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# A DSC study of the order–disorder transition of SEPS block copolymers in the block copolymer/paraffin oil/layered silicate nanocomposite gels

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## Abstract

The effect of addition of layered silicate on the order–disorder transition of poly[styrene-b-(ethylene-propylene)-b-styrene] (SEPS) block copolymer in the block copolymer/paraffin oil/layered silicate nanocomposite gels was investigated by means of differential scanning calorimetry (DSC) and modulated DSC (MDSC). The addition of layered silicate has significant influences on the order–disorder transition and the phase structure of the block copolymer. With increasing layered silicate, the order–disorder transition temperature of the block copolymer decreased. The addition of layered silicate reduced the ordering level of the block copolymer. MDSC results revealed that the order–disorder transition is a glass transition-like process. A thermal event related to the ordering process, which took place in the preparation of the sample and hid in the specimen, has been revealed by MDSC first time.

*Keywords:* Block copolymer; Order–disorder transition; Nanocomposite gel; DSC

## 1. Introduction

Block copolymers represent an interesting class of soft polymeric materials, which are produced by covalently linking two or more contiguous linear sequences (blocks) of chemically dissimilar species [1–3]. A feature of block copolymers is that they can form a variety of self-assembled nano-scale ordered structures [3–5], which depend primarily on molecular composition, thermodynamic incompatibility of blocks, and monomer asymmetry [6]. Block copolymers have a number of unique properties resulting from their morphological features. Because of these properties block copolymers have been used in a wide range of material fields [1–4], for example, thermoplastic elastomers for impact modification, compatibilization and pressure-sensitive adhesion. Upon thermodynamic variation such as temperature, pressure and solvent concentration these microstructures can undergo thermal transformations from one ordered structure to another (order–order transition) one and from an ordered to disordered (isotropic) state (order–disorder transition) [7]. These effects occur because the systems under consideration have a small characteristic length scale owing to a connectivity of the block chains in block copolymers [1–3]. With the aim of understanding under which conditions affecting the block connectivity, the phase transition of block copolymers have been extensively studied both theoretically and experimentally over last 2 decades [1–4]. The factors that control micro-phase behaviour and govern the formation of micro-domain size are well clearly understood.

Recently, the development of new kinds of functional materials such as nano-scale templates [8], nano-structured damping and porous materials [9,10], and nanocomposites based on block copolymers has been widely investigated [11–14]. Among all the potential nanocomposite precursors, those based on layered silicate reinforcements have received considerable attention over the last few years because a relatively low level of clay can lead to significant enhancements in mechanical, thermal and barrier properties [11,12]. One has started to pay attention on the development of block copolymer–clay nanocomposites [13,14]. However, it is not clear whether such a nanofiller affects the microstructure and the order–disorder transition of the block copolymer in the nanocomposites. Although the order–disorder transition of block copolymers has been widely investigated [3,15], it is believed that the order–disorder transition in block copolymers is melting-like one revealed by DSC.

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Recent research showed that the understanding is questionable [16].

In this communication, the effect of addition of layered silicate on the order–disorder transition and phase structure of poly[styrene-b-(ethylene-propylene)-b-styrene] (SEPS) in the block copolymer/paraffin oil gels was investigated. The order–disorder transition behaviour of block copolymers was re-examined. The aim of the use of paraffin oil is to lower the order–disorder transition temperature which makes the observation of the order–disorder transition more easy by means of DSC.

## 2. Experimental

### 2.1. Materials

Poly[styrene-b-(ethylene-propylene)-b-styrene] with 30% of styrene was provided kindly by Kuraray Co. Ltd., Japan. The molecular weight of SEPS is $M_w = 76{,}500$ and $M_w/M_n = 1.3$ determined by using gel permeation chromatography. Paraffin oil was kindly provided by Idemitsu Kosan Ltd., Japan, and its molecular weight is 770. Layered silicate (clay) used in our experiments was Cloisite® 20A (C20) and $Na^+$-clay, purchased from Southern Clay Products (TX, USA). The modifier for organoclay C20 is dimethyl, dehydrogenered tallow ammonium.

### 2.2. Sample preparation

The blends of SEPS and C20, and of SEPS and Na+-clay with different weight fraction (0, 0.5, 1, 3 and 5%) were prepared by a solution method, respectively. Toluene was used as a solvent. The ratio of polymer component to toluene was about 10 wt.%. The solutions of SEPS/clay/toluene were stirred (400 rpm) at room temperature for 24 h. Mixtures of SEPS/clay/toluene and paraffin oil with different contents (80%, 70%, 60% and 50%) were prepared. The mixtures were stirred at room temperature for 36 h. After that, the mixture was cast on to a PET film, and the solvent was gradually evaporated for 2 days at room temperature. The films obtained were finally dried in vacuum over at 80 °C for at least 3 days.

