

Shape memory epoxy systems with a wide range of switching temperature

Ye.P. Mamunya, O.K. Matkovska, O.V. Zinchenko, E.V. Lebedev

Institute of Macromolecular Chemistry NAS of Ukraine
48, Kharkivske shose, Kyiv, 02160, Ukraine

The shape memory epoxy polymers (SMEPs) based on the epoxyurethane resin (EPU) modified with poly(ethylene glycol) diglycidyl ether (DEG-1) have been synthesized. Differential scanning calorimetry (DSC) as well as thermomechanical analysis (TMA) have shown that switching temperature (T_{sw}) can be controlled by variation of EPU/DEG-1 ratio (EPU/DEG-1 ratio was varied from 100/0 to 70/30) in the reactive mixture. According to this T_{sw} deviation, associated with glass transition temperature T_g , is in the range 114-45 °C. Shape memory characteristics were determined by cyclic TMA. It was found that shape recovery ratio R_r was changed from 72 % to 84 % in the first cycle and was close to 100 % in the following cycles that demonstrated good reproducibility in multiple shape memory cycles deformation/relaxation.

Key words: epoxy polymer, shape memory effect, switching temperature, glass transition temperature, thermomechanical properties.

Introduction.

Shape memory polymers (SMPs) have attracted increasing interest of both academia and industry. Such materials have a high innovation potential and can be formed as films for packaging, heat-shrinkable tubes, smart fabrics, intelligent medical devices, implants for minimally invasive surgery, self-deployable sun sails in spacecrafts and so on [1–3].

SMPs are stimuli-sensitive polymers which gain a temporary shape under load, can keep a temporary shape for a long time and recover to their original, permanent shape upon applying a stimulus such as heat, light, moisture, pH, chemical or electrical or magnetic field [1, 2, 4]. SMPs with different recovering stimulus are carefully considered in the reviews [4–6]. Thermal activation is the most widely used among all stimuli [7].

Shape memory polymers are generally classified into two groups depending on their chemical structures: thermoset and thermoplastic SMPs. The switch temperature (T_{sw}) between permanent and temporary shape of thermoset SMPs is usually related towards the glass transition temperature (T_g), so it is a key parameter defining the temperature range of their shape deformation and recovery [8, 9]. A typical thermoset polymer is a mixture of many long, entangled molecular chains that are connected by net points created by either chemical cross-linking or physical cross-linking. At temperatures higher than the glass transition temperature, those polymer chains are flexible and a polymer is soft and is characterized by the rubbery state. Authors [10] claim that when an external force is applied, the polymer chains are stretched or contracted to

a large extent and the net points can also be displaced. Reducing the temperature below the T_g in the deformed shape creates some secondary cross-links that can be formed among the deformed polymer chains. These secondary cross-links help to fix the polymer at the temporary shape after the external force is removed. On reheating the polymer above the T_g , the secondary cross-links are released and the original shape is recovered.

Compared with extensive applications of thermoplastic SMPs in civilian commodities the thermoset resin systems are very attractive for the industrial and aerospace applications due to their advantages in chemical stability and high resistance to environmental radiation degradation, unique thermal and mechanical properties [1, 2, 11]. These materials possess excellent shape memory performance such as high shape fixity ratio, shape recovery ratio, rapid response) [8, 12]. Many studies have proven the good shape memory properties of epoxies [13, 14].

Epoxy, as a kind of thermosetting SMPs with high mechanical characteristics and stability in different environments is successfully used in aerospace, medicine (surgery, endo- and exo-prosthesis, blood vessel micro-instruments), automotive, for self-healing polymer materials, membranes, etc. [15–17]. Shape memory epoxy foams and composites were tested on board the BION-M1 spacecraft in the space [18, 19]. Thus widening the range of T_{sw} is very important for successful application of shape memory epoxy polymers (SMEPs) as a final product. Plenty of researchers try to solve this problem.

