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# Solar Energy Materials & Solar Cells

journal homepage: [www.elsevier.com/locate/solmat](http://www.elsevier.com/locate/solmat)

## Investigation on the recovery performance of olefin block copolymer/hexadecane form stable phase change materials with shape memory properties

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### ARTICLE INFO

#### Article history:

Received 19 August 2014  
Received in revised form  
30 September 2014  
Accepted 16 October 2014

#### Keywords:

Phase change material  
Shape memory  
Olefin block copolymer  
Recovery performance

### ABSTRACT

In this work, recovery performance of a novel sort of form stable phase change materials (FSPCMs) with simultaneously shape memory properties was investigated systematically. The FSPCMs comprising hexadecane as latent heat storage material and olefin block copolymer (OBC) as supporting material were prepared by simple swelling preparation method. This kind of difunctional materials exhibits fantastic shape memory properties with mass fraction of hexadecane reaching 70 wt%, which indicates excellent energy storage ability as well. It is found that the recovery performance under 50% strain is obviously better than that under higher strains for all the specimens investigated, while surprisingly, the composite with 70 wt% hexadecane exhibits better recovery performance than pure OBC as well as the composite with 30 wt% hexadecane at strains of 100 and 200%. The structural change brought by incorporation of hexadecane was explored with *in situ* two-dimensional X-ray scattering measurements. According to experiment results, we find that the recovery performance is closely correlated to change of long period, indicating the irreversible deformation of the difunctional materials may originate from lamellar fragmentation of OBC, while addition of massive hexadecane is beneficial to restrain lamellar fragmentation and thus improves the recovery performance. We believe this exploration may shed light on comprehending the relationship between structural change and shape memory performance, thus contributing to adjusting the properties of the difunctional materials and expanding their potential applications.

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### 1. Introduction

Thermal energy storage technology, which can solve the problem in time and spatial mismatch between thermal energy supply and demand, is of vital importance in solar thermal energy applications. Among various energy storage methods, the latent heat storage based on fusion and freezing of phase change materials (PCMs) attracts great attention owing to its high storage density and small temperature variation between storage and retrieval [1–3]. According to the phase change states, PCMs are often divided into three categories: solid-liquid PCMs, solid-solid PCMs and liquid-gas PCMs [4–5]. Compared with other categories, solid-liquid PCMs possess the advantages of high latent heat

density, lower cost as well as various choices, and have been utilized in various applications [6]. However, this kind of PCMs often suffers from the disadvantage of liquid leakage during phase change, which would result in decrease of heat energy storage efficiency and pollution of environment [7]. In order to solve this problem, form-stable phase change materials (FSPCMs) constructed by blending PCMs with polymers many methods have been developed for package [8–12]. In this kind of FSPCMs, the polymer compound is considered as a supporting material and PCMs are dispersed into polymer network. Therefore, FSPCMs can keep solid state even when the ambient temperature is higher than the melting point of PCMs.

For FSPCMs, the selection of supporting materials for specific PCMs is the key consideration for preparation of high-quality products. In order to achieve good package ability, the supporting materials should possess favorable compatibility with corresponding PCMs. Furthermore, by elaborate selection of supporting materials,

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FSPCMs with other fantastic functions can be constructed. Specially, if the supporting materials possess micro phase separation morphology, which is essential for elasticity with regards to thermoplastic polymers, and the melting and crystallization of PCMs can lead to great variation of chain mobility of continuous phase, regulating shape fixing and recovery, the materials may possess shape memory property [13–17]. Shape memory refers to the property that materials can be deformed and subsequently fixed into a temporary shape, which would remain stable unless an appropriate external stimulus triggers the materials to recover permanent shape. Great interests have been attracted to the research of shape memory polymer (SMP) due to its promising potential applications in foams, textiles, morphing structures, deployable structures and biomedical devices [18–23]. As FSPCMs have the ability of controlling temperature to some extent, while shape memory materials with heat as stimulus can be temperature responsive, the difunctional materials prepared in this way are both temperature-controlled and temperature-sensitive, which may show great advantages in some advanced applications, such as intelligent textiles [24]. Through the process of energy storage and retrieval, the textile can regulate temperature and preserve heat to some extent. Meanwhile, with appropriate control of shape memory properties, the textile can be either hard or soft, and it can recover to the original shape with elevated temperature, erasing wrinkles easily.