### 2.3. Wide angle X-ray diffraction (WAXD)

X-ray diffraction experiments were performed on film samples on a Bruker X-ray diffractometer (AXS D8 Advance) using Kα ($\lambda = 0.154$ nm) radiation. Samples were scanned at a rate $0.24^\circ\,\text{min}^{-1}$ from about 1° to 20° in $2\theta$.

### 2.4. Differential scanning calorimetry and modulated differential scanning calorimetry

Differential scanning calorimetry (DSC) and modulated DSC (MDSC) were used in this research for the measurement of the order–disorder transition temperature. A TA Instrument DSC 2920 calorimeter was employed. All measurements were run in nitrogen atmosphere and temperature ramp was set at 10 °C/min for DSC measurements. For MDSC measurements, oscillation amplitude of ±1.0 °C, oscillation period of 60 s and a heating rate of 3 °C/min were used. The calorimeter was calibrated with a standard method [see the operator's manual of TA DSC 2920 Differential Scanning Calorimeter] including baseline, temperature and cell constant calibrations.

### 2.5. Small angle X-ray scattering

The phase structure of SEPS/oil and SEPS/oil/clay nanocomposite gels were determined by means of small angle X-ray scattering (SAXS) using a the Kratky Compact Small Angle System with a stationary-anode copper-target X-ray tube (wavelength 0.1542 nm) at room temperature. The fine-focus X-ray generator was operated at 45 kV and 40 mA. The measured intensity was desmeared and corrected for background scattering and photoelectric absorption in the samples.

## 3. Results and discussions

### 3.1. Effect of the addition of layered silicate on the order–disorder transition of SPES block copolymer in SEPS/paraffin oil/clay nanocomposite gels

Normally, WAXD is used to identify intercalated structure [17]. The intercalation of the polymer chains usually increase the interlayer spacing, in comparison with the spacing of the clay used, leading to a shift of the diffraction peak towards the lower angle values.

Fig. 1A and B shows WAXD patterns for pure clay and SEPS nanocomposite gels (5% clay content) for the two kinds of clays, $Na^+$-clay and C20, respectively. The obvious shift of peak position to lower value of the nanocomposite (for Na+-clay system, from 7.4° to 5.5°; for C20 system, from 3.8° to 2.3°) suggests that an intercalated nanocomposite gels was obtained. The two peaks shown in Fig. 1B are first and second order ones. Beside, it can be seen that the change of the peak positions for the $Na^+$-clay nanocomposite was smaller than that of C20 nanocomposite showing that the compatibility of C20 with SEPS is higher than that of $Na^+$-clay with SEPS.

Thermal measurements [18] are able to identify a heat of fusion at the order–disorder transition temperature ($T_{ODT}$). Fig. 2 shows the plots of the heat flow versus temperature for SEPS/oil gels. The results indicate that the order–disorder transition is likely to be a melting transition of semi-crystalline polymers. It is obvious that $T_{ODT}$ depends on the polymer/oil concentration. The higher the concentration of the block copolymer, the higher the $T_{ODT}$ was. The reason for this is that the addition of paraffinic oil in the gel swells the block copolymer and increases free volume, which result in the decrease of the $T_{ODT}$. The effects of composition of paraffinic oil on $T_{ODT}$ in SEPS/oil gels have been intensively investigated in our group [19] and will not be discussed here. However, it must indicate that these transition peaks became weak in second heating runs.

Fig. 3 shows heat flow versus temperature for SEPS/oil/$Na^+$-clay (0.5 wt.%) nanocomposite gels. In comparison between

