

Improvement of mechanical characteristics of glycidyl azide polymer binder system by addition of flexible polyether

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ABSTRACT: Two kinds of flexible chain polymer, poly(ethylene oxide-*co*-tetrahydrofuran) [P(EO-*co*-THF)] and polyalkylene oxide (PAO), were chosen to improve the mechanical properties of the network of glycidyl azide polymer (GAP)-based elastomers. The mechanical properties of the GAP binder system at 25 and -40°C can be improved effectively. The effects of P(EO-*co*-THF) and PAO on the network parameters, hydrogen bonding effect, and crystallization property were studied to determine the enhancement mechanism. Based on the results, it can be concluded that for copolyurethane elastomers prepared with PAO content less than 15 wt % and P(EO-*co*-THF), the mechanical properties were enhanced by the reduction of bulk side groups in GAP, which improved the chemical crosslinking density, hydrogen bonding effect in elastomers, and the motility of the molecular chains, while for elastomers prepared with more than 15 wt % PAO, the crystallization of the PAO segments played a major role in the improvement of mechanical properties. © 2016 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2016**, *133*, 43840.

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INTRODUCTION

Solid composite propellants are mainly composed of binders, metallic fuels, oxidizers, and other minor additives. Among these ingredients, the binder acts as a matrix to hold the solid fillers together and gives the desired shape and structural integrity to the propellant.¹ Glycidyl azide polymer (GAP) is a unique binder of high density with a positive heat of formation of $+117.2\text{ kJ/mol}$,² and it is greatly insensitive to impact and at the same time provides high burning rates. For its positive effect on the energetic performance and burning rate, the GAP binder has attracted the attention of researchers for the past two decades.³

However, the mechanical properties, especially the low-temperature characteristics of GAP polyurethane, are not as good as some other binder systems, such as hydroxyl-terminated polybutadiene (HTPB) and hydroxyl-terminated polyether (HTPE), because of the chemical structure of the azido binder.⁴ A large number of methylene azido side groups exist in the molecular structure of GAP, which hinder the motion of the molecular chain and lead to a stiff backbone for GAP, while according to the kinetic theory of rubber elasticity⁴ both stress and strain rely on the chain flexibility. Accordingly, the mechanical properties of GAP propellant are poor.

Therefore, different methods have been employed to improve the mechanical properties of GAP propellant, such as the syn-

thesis of higher molecular weight GAP, branched GAP, modification of the chain of GAP with monomers or polymers, which can offer flexibility to the polymer backbone, and introduction of some kinds of flexible chain polymers into the network of the GAP binder system by blending modification.^{5–8}

In recent years, improving the mechanical properties of GAP propellant by blending modification has been paid more attention for its efficiency and simplification. The mechanical properties of GAP elastomers can be improved markedly by introducing different contents of polymers that have a flexible backbone, such as poly(ethylene glycol) (PEG), polycaprolactone (PCP), poly(ethylene oxide-*co*-tetrahydrofuran) [P(EO-*co*-THF)], and HTPB.^{9–14} Although many polymers have been found to have a positive effect on improving the mechanical properties of the GAP binder system, the specific effect of these polymers on a GAP-based network has rarely been reported.

P(EO-*co*-THF) is a copolymer of EO (ethylene oxide) and THF (tetrahydrofuran), in which the THF segments offer superior strength and low-temperature properties, while the EO segments give rise to a favorable flexibility of the polymer chain, and it has been reported that P(EO-*co*-THF) has a positive effect on the improvement of mechanical properties of a GAP binder system with TMP (trimethylolpropane) as crosslinking agent and IPDI (isophorone diisocyanate) as curing agent.¹⁴ Because of the low degree of regularity of the P(EO-*co*-THF) binder