It is shown [8] that the T_{sw} (T_g) of SMEPs can be well

controlled by simply changing the added content of curing agent. The value of T_g was changed in range from 65 to 140 °C due to the increasing degree of curing in the materials. The samples formed with smaller amount of curing agent contained a part of uncured epoxy affected as a plasticizer. The same idea to change SMEPs T_g was used in ref. [12]. Samples were prepared with varying content of curing agent and as a result the curing degree of epoxy resin was changed from 50 % to 100 % of theoretically fully cured. For those systems the value of T_g was varied from 44,7 to 145,3 °C. The results showed high shape retention ratio (more than 99,5 %) and shape recovery ratio (around 100 %).

Other way was used in ref. [7]. The epoxy-based SMPs were prepared using a mixture of two amine curing agents (IPDA and D230). The values of T_g gradually decreased from 76,5 to 39,4 °C as the content of D230 in the mixture increased. A series of SMEPs [20] were prepared on the base of epoxy resin E-51 and curing agents of 4,4-methylenedianiline (DDM) and m-phenylenediamine (m-PDA) by varying the type of curing agents and their content. The critical shape transition temperature T_{sw} of SMEPs was determined by direct thermal frozen/recovery tests and was changed in the range of 95,5–137,0 °C. Note, that this temperature range is not so wide. The idea to change switching temperature of shape memory epoxy in a large range by varying the formulation needs further elaboration for improving their performances for applications in the field of industry, aerospace, and the others.

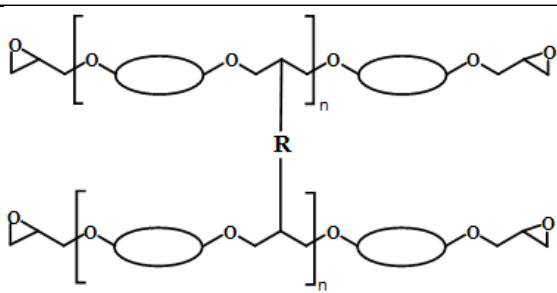

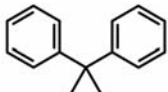
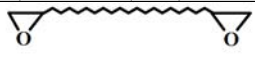

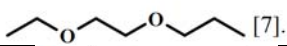
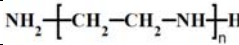
In our work we propose an approach to modify the basic epoxy resin (we used epoxyurethane resin) having high T_g value (114 °C) with poly(ethylene glycol) diglycidyl ether with low T_g value (-10 °C) in different ratios while content of curing agent was the same in all composites. We consider the compatibility of these components and a presence of the sole glass transition in the range between mentioned T_g values. Such an approach allows us to create an epoxy material with controlled temperature of memory shape effect in wide temperature range. Shape memory characteristics were determined by thermomechanical analysis.

Experimental.

The objects of the research were polymer materials synthesized from reactive mixtures of two epoxy oligomers, aromatic and aliphatic: epoxyurethane resin (EPU) taken as a basic polymer and poly(ethylene glycol) diglycidyl ether (DEG-1 trademark) serving as modifier. Also we used polyethylene polyamine (PEPA) as hardener (10 % of reactive mixture). The ratio of EPU/DEG-1 in reactive mixture was in a range of 100/0 - 70/30 (Table 1).

Epoxyurethane resin was produced as a result of reaction of aromatic diglycidyl ether of bisphenol A based resin (ED-20 trademark) with urethane oligomer macrodiisocyanate (MDI) with addition of 1,4-butandiole (BD) as lengthener of molecular chains in proportion ED-20/MDI/BD=65/33/2. MDI was previously synthesized in reaction of polypropylene glycol having MM=1052 with toluene diisocyanate (of T80/20 mark) taken in stoichiometric ratio 75/25. Reaction of synthesis was maintained up to achievement of ca. 6 % of free NCO-groups in the MDI product. Afterwards these NCO-groups reacted with OH-groups of epoxy resin providing integrated chemical structure of epoxyurethane resin. EPU contains the rigid segments remained from the molecular structure of aromatic