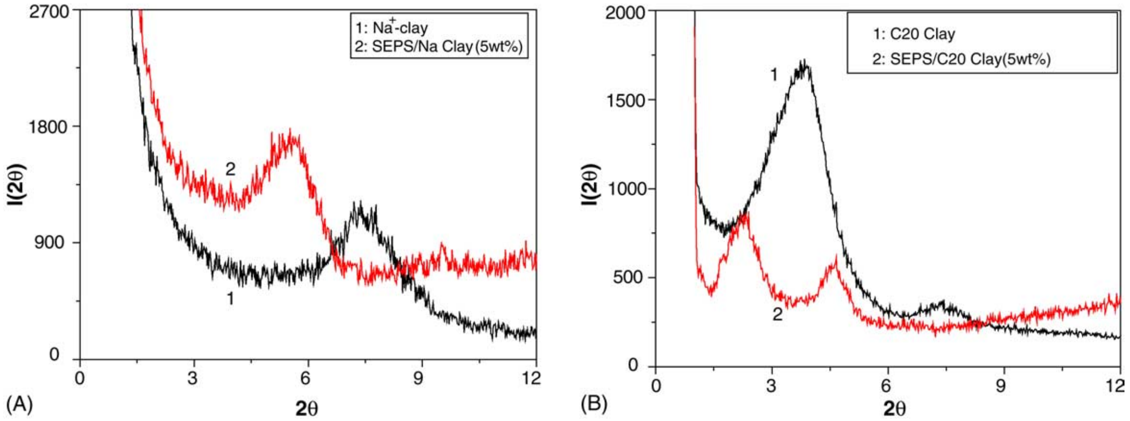

Fig. 1. X-ray diffraction patterns for pure clay and SEPS/clay/paraffin oil nanocomposite gels containing 5% clay: (A) Na$^+$-clay and (B) C20.

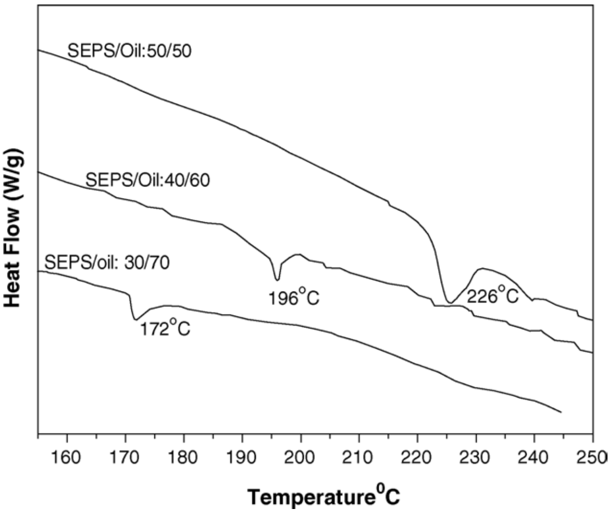

Fig. 2. DSC thermograms of SEPS/oil gels.

Figs. 2 and 3 it can be seen that addition of only 0.5% clay resulted in a decrease of 1° at $T_{ODT}$.

Table 1 lists $T_{ODT}$ for a series of block copolymer nanocomposite gels with different contents of Na$^+$-clay. It can be seen that $T_{ODT}$ is affected by the addition of the layered silicate. With increasing layered silicate, the $T_{ODT}$ decreases and it is lowered by 4 °C when 5 wt.% of layered silicate was introduced into the gel.

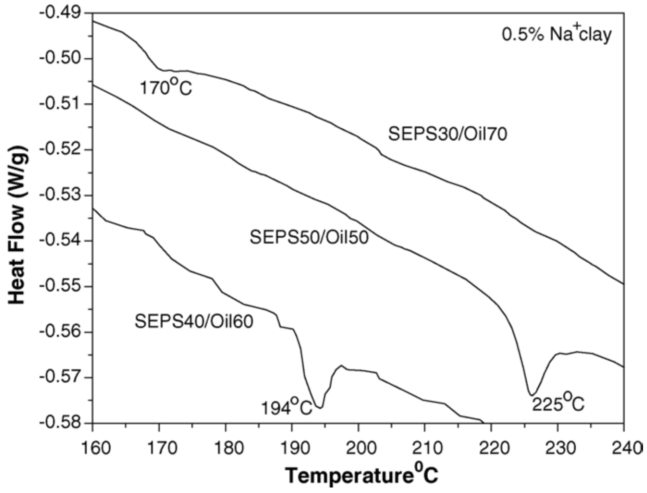

Fig. 3. DSC thermograms of SEPS/oil/Na$^+$-clay (0.5 wt.%) nanocomposite gels.

In order to compare the enthalpy change in the order–disorder transition for different nanocomposite gels, a normalised enthalpy function, $\phi(c)$, was introduced as ratio of $\Delta H_i/\Delta H_0$, $\Delta H_0$ is the enthalpy of the pure block copolymer, $\Delta H_i$ the enthalpy of the nanocomposite gel at the order–disorder transition and $c$ is the concentration of the layered silicate. The relationship between the normalised enthalpy function and layered silicate concentration is shown in Fig. 4. With increasing the clay content, the enthalpy function decreased. It can also be seen there is influence of the oil content on the normalised enthalpy function at the present of the layered silicate. The effect in the 70% oil system is the biggest. This indicates that there may be cooperation between oil molecules and the layered silicate.