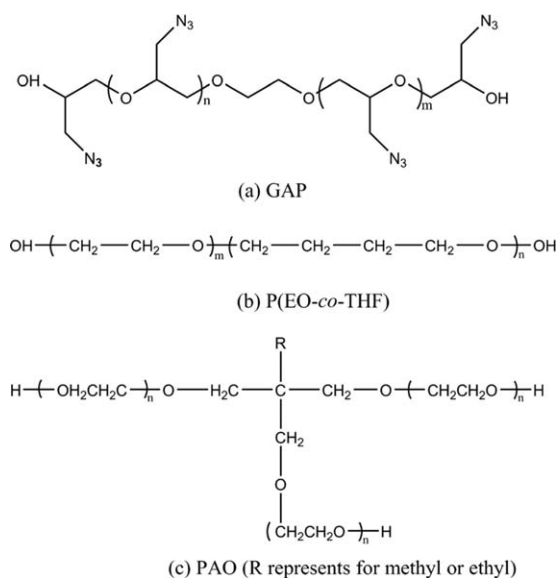


Figure 1. Chemical structures of GAP, P(EO-co-THF), and PAO.

system, a large number of flaws exist in the P(EO-co-THF) polyurethane network.¹⁵

Polyalkylene oxide (PAO) is a polymer with a well-defined structure that consists of flexible ethyl ether segments and has a relatively large molecular weight. It can be used as a binder in NEPE (nitrate ester plasticized polyether) propellant and exhibits good mechanical properties.¹⁶ Hence, it can be expected that the mechanical properties of a GAP binder system can be improved by blending PAO into it. The chemical structures of GAP, P(EO-co-THF), and PAO can be seen in Figure 1.

In this study, P(EO-co-THF) and PAO were chosen to be blended with GAP to form copolyurethane networks with Desmodur N-100 and IPDI as mixed curing agents, and the effects of these two kinds of flexible polyethers on the mechanical properties, hydrogen bonding effect, and network structure parameters of GAP-based polyurethane were studied to determine the enhancement mechanism. Finally, the low-temperature mechanical properties were tested.

EXPERIMENTAL

Materials

GAP (OH index: 0.49 mmol/g, $M_n = 4000$), P(EO-co-THF) (OH index: 0.475 mmol/g, $M_n = 4125$), and PAO (OH index: 0.1 mmol/g, $M_n = 30,000$) were supplied by Liming Research Institute of Chemical Industry, Henan, China. P(EO-co-THF) is a liquid-state copolymer consisting of ethylene oxide and tetrahydrofuran (molar ratio 1:1). Isophorone diisocyanate (IPDI, NCO index: 8.99 mmol/g), and Desmodur N-100 (N-100, NCO index: 5.38 mmol/g), used as curing agents, were provided by Bayer (Beijing, China). The curing catalyst used was a 5% solution of triphenyl bismuth (TPB, 99%, Beijing Chemical Industry) and dibutyltin dilaurate (DBTDL, Beijing Chemical Industry) with a mass ratio of 3:1 in dibutyl phthalate (DBP, Beijing Chemical Industry). Dimethylacetamide (DMAC) was purchased from Beijing Chemical Industry (Beijing, China) and was used as received.

Preparation of Copolyurethane Elastomers

To prepare the copolyurethane elastomers, P(EO-co-THF) or PAO was first mixed with GAP and dried in a vacuum oven at 60 °C for 2 h. Then the required amounts of curing agents IPDI and N-100 were added. The NCO/OH equivalent ratios of all samples were 1.2:1. After the mixing of polymers and curing agents, a curing catalyst was added and mixed in thoroughly, followed by degassing in a vacuum oven at 40 °C. The mixture was finally poured into a Teflon mold and cured at 60 °C for 7 days. GAP/P(EO-co-THF) copolyurethane elastomers were prepared with P(EO-co-THF) weight contents of 5%, 10%, 15%, 20%, 25%, and 30%, and GAP/PAO copolyurethane elastomers were prepared with PAO weight contents of 5%, 10%, 15%, 20%, 25%, and 30%.

Characterization

The mechanical properties of dumbbell specimens were measured with a tensile testing machine [AGS-X, Shimadzu Co., Ltd. (Kyoto, Japan)] at a crosshead speed of 100 mm/min at 25 and -40 °C. Five specimens were tested for each sample to obtain average values. Samples were measured after conditioning the specimen at a given temperature for a minimum duration of 4 h.