Table 1. Reagents for synthesis of EPU/DEG-1/PEPA shape-memory epoxy polymer

| Abbreviation | Chemical structure |
|--------------|---|
| EPU |  <p>where  are rigid segments  [7].</p> <p>R is a radical which contain $-\text{NC}(\text{O})\text{O}-$, $-\text{CH}(\text{CH}_3)\text{CH}_2\text{O}-$, $-(\text{CH}_2)_4-$ and $-\text{C}_6\text{H}_5(\text{CH}_3)-$ groups.</p> |
| DEG-1 |  <p>where  is a flexible segment</p>  [7]. |
| PEPA |  |

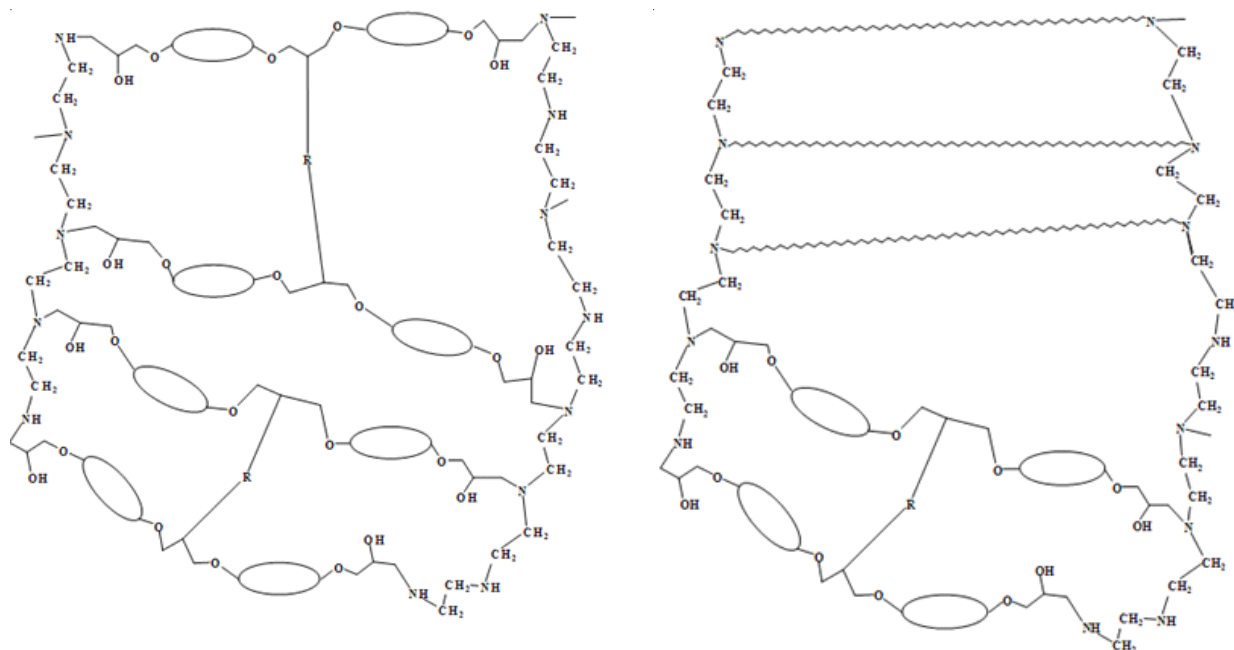


Fig. 1. Chemical structure of the SMEPs where the EPU/DEG-1 ratio equals to 100/0 (a) and is varied from 85/15 to 70/30 (b) specimens, where are rigid and are flexible segments, respectively; R is a radical which contain $-\text{NC}(\text{O})\text{O}-$, $-\text{CH}(\text{CH}_3)\text{CH}_2\text{O}-$, $-(\text{CH}_2)_4-$ and $-\text{C}_6\text{H}_5(\text{CH}_3)-$ groups

resin ED-20, namely the pairs of benzene rings (see Table 1). On the other hand, it also includes the flexible groups of MDI marked as radical R in the EPU molecules structure which is shown in Fig. 1a.

Since the modifier DEG-1 resin includes the flexible $-\text{CH}_2-\text{CH}_2-\text{O}-$ groups the chemical structure of EPU/DEG-1 epoxy polymer consists of a mixture of rigid and flexible chains where a part of rigid structure fragments of EPU is substituted by flexible DEG-1 chains (Fig. 1b). Growing number of flexible chains with increasing of DEG-1 content in SMEPs imparts softer structure of polymer material with lower T_g value.