As known, the thermal event of the order–disorder transition in block copolymers is weak one. Sometimes the small thermal event cannot be detected accurately by DSC measurement [20,21]. In order to further confirm the order–disorder transition behaviour, the micro-phase-separated structure of the block copolymer/paraffin oil/organoclay nanocomposite gels was examined by SAXS.

Typical SAXS patterns for SEPS/oil and SEPS/oil/clay systems are shown in Fig. 5.

The scattered intensity is proportional to the mean square of the electron density difference in a system. If the electron density is the same in block copolymer, which means in entire disordered

Table 1
The order–disorder transition temperature for SEPS/oil/Na$^+$-clay nanocomposite gels

| Na$^+$-clay content (wt.%) | $T_{ODT}$ (°C) (SEPS30/oil70) | $T_{ODT}$ (°C) (SEPS40/oil60) | $T_{ODT}$ (°C) (SEPS50/oil50) |
|---|---|---|---|
| 0 | 172 | 196 | 226 |
| 0.5 | 170 | 194 | 225 |
| 1 | 170 | 193 | 224 |
| 3 | 169 | 193 | 223 |
| 5 | 169 | 192 | 222 |

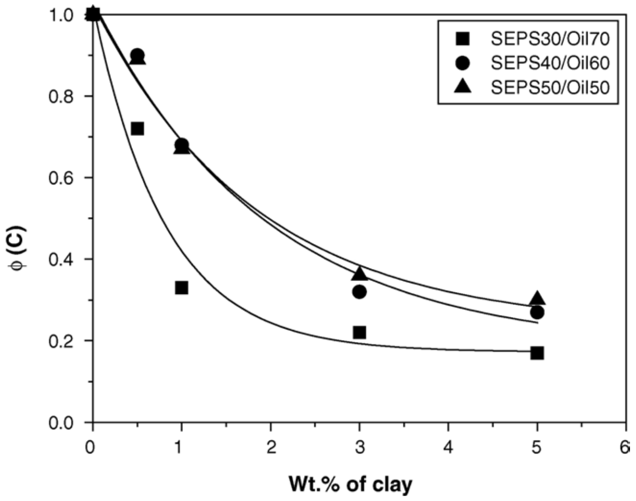

Fig. 4. Normalised enthalpy functions of SEPS/oil/Na$^+$-clay nanocomposite gels with different oil and Na$^+$-clay contents.

state, the scattered intensity only represents for background scattering. For a micro-phase-separated system, in which there is a difference in the electron densities, the higher scattering intensity with scattered peak is detected. The measured intensity of the peak is proportional to the fraction of ordered material in the sample.

As seen in the figure, the observed highest peak satisfies the characteristics of ordered structure for the SEPS/oil gel. However, when the layered silicate was introduced into the system, the scattered peak become broader and the intensity decreased. The reduction of the scattered intensity indicates that the degree of ordered structure of the block copolymer decreased resulting from the micro-phase diffusion between the blocks [22]. The broadening of the scattered peak illustrates that the addition of layered silicate reduced the repulsion forces between block-A and block-B segments [22].

A block copolymer is, at least, two incompatible blocks bonded covalently together, and that cannot macro-phase separate. The unfavourable enthalpy interactions between the different monomers may lead to a local segregation on the length-scale of the copolymer chains. Therefore, their equilibrium phase morphology is controlled by the degree of incompatible $\chi N$ ($\chi$ is the Flory–Huggins A–B segment–segment interaction parameter and $N$ is the total number of segments), volume fraction and the macro-molecular architecture [3,23].

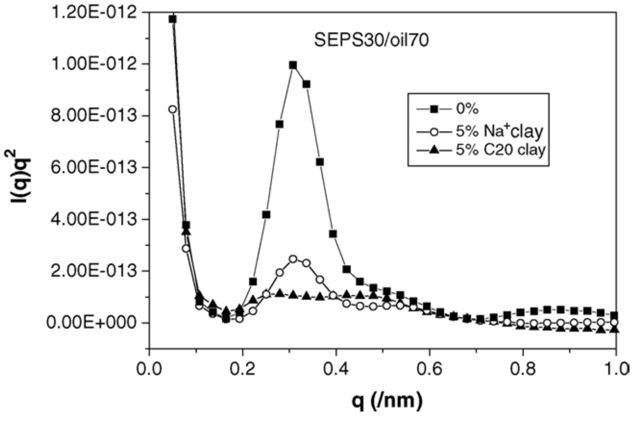

Fig. 5. SAXS results for SEPS30/oil70, SEPS30/oil70/Na$^+$-clay (5 wt.%) and SEPS30/PO70/C20 (5 wt.%) clay nanocomposite gels.

