with the assistance of cell disrupter for use.

2.3. Preparation of reduced GO/PUSSPCMs

Reduced GO/PU-SSPCMs, named RGPCM-X-x (the formulation of the products is listed in Table S1), where X is the size of GO (eg. RGPCM-80 is the product with GO obtained from 80 mesh graphite powder) and x is number of the sample, were synthesized according to our previous work [24]. Typically, HDIB was first dropped into the mixture of GO and PEG solution with the assistance of ultrasound. Then the resultant was poured into Teflon-lined autoclave and maintained at 120 °C for 12 h. After that, the autoclave was naturally cooled to room temperature and the products were dried at 80 °C until no weight change.

2.4. Characterization

The chemical structure of GO and RGPCM-X-x was analyzed using Fourier transform infrared spectrometer (FT-IR, Vertex 70, Bruker, Germany). The size distribution of GO and the microstructure of RGPCM-X-x were visually characterized via XL-30 field emission scanning electron microscopy (SEM) under an acceleration voltage of 10 kV. Additionally, the BET specific surface area was obtained through N₂ adsorption/desorption (Quantachrome Instruments, USA). An Escalab 250 energy spectrometer (Thermo Scientific, USA) was employed to obtain the X-ray photoelectron spectra (XPS) under Al-K α radiation. The X-ray diffraction (XRD) patterns of samples were measured in the range of diffraction angle $2\theta = 5\text{--}45^\circ$ on Bruker D8 Advance (Germany) with Cu-K α radiation ($\lambda = 0.154\text{ nm}$).

The phase change properties were obtained by differential scanning calorimeter (DSC) using TA Q20 instrument (USA) at a heating/cooling rate of 10 °C/min under nitrogen flow. The thermal stability of samples was assessed by means of Thermogravimetric analysis (TGA, Mettler Toledo, Switzerland) at a heating rate of 10 °C/min from room temperature to 800 °C in a nitrogen stream. The light-thermal conversion was carried out using Sun 2000 Solar Simulator (Abet Technologies, USA) with an AM 1.5 filter and an optical power meter (Cel-NP 2000, Cealight, China). The temperature evolution was recorded by a paperless recorder equipped with a thermocouple.

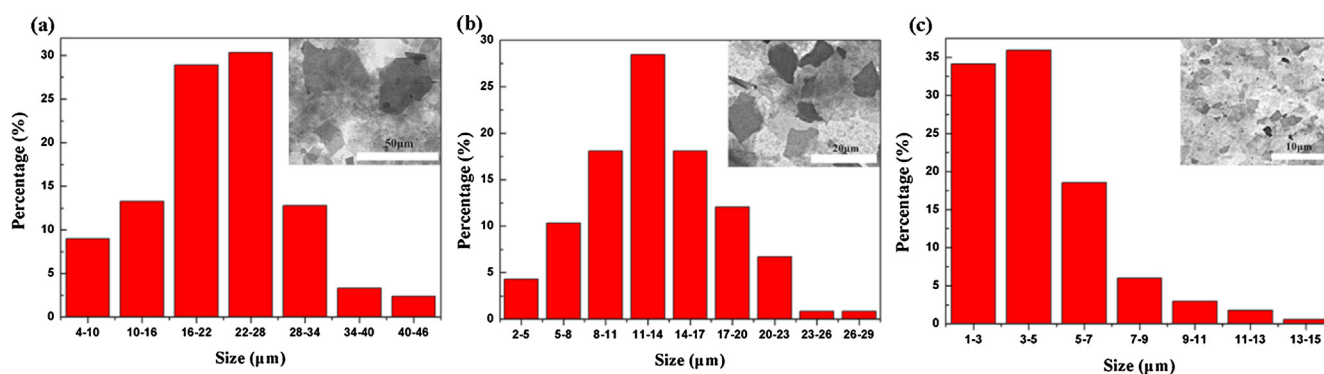


Fig. 1. Size distribution of (a) 80-GO, (b) 300-GO and (c) 1200-GO.

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