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

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\section*{A B S T R A C T}

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 \textit{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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\section*{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 \textsuperscript{[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,
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(tetramethyletheneoxide)] 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/cm³ 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 melting 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 (∆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% (ε_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 (ε_p) 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 (ε_r). In order to cooperate with in situ WAXS and SAXS measurements and comprehend the recovery performance, further recovery testing was performed. Samples were stretched...
with a rate of 4.23 mm/min at room temperature of 25 °C, which is still higher than the melting point of hexadecane, and the corresponding recovery strain was recorded. The fixing ratio ($R_f$) and recovery ratio ($R_r$) were calculated according to the following equations, where $N$ corresponds to the cycle number.

$$R_f(N) = \frac{\varepsilon_{fi}(N)}{\varepsilon_{fi}(N)} \times 100\%$$

$$R_r(N) = \frac{\varepsilon_{re}(N) - \varepsilon_{fi}(N)}{\varepsilon_{fi}(N)} \times 100\%$$

In situ WAXS and SAXS measurements were used to monitor the evolution of structure and morphology during stretching process. 2D WAXS and SAXS devices are made using an in-house the evolution of structure and morphology during stretching.

3. Results and discussion

3.1. Thermal property and mechanical characterization

The cooling and heating thermograms are shown in Fig. 1. The exothermic peaks located above 90 °C can be attributed to the crystallization of OBC. The peak temperature keeps nearly invariant with addition of 30% hexadecane and only decreases slightly when hexadecane becomes the main component. The exothermic peaks below 15 °C result from the crystallization of hexadecane. The average melting and crystallization parameters of all the samples including the melting peak temperature ($T_m$), crystallization peak temperature ($T_c$), and the latent heat ($\Delta H_m$) were summarized in Table 1. The results exhibit that the endothermic peaks and exothermic peaks of OBCs/hexadecane composites both shift to lower temperatures with increase of OBC content, and the melting enthalpy of hexadecane in composites is lower than the theoretical calculation for OBC-70 and OBC-30 to different degrees. The differences between neat hexadecane and OBC/hexadecane samples may be attributed to that the confinement of three-dimensional network structure formed by OBC, the effect of which is closely correlated with OBC content [27]. With regards to the preparation of OBC/hexadecane composites, swelling method was chosen to simplify the process and elevate the content of PCMs [28]. The results exhibit that 70 wt% hexadecane can be encapsulated in FSPCMs with this method, which is obviously higher than our previous work and indicates much better energy storage ability. Meanwhile, this sample with high hexadecane content also maintains good form-stable effect during phase change, as shown in Fig. S1. In order to verify uniformity of the prepared samples, five species from different parts of each sample were tested. As shown in Table 1, the deviation is no more than 8% for all the samples, which indicates that the prepared OBC/hexadecane FSPCMs are uniform and it is credible to perform further experiments.

The large-strain tensile testing results are illustrated in Fig. 2. OBC-100 displays a typical elastomeric characteristic with no distinctive yield point. The elongation at break exceeds 1500%, demonstrating excellent tenacity and deformation ability. When incorporated with hexadecane, the mechanical properties of the samples changes dramatically. The tensile stress of OBC-30 and OBC-70 decrease significantly compared to OBC-100. As small molecule hexadecane keeps in molten state during tensile tests, its chain mobility is highly active. Therefore, the component of hexadecane can reduce the chain entanglement and lead to drop of tensile stress. As for the elongation at break, we notice that there is no obvious distinction between OBC-100 and OBC-70. However, when the mass fraction of hexadecane increases to 70%, the elongation at break decreases below 900%. This may be attributed to that the three-dimension network formed by OBC almost retains with 30% content of hexadecane, while the incorporation of 70% hexadecane changes the crystallization morphology dramatically and destroys the network to some extent, which is demonstrated by POM observation. Despite different tensile properties, all the three samples exhibit typical elastomeric

\[
d q = 4\pi \sin \theta / \lambda
\]

where $q$ is the module of the scattering vector, $\lambda$ the wavelength of X-ray and $2\theta$ the scattering angle.

<table>
<thead>
<tr>
<th>Sample</th>
<th>OBC content (wt%)</th>
<th>$T_m$ (°C)</th>
<th>$T_c$ (°C)</th>
<th>$\Delta H_m$ (J/g)</th>
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</thead>
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<tr>
<td>hexadecane</td>
<td>0</td>
<td>21.0</td>
<td>13.6</td>
<td>226.5 ± 4.5</td>
</tr>
<tr>
<td>OBC-30</td>
<td>30</td>
<td>16.6</td>
<td>4.8</td>
<td>148.0 ± 7.3</td>
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<tr>
<td>OBC-70</td>
<td>70</td>
<td>9.8</td>
<td>4.2</td>
<td>45.0 ± 3.6</td>
</tr>
</tbody>
</table>

Table 1

Thermal property of hexadecane and OBC/hexadecane FSPCMs.