The reactive mixture consisting of EPU, DEG-1 and hardener after thorough mixing was placed on Teflon plate and cured at ambient conditions during 24 hours followed by film forming of 0,5-0,7 mm thickness.

The TMA self-designed original equipment for thermomechanical analysis at penetration mode was applied to study the shape-memory behavior of the composites synthesized. The resolution of TMA device was 0,5 mm, the heating rate was $5^\circ\text{C}/\text{min}$. The sample with size 7x7 mm cut from the film was put in a cell of TMA device where a probe with diameter 1,6 mm impacted the epoxy sample with constant stress 1,0 MPa. The penetration of probe into the sample was considered as a deformation of polymer (L , %) and calculated according to equation:

$$L = \frac{\Delta l}{l_0} \cdot 100, \quad (1)$$

where Δl is a change of size of the sample along penetration, l_0 is the initial size of the sample.

Glass transition temperature of epoxy polymers (T_g)

was determined using differential scanning calorimetry (DSC) by DSC Q2000 device from TA Instruments, USA, in the temperature range from -60 to $+140^\circ\text{C}$ with heating rate $20^\circ\text{C}/\text{min}$.

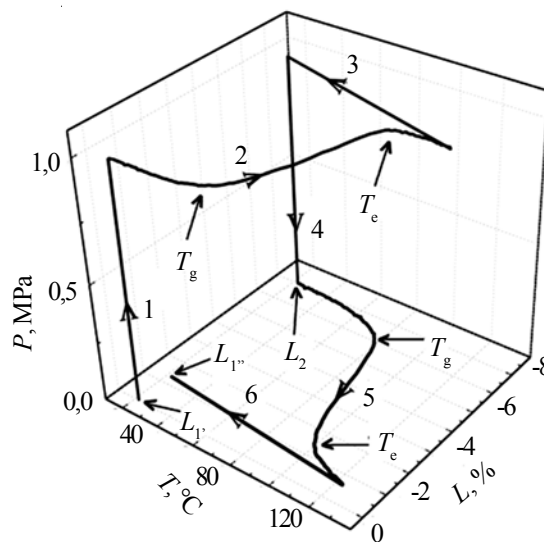


Fig. 2. A scheme of shape memory experiment by TMA for epoxy polymer: 1 – loading at room temperature; 2 – heating under load; 3 – cooling under load; 4 – unloading at room temperature; 5 – heating without load; 6 – cooling without load. The parameters of TMA curves: T_g is glass transition temperature; T_e is temperature of elastic state; L_1' is the value of initial deformation for permanent shape; L_1'' is the value of deformation for permanent shape after relaxation and L_2 is the value of deformation for deformed (temporary) shape of the sample

Results and discussion.

TMA investigation of shape memory effect was carried out in a cycle manner. One cycle includes the following main four stages (Fig. 2):

- heating the sample from room temperature to 120 °C under load of 1.0 MPa getting deformed shape of the polymer sample (curve 2);
- cooling to room temperature under load in order to fix the deformed shape of the sample (curve 3);
- heating the sample to 120 °C without load to provide the relaxation of deformed state of epoxy polymer (curve 5);
- cooling to room temperature without load to fix the original shape of the sample (curve 6).

Following the process of deformation along the TMA curve the glass transition temperature T_g was defined as onset of glass transition region. Above T_g value the deformation of polymer begins and then polymer passes into elastic state that is defined by temperature T_e . In this state polymer is deformed completely by applied load and after cooling under load and subsequent unloading the sample (lines 3 and 4) gains the fixed value of deformation L_2 which is inherent to the temporary shape of polymer. The process of relaxation (shape recovery) includes heating of sample without load and passing of polymer through the temperatures T_g and T_e into elastic state (curves 5 and 6) with fixed value of deformation L_1'' that is inherent to the permanent shape of polymer. Hence, the glass transition temperature T_g can be considered as the switching temperature T_{sw} between permanent and temporary shape of the polymer. Authors [9] point out the important role of difference between temperature of polymer deformation

T_d and onset of transition temperature T_{trans} (which associates with T_g) that influences on recovery behavior.