For a given block copolymer, if the degree of the ordered structure reduces this could be certainly resulted by the increasing compatibility between the A–B segments. This fact could force A–B segments contacts and lead them to mix in small domains. From this point of view, the addition of layered silicate in the SEPS/oil system resulted in reduction of ordering level of block copolymer due to weakening repulsive interactions between A–B segments.

Fig. 6 shows DSC results for SEPS/oil/Na$^+$-clay and SEPS/oil/C20 nanocomposite gels. In comparison of two nanocomposite gels, the order–disorder transition of the block copolymer for SESP/oil/C20 system was almost not observed. This indicates that the ordered structure of block copolymer in the nanocomposite gel was completely disturbed by the addition of C20 clay. As shown in Fig. 5, the SAXS results also indicated that the interactions between SEPS and clay have a significant influence on the ordering level of the block copolymer. For SEPS/oil/C20 system, the scattered peak was dismeared and the curve became nearly smooth.

### *3.2. The order–disorder transition behaviour of block copolymers revealed by MDSC*

As indicated above from the DSC results, the order–disorder transition is likely to be a melting transition. The transition behaviour for macro-phase separation in polyether suphone/poly(ethylene oxide) blends was discussed [24]. Macro-phase separation in polymer blends is likely to be a melting transition. From DSC results, it is clear that heat flow versus temperature signals for macro-phase separation of polymer blends and for order–disorder transition of block copolymers are the same [24]. However, according to the mechanism of the order–disorder transition of block copolymers, it is believed that the heat flow (or heat capacity) versus temperature signal of the order–disorder transition of a block copolymer could be different from that of the macro-phase separation of polymer blends. The process of the order–disorder transition of block copolymers could include more complex thermal events. DSC cannot separate the overlapped thermal events [20,21].

MDSC is a relatively new technique [25,26], which can produce not only the total heat flow obtained from conventional DSC, but also separate complex transitions into more easily interpreted components and directly measure heat flow and heat capacity changes from a single experiment. MDSC measurements can present a series of thermographs in the characteristic of modulated flow, total heat flow, reversing and non-reversing heat flows indicating that when the processes encounter the thermodynamic phase transitions and reveals thermal behaviours [25,26]. The thermodynamic phase transitions, for examples glass transition and melting transition, are the reversible events [27,28] detected in reversing signal. The non-reversing signal contains important information on non-reversible kinetic processes such as molecular relaxation, crystallisation, etc. [25–28].