![Fig. 1. DSC (a) cooling curves and (b) heating curves of hexadecane, OBC and OBC/hexadecane composites.](image-url)
behaviors with large elongation at break, which is an important character in practical applications.

3.2. Polarized optical microscopy (POM) observation

As OBC is crystalline material which exhibits strong birefringence under polarized light while hexadecane keeps in molten state during our experiment, POM can be used to observe the crystallization morphologies of OBC and detect the structural change brought by addition of hexadecane. Fig. 3 shows the pictures of OBC-100, OBC-70 and OBC-30. As can be seen from the pictures, the spherulites of OBC disperse uniformly as island phase, while the amorphous blocks assemble into continuous phase between spherulites. The phase separation morphologies insure good elasticity of OBC/hexadecane composites. The incorporation of 30% content of hexadecane does not alter the integral morphologies dramatically, while the spherulites become smaller and less regular. However, with regards to OBC-30, the main component of which becomes hexadecane, the overall morphologies turn distinct from the others. The spherulites are isolated with each other, and they become much smaller and quite irregular. The phenomenon indicates that massive encapsulated hexadecane assembles together and space out the crystals of OBC, which may destroy the network formed by OBC to a certain degree. Interestingly, ring-banded spherulites are found in series of OBC samples during nonisothermal crystallization. The phenomenon may explicit some results of our further experiments, while the forming mechanism of ring-banded spherulites will be investigated intensively in our later work.

3.3. Shape memory and extensive recovery performance characterization

A typical visual shape memory process is illustrated in Fig. S2, showing obvious shape memory effect. Quantitive evaluation of shape memory behaviors has been performed according to previous literatures [16,29]. Samples were stretched to a strain of 100% under external stress after which fixing and recovery ratio was calculated. As exhibited in Table 2, OBC-100 possesses a bad shape memory fixing performance that only 40% elongation is preserved after the release of stress. Apparently, this can be attributed to lack of switch which responses to the decrease of temperature. After incorporating hexadecane as switch phases, OBC-70 composite exhibits a greatly improved fixing performance. With elevated hexadecane concentration, nearly complete fixing performance is achieved for OBC-30 and it keeps fairly stable. With regards to the recovery performance, we notice that a

<table>
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<th>Sample</th>
<th>Cycle number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>OBC-100</td>
<td>Rf (%)</td>
<td>44</td>
<td>39</td>
<td>38</td>
<td>38</td>
<td>39</td>
</tr>
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<td>99</td>
<td>99</td>
<td>99</td>
<td>99</td>
</tr>
<tr>
<td>OBC-70</td>
<td>Rf (%)</td>
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<td>95</td>
<td>94</td>
<td>96</td>
<td>96</td>
</tr>
<tr>
<td></td>
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<td>93</td>
<td>99</td>
<td>98</td>
<td>99</td>
<td>98</td>
</tr>
<tr>
<td>OBC-30</td>
<td>Rf (%)</td>
<td>99</td>
<td>99</td>
<td>99</td>
<td>99</td>
<td>99</td>
</tr>
<tr>
<td></td>
<td>Rr (%)</td>
<td>97</td>
<td>99</td>
<td>99</td>
<td>98</td>
<td>99</td>
</tr>
</tbody>
</table>

<table>
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<tr>
<th>Fig. 2. Stress-strain curves of three OBC samples.</th>
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<tbody>
<tr>
<td>Fig. 3. Polarized optical microscope pictures of (a) OBC-100, (b) OBC-70, (c) OBC-30 and (d) magnification of local region in (b).</td>
</tr>
</tbody>
</table>
considerable change occurs between the first cycle and the second cycle. During the first cycle, a certain amount of irreversible deformation arises for all the specimens, while nearly complete recovery has been achieved during subsequent cycles. Interestingly, we find that OBC-30, which possesses the best energy storage ability, exhibits obviously better recovery performance during the first circle. This is not consistent with prediction in respect that hexadecane in OBC-30 congregates together and may destroy the network of OBC to some extent as indicated by tensile tests as well as POM observation. As controlling the recovery performance is of vital importance for the difunctional materials, extensive recovery testing was performed to explore the origin of irreversible deformation and the influence of small molecular PCMs on the recovery behaviors.