In Fig. 2 there are shown the parameters which characterized shape memory effect, namely glass transition temperature T_g , temperature of elastic state T_e , value of initial deformation for permanent shape L_1' , value of deformation for permanent shape after relaxation L_1'' and value of deformation for deformed (temporary) shape of polymer L_2 .

For each sample with certain EPU/DEG-1 ratio three successive cycles of measurements was fulfilled (Fig. 3). As it is seen, only loading stage of the first cycle is definitely distinct in L_1' and L_1'' values, rest of cycles are very similar. One of the important characteristics of SMPs is the shape recovery ratio (R_r , %) that reflects the ability of material to reproduce its permanent shape after deformation and relaxation. R_r can be calculated for every cycle represented by TMA curve in accordance with formula:

$$R_r = \frac{L_2 - L_1''}{L_2 - L_1'} \quad (2)$$

Thus, the difference between L_1' and L_1'' defines a shape recovery behavior. The values of shape recovery ratio R_r are given in Table 2. It is seen that for the first cycle the R_r value varies from 72 to 84 % for different EPU/DEG-1 ratios while for the second and third cycles R_r is close to 100 % that demonstrates an excellent shape recovery in a process of deformation/relaxation. Authors [21] also observed the difference of first cycles from each other, the curves became more similar with increasing number of cycles. They claim that during the first cycles a reorganization of the polymer on the molecular scale takes place and single polymer chains arrange in a more favorable way in regard to the direction of deformation. The R_r value is defined by chemical structure of polymer and conditions of measurements and was observed in a wide interval, from 20,0 to 99,7 %.

DSC results of SMEPs measurements are shown in Fig. 4. The curves demonstrate weak kink in the heat flow curves reflecting a presence of glass transition for pure EPU and polymers with low content of DEG-1. With increasing of DEG-1 content the features of glass transition become more expressed and most displayed kink of the heat flow curve is observed for pure DEG-1. It is an evidence that the flexible PU fragments in EPU structure slur over the heat effect of the glass transition. Introduction

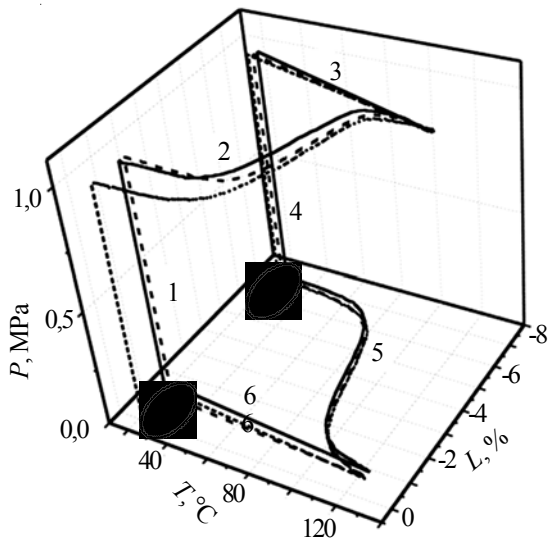


Fig. 3. Three cycles of deformation/relaxation for epoxy polymer with ratio of EPU/DEG-1=85/15 where cycles I, II and III are short dash, dash and solid lines, respectively; A and B are the values of deformation for permanent and temporary shapes, respectively. The curve numbers mean the stage of deformation/relaxation process and are identical to Fig. 2

Table 2. The values of shape recovery ratio depending on composition of epoxy mixture

| EPU/DEG-1 ratio, % | Shape recovery ratio R_r , % | | |
|--------------------|--------------------------------|----------|-----------|
| | Cycle I | Cycle II | Cycle III |
| 100/0 | 72 | 95 | 95 |
| 85/15 | 81 | 99 | 97 |
| 80/20 | 84 | 99 | 100 |
| 75/25 | 82 | 100 | 98 |
| 70/30 | - | - | - |