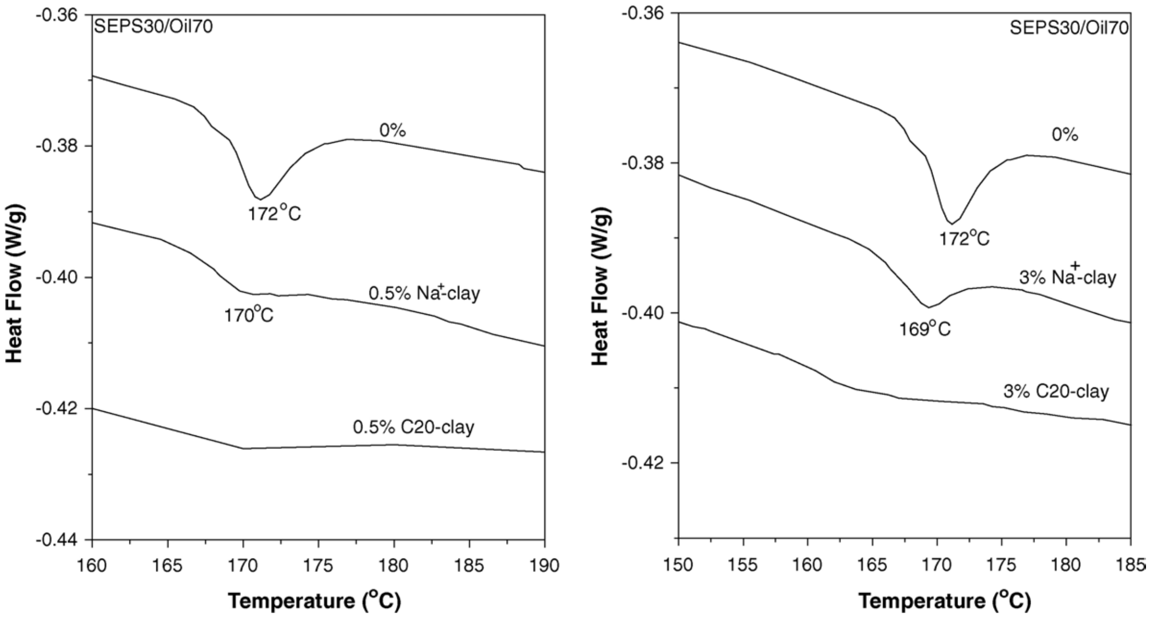

Fig. 6. A comparison of order–disorder transition temperature for different block copolymer nanocomposite gels.

And this equipment is also sensitivity to detection of weak transitions [25,26]. So all of these make it as a more powerful technique. In order to further clarify the behaviour of polymer chain motion at the order–disorder transition, an MDSC technique was applied in the present study.

The total heat flow signal for SEPS30/oil70 gel shown in Fig. 7 was the obvious melting-like transition, which was the similar result with the conventional DSC measurements shown above.

Fig. 8 shows the plots of heat capacity versus temperature, for SEPS30/oil70 gel and its nanocomposite gels. A new phenomenon has been revealed by the reversing signal showing a clear step-change in heat capacity. The change of heat capacity at the temperature associated with the order–disorder transition is similar to that of the glass transition of a polymer.

Macro-phase separation behaviour in polymer blends is melting-like one. For block copolymers only nano-scale separation is possible due to the covalent bond linking the different blocks, which forces them regroup in smaller domains. The micro-phase transition of block copolymers occurred results from that the motion of A–B segments confined in the nanostructures takes place in small domains. According to the kinetics of micro-phase transition, the order–disorder transition is more likely to be described as the glass transition-like one.

Comparing the heat capacity signals of nanocomposite systems shown in Fig. 8 with the system without adding clay, the transition signal obviously becomes weak. Na$^+$-clay partially weakened the ordered structure of the block copolymer. However, the step-change was not observed for C20-clay system, which means that the addition of C20-clay entirely destroyed the ordered structure of the block copolymer in the nanocomposite gel.

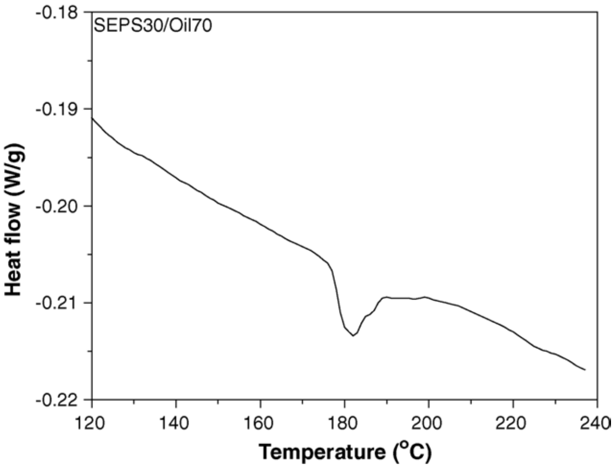

Fig. 7. MDSC total heat flow vs. temperature for SEPS30/oil70 gel.

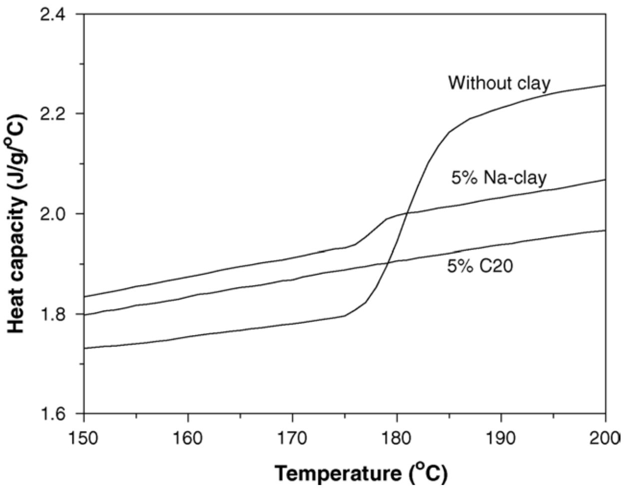

Fig. 8. Heat capacity vs. temperature signals for SEPS30/oil70 gel and its nanocomposite gels.