We first compared the recovery performance of OBC/hexadecane FSPCMs under different applied strain. As exhibited in Fig. 4, all the samples show nearly complete recovery under 50% strain. However, when the strain is elevated to 100%, the specimens exhibit a certain amount of irreversible deformation, and the recovery ratio almost keeps constant with strain further increased to 200%. This indicates that a significant structural change may occur with strain raised from 50 to 100%. Similarly, OBC-30 exhibits obviously better recovery performance than OBC-100 and OBC-70 under strain of 100 and 200%.

In situ 2D WAXS and SAXS measurements were carried out to detect the change of crystals during stretching. Selected 2D WAXS and SAXS patterns of OBC/hexadecane samples during stretching are shown in Fig. 5. In general, the 2D WAXS pattern of OBC shows two rings from the (110) and (200) reflections of the orthorhombic crystal form superimposed with an amorphous halo [30–32]. All the samples are isotropic without strain. As for OBC–100, both the (110) and (200) reflections are still nearly isotropic with strain of 50%, indicating little orientation in crystalline phase. When the strain is elevated to 100%, the (110) reflection turns to pattern of arcs with meridional preferential orientation, which arises from rotation of the lamellae into the draw direction. With regards to samples in which hexadecane has been added, the circumstances become quite different. Both OBC–70 and OBC–30 exhibit obvious orientation under 50% strain. This may be attributed to that the incorporation of hexadecane increases chain mobility, making lamellae easier to rotate to stretching direction. Considering that OBC–70 and OBC–30 exhibit almost complete recovery under 50% strain, rotation of lamellae which results in orientation may not be the intrinsical origin of irreversible deformation. Interestingly, we find that the preferential orientations of OBC–70 and OBC–30 are opposite from OBC–100 for WAXS, with arc appearing along the equator, while they keep the same for SAXS. As the orientation originates from rotation of lamellae for WAXS, we conjecture that the difference of preferential orientations may be related to formation of ring-banded spherulites, which results from lamellar twisting [33–36]. However, the concrete reason for reversal of preferential orientations still needs to be investigated. In consideration that the orientation is not correlated directly with recovery performance, and it may be improper to compare orientation parameters among the three specimens, we mainly focused on SAXS data in our subsequent discussion.

As exhibited in Fig. 5, the orientation of SAXS, which is derived from stack of lamellae, keeps in the same direction for all the specimens. The selected integrated SAXS profiles are shown in Fig. 6. The q value at the scattering peak is used to determine the long period between the adjacent lamellar layers. As for OBC–100, q value at the scattering peak keeps almost invariant when the strain increases from 0 to 50%. However, with strain elevated to 100%, q shifts to higher value, indicating a decreasing long period along the equator direction. We also notice that the crystallinity nearly keeps constant during the stretching (Fig. S3 and Table S1), suggesting that the effect of strain-induced crystallization is rather weak and the decrease of long period may not be ascribed to newly formed crystals under this circumstance. On account of our experiment results and existing conclusions of investigation on changing of OBC lamellae during stretching [30,32], we conjecture that the decreasing long period implies fragmentation of the lamellar structure, which results in some smaller but larger population of lamellar crystals in OBC. Compared to OBC–100, the change of q between strain of 50% and 100% for OBC–30 is significantly smaller. The calculated long periods of series of OBC samples along the equator direction are illustrated in Fig. 7. It is found that the long periods nearly keep invariant under 50% strain.

![Fig. 4. Recovery ratio of OBC samples as a function of applied strain.](image)

![Fig. 5. Selected 2D WAXS (upper) and SAXS (lower) patterns of OBC samples under (a) 50% strain and (b) 100% strain. The stretching direction is horizontal.](image)
for all the specimens. While when the strain increases to 100 or 200%, the long periods reduce to varying degrees. The results indicate that fragmentation of lamellae under 50% strain is rather insignificant compared to under 100 or 200% strain. Considering the different recovery performance under corresponding strain, the irreversible deformation may be closely related to the fragmentation of lamellae. We also notice that although the long periods exhibit similar changing tendency, the decreasing value for OBC-30 is much smaller than OBC-70 and OBC-100, which corresponds to the recovery performance and further demonstrates the correlation between fragmentation of lamellae and inferior recovery performance. The obvious elevated recovery performance of OBC-30 can be attributed to the incorporation of massive hexadecane. It is shown in Fig. 7 that the long period of OBC-30 without strain exceeds 40 nm, which is obviously larger than OBC-100 and OBC-70. This result indicates that the lamellae of OBC-30 become more isolated and force on lamellae under certain strain will be smaller correspondingly, resulting in lower extent of lamellar fragmentation.