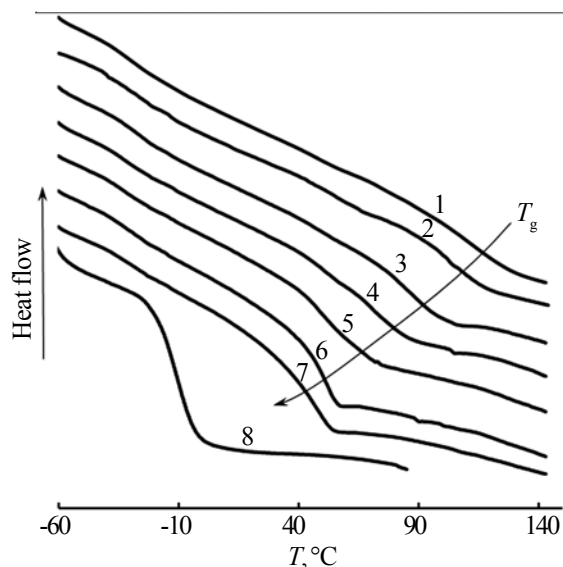


Fig. 4. DSC thermograms of the SMEPs where the EPU/DEG-1 ratio equals to: 1 – 100/0; 2 – 95/5; 3 – 90/10; 4 – 85/15; 5 – 80/20; 6 – 75/25; 7 – 70/30 and 8 – 0/100

of DEG-1 in the EPU changes a part of EPU molecules by DEG-1 chains (Fig. 1) that shifts the value of T_g to lower temperatures and intensifies its displays. The sole glass transition indicates the miscibility of the epoxy oligomers EPU and DEG-1 on the molecular level.

The glass transition temperature T_g of SMEPs measured by DSC and TMA (as onset of glass transition region) methods are shown in Fig. 5. The T_g values decrease (starting from 114 °C for pure EPU) with increasing of DEG-1 content in the reactive mixture. Behavior of T_g for the system with fully miscible components can be described by Fox equation [22]:

$$\frac{1}{T_g} = \frac{w}{T_{g1}} + \frac{1-w}{T_{g2}}, \quad (3)$$

where T_{g1} and T_{g2} are temperatures of glass transition for low-temperature and high-temperature components, respectively, w is weight fraction of low-temperature component. As it is seen, a curve calculated by the Fox equation is far from the experimental points. This can indicate the partial miscibility of epoxy oligomers, EPU and DEG-1. For such case the Taylor-Gordon equation (TG) can be used [23]:

$$T_g = \frac{wT_{g1} + k(1-w)T_{g2}}{w + k(1-w)}, \quad (4)$$

where k is adjustable coefficient. If the Fox equation is symmetrical and implies equal contribution of both com-

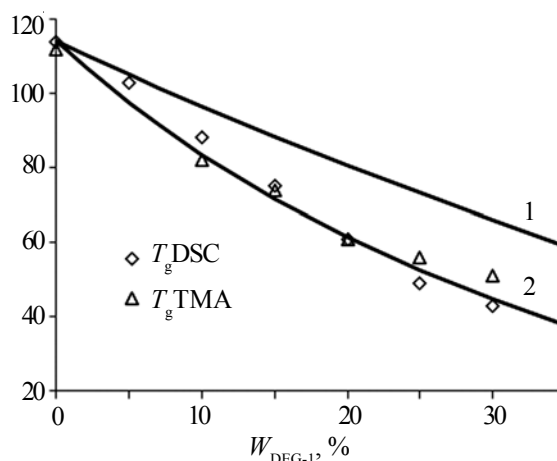


Fig. 5. Glass transition temperatures T_g of the SMEPs versus DEG-1 content in the reactive mixture measured by DSC (triangles) and TMA (diamonds). Calculations in accordance to Fox model (curve 1) and Gordon-Taylor model (curve 2)

ponents, the latter equation is asymmetrical and coefficient k defines a contribution ratio of low-temperature and high-temperature components in T_g value of epoxy polymer. At $k = 0,34$ the TG equation precisely describes the experimental results (Fig. 5). Hence, the modifying of aromatic epoxy oligomer EPU with aliphatic epoxy oligomer DEG-1 provides the change of T_g in wide temperature range. Since the T_g value is identified with switching temperature T_{sw} such an approach enables to control the behavior of shape memory effect in epoxy polymer.