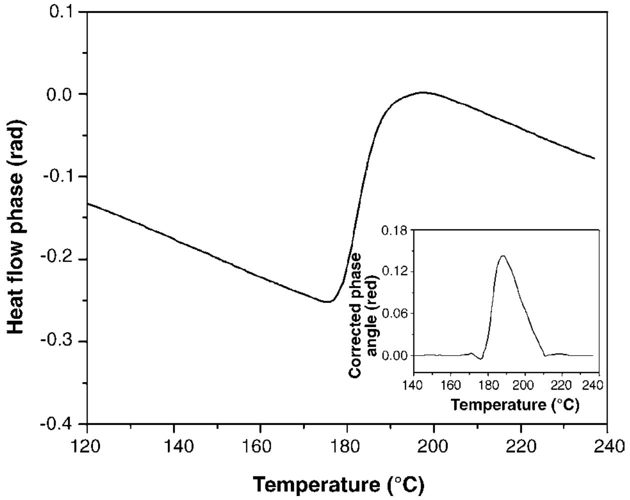

Fig. 9. Heat flow phase angle vs. temperature signal for SEPS30/oil70 gel. Inset is the corrected heat flow phase angle.

The change of heat capacity, $\Delta C_\mathrm{p}$, at $T_\mathrm{ODT}$ can be described by the following equation:

$$\left[\frac{\partial(\Delta H)}{\partial T}\right]_\mathrm{p} = \Delta C_\mathrm{p} = C_\mathrm{p}\,(\text{disordered state}) - C_\mathrm{p}\,(\text{ordered state})$$

$$\Delta H = H\,(\text{disordered state}) - H\,(\text{ordered state})$$

where $H$ is the enthalpy. The difference of the enthalpy between the disordered and the ordered states can be described as follows [29]:

$$\Delta H = \frac{V\phi_\mathrm{A}\phi_\mathrm{B}\chi k_\mathrm{B}T}{V_\mathrm{A}} - \frac{N\chi k_\mathrm{B}T\lambda}{2T_\mathrm{A}}$$

where $V$ is the volume of the system, $V_\mathrm{A}$ the molar volume of an A segment and $\phi_\mathrm{A}$ and $\phi_\mathrm{B}$ are the volume fractions of the A and B components, respectively. $k_\mathrm{B}$ is the Boltzmann constant and $T$ is the temperature. $N$ is the number of A–B molecules in the area of the interface, $\lambda$ the interfacial thickness and $T_\mathrm{A}$ is the size of A-domain. In the above equation, the first term represents the heat mixing of segments in the disordered state and the second term represents the heat mixing of segments in the ordered state in the interface region. Addition of clay could lead to the increase of interfacial phase, which results in the decrease of $\Delta H$. These results shown in Fig. 8 support the analysis on the DSC and SAXS results.

Fig. 9 shows the heat flow phase angle versus temperature signal for SEPS30/oil70 gel, which is the difference between the phase angle of modulated heating rate and the phase angle of modulated heat flow. The inset is the corrected heat flow phase angle. The change of heat flow phase angle at the order–disorder transition is similar to that of semi-crystalline polymers such as poly(ethylene terephthalate) (PET), during crystallisation (see Fig. 10).

Fig. 11 shows non-reversing heat flow, $F$(nr), versus temperature signal for the SEPS30/oil70 gel. The insert is the corrected non-reversing heat flow. An exothermic peak in the corrected non-reversing heat flow signal is obtained at the order–disorder transition as observed in PET during crystallisation (see Fig. 12).

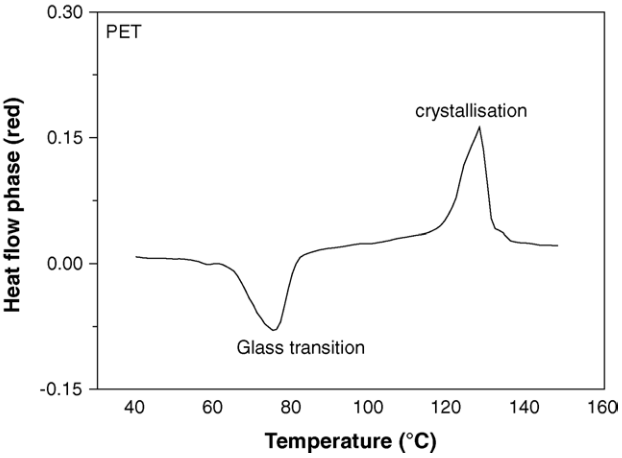

Fig. 10. Variation of heat flow phase vs. temperature for PET.