The specimens were also subjected to cyclic loading for further investigation on recovery performances. As exhibited in Fig. 8, a similar recovery tendency is obtained as previous characterization. The obviously distinct phenomena among different cycles suggest that a permanent structural change only occurs during the first cycle, which leads to decrease of recovery ratio. The equatorial long periods for all the specimens calculated from SAXS data are exhibited in Fig. 9. It is found that the long period decreases after first cycle, and it maintains almost invariant during subsequent cycles. The results indicate that the fragmentation of lamellae is irreversible, and the fragmentized small lamellae can’t assemble into large lamellae with loading removed. When exposed to same strain again, the “conditioned” lamellae do not further fragmentize. Therefore, fragmentation of lamellae occurs during the first cycle, and it may be closely related to the drop of recovery performance.

### 3.4. Schematic model for the structural change during deformation

As reported in literature, the soft and hard blocks are different in chain architecture and crystallizability for OBC. Upon cooling the hard blocks can crystallize into lamellar structures, while the nearby soft blocks have to be pushed away from the crystalline region into the amorphous region. Thus, tiny crystalline and amorphous local domains with distinct crystallinity can be observed. During stretching, the amorphous region experiences much higher strain ascribed to its lower strength. However, as samples were held at 60 °C before measuring the recovery ratio, the permanent set resulting from chain
entanglement of amorphous region has been eliminated. Therefore, the irreversible deformation should originate from structural change of crystalline region. According to our results, schematic model for the structural changes in crystalline region during uniaxial stretching is illustrated in Fig. 10.

For pure OBC, it exhibits little change of orientation in WAXS patterns as well as calculated long period under 50% strain. The results suggest that the force applied to crystalline region is not enough to induce intense fragmentation of lamellae under this circumstance. Thus nearly complete recovery achieves as exhibited by our tensile and recovery testing results. When the strain increases to 100%, an obvious decrease of long period occurs, which indicates lamellar fragmentation takes place. As the fragmentation is an irreversible structural change, specimens cannot fully recover to its original state after unloading, and recovery ratio drops to some extent. Compared to OBC-100, OBC-30 is easier to form obvious preferential orientation as shown in Fig. 5. This is attributed to that incorporation of hexadecane increases chain mobility, making lamellae easier to rotate to stretching direction. Considering the nearly complete recovery of OBC-30 under 50% strain, we conclude that the orientation is not directly correlated to irreversible deformation. It is found that the recovery performance of OBC-30 is obviously better than OBC-100 and OBC-70 when the strain is elevated to 100 and 200%. Considering the larger long period of OBC-30 compared with other samples, the improved recovery performance can be ascribed to that the crystals of OBC-30 are comparatively isolated with massive hexadecane, which results in much smaller force on lamellae under certain strain and restrains lamellar fragmentation effectively. In summary, the changing trend of long period matches well with the decrease of recovery performance for all the samples, demonstrating the irreversible deformation for OBC/hexadecane difunctional materials originates from

![Fig. 9. Equatorial long periods of OBC samples under five continuous cyclic loading.](image)

![Fig. 10. Schematics of the crystalline region in OBC-100 and OBC-30 during deformation.](image)
fragmentation of OBC lamellae. Meanwhile, addition of massive small molecular hexadecane can restrain lamellar fragmentation effectively and thus improves the recovery performance.

4. Conclusions

In this work, the recovery performance of OBC/hexadecane difunctional materials was investigated systematically, and the relationship between recovery performance and structural change of supporting materials brought by incorporation of PCMs was explored. Combining the results of recovery tests and long period of supporting materials brought by incorporation of PCMs was relationship between recovery performance and structural change of OBC-30, which possesses better energy storage ability, shows higher recovery ratio than OBC-70 as well as OBC-100 under strain of 100 and 200%. It is found in SAXS tests that the long period of OBC-30 without strain exceeds 40 nm, which is obviously larger than OBC-100 and OBC-70. This result indicates that the lamellae of OBC-30 are more isolated with massive hexadecane and force on lamellae under certain strain will become smaller correspondingly, restraining lamellar fragmentation effectively. We believe the results may shed light on comprehending the relationship between structural change and shape memory performance for this difunctional material. With a deep understanding of the intrinsic relationship, the recovery performance may be controlled to some extent, providing the possibility for extended applications.

Acknowledgments

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.solmat.2014.10.023.

References