Conclusions.

In this work the shape memory effect of epoxy polymers based on the mixture of two epoxy oligomers, epoxyurethane resin (EPU) and poly(ethylene glycol) diglycidyl ether (DEG-1) has been investigated. It was found that alteration of EPU/DEG-1 ratio from 100/0 to 70/30 (at constant content of hardener PEPA) leads to the change of glass transition temperature T_g from 114 to ca 45 °C. Since T_g value is identified with switching temperature T_{sw} , such an approach makes possible to obtain the materials with shape memory effect in different temperature intervals that gives them the attractiveness for use in different applications, for example, materials with T_{sw} close to temperature of human body are of interest for applications in the biomedical fields [24]. Cyclic study fulfilled by TMA has shown that such a parameter as recovery ratio R_r equals to 70–80 % for the first cycle and close to 100 % for subsequent cycles that indicates good reproducibility under multiple cycles deformation/relaxation.

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Епоксидні системи, що мають ефект пам'яті форми, з широким діапазоном температури переходу

Є.П. Мамуня, О.К. Матковська, О.В. Зінченко, Є.В. Лебедев

Інститут хімії високомолекулярних сполук НАН України
48, Харківське шосе, Київ, 02160, Україна

Синтезовано епоксидні полімери, що мають ефект пам'яті форми, на основі епоксиуретанового олігомеру (ЕПУ), модифікованого дигліцидиловим ефіром поліетиленгліколю (ДЕГ-1). Методами диференційної сканувальної калориметрії (ДСК) та термомеханічного аналізу (ТМА) показано, що температуру переходу (T_g) можна в значних межах регулювати зміною співвідношення олігомерів ЕПУ та ДЕГ-1 в реакційній суміші (співвідношення ЕПУ/ДЕГ-1 змінювали в межах від 100/0 до 70/30). У відповідності до цього, діапазон зміни T_g , котра асоціюється з температурою склування (T_c), знаходиться в межах 114–45 °С. Ефект пам'яті форми вивчали методом циклічного ТМА. При цьому виявлено, що коефіцієнт відновлення форми змінюється в межах від 72 до 84 % на першому циклі дослідження та близький до 100 % на наступних циклах, що демонструє хорошу відтворюваність в багатократних циклах деформації/релаксації.

Ключові слова: епоксидні полімери, ефект пам'яті форми, температура переходу, температура склування, термомеханічні властивості.

Эпоксидные системы обладающие эффектом памяти формы с широким диапазоном температуры перехода

Е.П. Мамуня, О.К. Матковская, О.В. Зинченко, Е.В. Лебедев

Институт химии высокомолекулярных соединений НАН Украины
48, Харьковское шоссе, Киев, 02160, Украина

Синтезированы эпоксидные полимеры, обладающие эффектом памяти формы, на основе эпоксиуретанового олигомера (ЭПУ), модифицированного диглицидиловым эфиром полиэтиленгликоля (ДЭГ-1). Методами дифференциальной сканирующей калориметрии (ДСК) и термомеханического анализа (ТМА) показано, что температуру перехода (T_g) можно в значительных пределах регулировать изменением соотношения олигомеров ЭПУ и ДЭГ-1 в реакционной смеси (соотношение ЭПУ/ДЭГ-1 изменяли в пределах от 100/0 до 70/30). В соответствии с этим, диапазон изменения T_g , которая ассоциируется с температурой стеклования (T_c), находится в пределах 114–45 °С. Эффект памяти формы изучали методом циклического ТМА. При этом выявлено, что коэффициент восстановления формы изменяется в пределах от 72 до 84 % в первом цикле исследования и близок к 100 % на следующих циклах, что демонстрирует хорошую воспроизводимость в многократных циклах деформации/релаксации.

Ключевые слова: эпоксидные полимеры, эффект памяти формы, температура перехода, температура стеклования, термомеханические свойства.