Optimizing the shape memory properties to achieve excellent recovery performance has always been a major concern for SMP [25,26]. With regards to the difunctional material, controlling shape memory performance can be more essential, which determines whether it can be utilized in specific applications. For example, in order to be used as intelligent wrinkle-resisting texture, the recovery performance should be as good as possible. Pioneering investigations have been conducted to explore the relationship between structure change and recovery performance for SMP. Kim et al. [25] investigated the shape memory effect of various polyurethane ionomers and non-ionomers. They found that the incorporation of ionomer initiated higher rubbery state modulus and endowed the materials with higher recovery strain compared with the non-ionomer. Zhang et al. [26] developed poly(p-dioxanone)–poly(tetramethyleneoxide) glycol (PPDO–PT-MEG) multi-block copolymers and investigated the correlation between shape memory performance and chain structure. It is found that better crystallization ability of PPDO segments would result in increased recovery performance. These works lead to intensive comprehensions of corresponding recovery performance. However, as for the difunctional materials, the situations can be more complicated. With massive PCMs dispersing into the supporting materials, change of condensed state may occur in the polymeric matrix, leading to variation of recovery behaviors. Considering the complex influence factors, it can be quite challenging to regulate the shape memory properties. As far as we know, no researches have been concentrated on comprehending and controlling the recovery performance for this kind of difunctional materials (blends of polymer with small molecules).

Recently, we designed a fantastic sort of polyolefin-based FSPCMs combined with shape memory property, in which olefin block copolymer (OBC), a multi-block thermoplastic elastomer, acted as supporting material, while paraffin was utilized as both PCMs and switch for shape changing [24]. Excellent shape memory properties were achieved while the maximum content of paraffin reached 40 wt%. However, we found that a certain amount of irreversible deformation always occurred in the first circle during shape memory testing, which was undesired for SMP. In order to understand the origin of irreversible deformation and explore the influence of small molecular PCMs on the recovery behaviors, in the present work, the recovery performance of the difunctional materials comprising OBC and hexadecane, which is

similar linear alkane as paraffin, is investigated systematically. *In situ* two-dimensional (2D) wide and small-angle X-ray scattering (WAXS and SAXS) measurements are carried out during deformation process to detect the structural change. Based on our results, it is found that the different recovery performances are closely correlated to the change of crystalline phase of OBC, which is strongly influenced by addition of hexadecane. This investigation may promote the understanding of the relationship between structural change and shape memory performance, and thus contributes to adjusting the properties of these difunctional materials for practical applications.

## 2. Experimental

### 2.1. Materials and sample preparation

OBC material synthesized by chain-shuttling technology is a commercial grade product produced by Dow Chemical Company; it has a density of 0.887 g/cm<sup>3</sup> and a melt flow rate of 5.0 g/10 min (230 °C, 2.160 kg), the hard block content is approximately 49 wt% while the octane content is 1.0 mol% in hard blocks and 19.3 mol% in soft blocks. The hexadecane with a melting temperature of approximately 21 °C is purchased from Aladdin Chemical Reagent Company (Shanghai, China). To simplify the preparation process and elevate the content of PCMs in the composites, swelling method was chosen for preparation. OBC granules were placed on culture dish with a flat base, and hexadecane liquid was dropped uniformly on the granules. Samples were held at 60 °C for 24 h to ensure adequate swelling of hexadecane into OBC. In order for further interdiffusion and molding for subsequent characterization, all the series of OBC samples were compression molded to films, which was folded and undergone another compression process at 160 °C. The feeding composition of OBC was 70 and 30 wt%, respectively, and the corresponding samples were denoted as OBC-70, OBC-30. Neat OBC was denoted as OBC-100.

### 2.2. Characterizations

A Mettler DSC-821e apparatus (Mettler Toledo, Switzerland) was used to evaluate the thermal properties of series of OBC samples and hexadecane. The heating and cooling rate was 10 °C/min. In order to get the melting latent heat ( $\Delta H_m$ ), five species from different parts of each sample were tested, and the average value as well as deviation was calculated on basis of the results. Large-strain tensile testing was conducted on a SANS CMT-6503 universal testing machine (Shenzhen, China) with a crosshead speed of 50 mm/min. The capacity of the load cell is 50 N in axial load. Crystallization morphologies of series of OBC samples were observed using DM2500P polarized optical microscope (POM) (Leica, Germany) with Linkam-THMS600 hot stage. Each sample was heated to 180 °C at 20 °C/min and kept at this temperature for 5 min to allow complete melting, and then it was subsequently cooled to 60 °C at 20 °C/min for observation.

Quantitative shape memory testing was carried out by the following procedure: the rectangular sample with a length of 20 mm was immersed into water-bath under a controlled constant temperature of 60 °C and stretched by a strain of 100% ( $\epsilon_m$ ). Then the sample was cooled to 0 °C while holding the stress constant for 5 min. The stress was quickly released when the sample was in the stretched state and the fixed strain ( $\epsilon_u$ ) was recorded. The unconstrained strain recovery was triggered by immersing the sample again into a water bath of 60 °C and the resultant strain was obtained ( $\epsilon_p$ ). In order to cooperate with *in situ* WAXS and SAXS measurements and comprehend the recovery performance, further recovery testing was performed. Samples were stretched

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