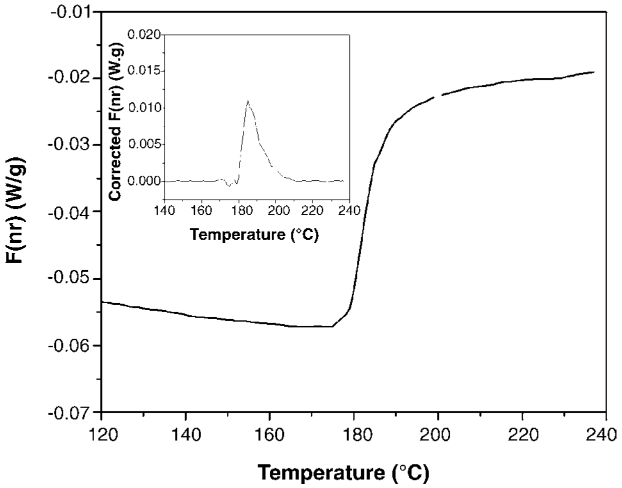

Fig. 11. Non-reversing heat flow vs. temperature signal for SEPS30/oil70 gel. Inset is the corrected non-reversing heat flow signal.

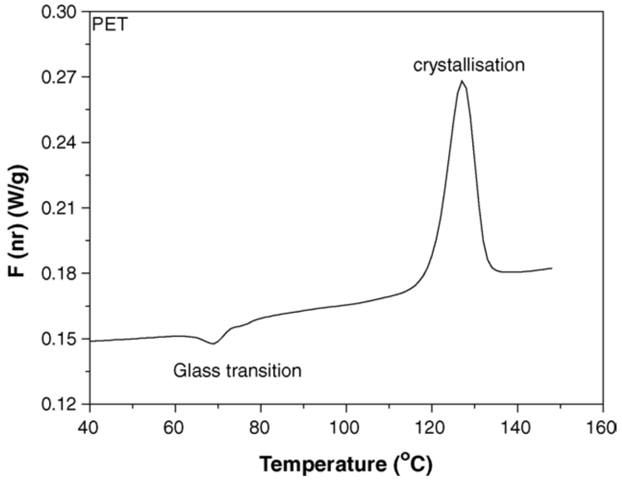

Fig. 12. Non-reversing heat flow vs. temperature signal for PET.

From heat capacity versus temperature signal, the transition from order to disorder is the glass transition-like, because there is a step change in heat capacity. However, non-reversing and the heat flow phase angle versus temperature signals show that the transition from order to disorder seems a crystallisation-like transition.

The kinetics of order–disorder transition in block copolymer has been intensively investigated by scattering and rheology experiments [30–33]. The ordering process in block copolymer bears many similarities with the crystallisation process of semi-crystalline materials [22,30–33].

In fact, during evaporation of the solvent in the casting films, ordering process took place. The thermal event for the ordering process is memorized and hidden in the sample. When the sample was tested by MDSC, it gave insight to the complex thermal processes. As indicated in Figs. 9 and 11, the heat flow phase angle curve and the heat flow distribution curve in non-reversing component show that there were similarities with the crystallisation process of semi-crystalline polymers. The thermal event resulting from the ordering process hidden in the sample was re-shown in the order–disorder transition, which can be revealed by MDSC.

From above studies, it can be concluded that MDSC experiments provide not only the information for understanding the order–disorder transition process and also gains new information relating to the history of the ordering process occurred in the sample.

## 4. Conclusions

DSC and MDSC were applied to study the order–disorder transition of SEPS block copolymer in the block copolymer nanocomposite gels. Introducing layered silicate into SEPS block copolymer has significant influences on the order–disorder transition temperature and phase structure of the block copolymer. With increasing the content of layered silicate, the order–disorder transition temperature decreased. It is lowered by 4 °C when 5 wt.% of layered silicate was introduced into the gel. DSC, MDSC and SAXS results were confirmed that the ordering level of block copolymer in block copolymer/paraffin oil gels was reduced by the addition of layered silicate. MDSC experiments revealed that the order–disorder transition is the process of segment diffusion of the block copolymer associated with the thermal event due to ordering process, which took place in the preparation of the sample.

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