The parameters of the network structure of elastomer crosslinking density (ν_e) and average molecular weight between crosslinks (M_c) were determined with a swelling experiment.¹⁰

Fourier transform infrared (FTIR) spectra were recorded on a Nicolet FTIR-8700 Thermo (Waltham, MA, United States) with a wave number resolution of 4 cm^{-1} and a single average of 32 scans at room temperature. Each sample for infrared analysis was obtained with attenuated total reflection (ATR).

A dynamic thermomechanical analysis (DMA) was carried out on a Mettler DMA/SDTA861e (Zurich, Switzerland) over the temperature range from 25 to 90 °C at a heating rate of 2 °C / min at a frequency of 10 Hz.

Differential scanning calorimetry (DSC) curves were measured over the temperature range from -100 to 80 °C on a Mettler DSC 1 at a scanning rate of 10 °C/min in a nitrogen atmosphere (40 ml/min).

RESULTS AND DISCUSSION

Effect of P(EO-co-THF) and PAO on the Mechanical Properties of Copolyurethane Elastomers

First, the effect of two different kinds of flexible polyether on the mechanical properties of blend networks at 25 °C was studied. The stress-strain curves are shown in Figure 2. As the addition of P(EO-co-THF) and PAO in the GAP system will lead to a reduction in the energy performance of the propellant, the added P(EO-co-THF) and PAO content in the GAP system was not more than 30 wt %.

It can be seen in Figure 2 that the stress-strain curves of all the elastomers exhibit the property of high elasticity. The two kinds of elastomers each show the tendency that the modulus, tensile strength, and elongation are enhanced with an increasing content of flexible polyether in the elastomers. Meanwhile, the areas under the stress-strain curves increase accordingly, which means

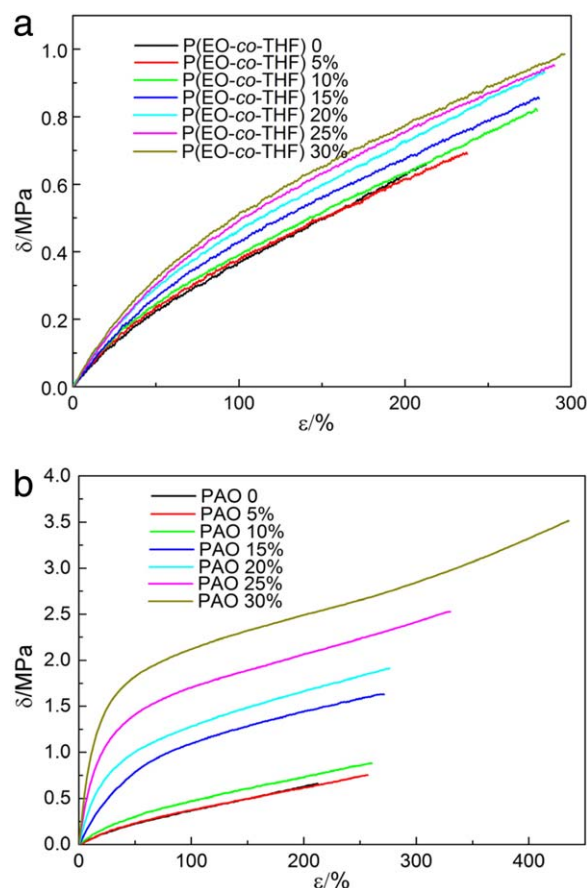


Figure 2. Stress–strain curves of elastomers: (a) GAP/P(EO-*co*-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

enhanced storing ability of volume-specific energy, and more energy could be absorbed when the elastomers were stretched. Furthermore, it can be noticed that for GAP/PAO elastomers, the modulus is elevated as strain is increased when the content of PAO is greater than 15 wt %. This could be because the enhanced molecular orientation by inducing strain was formed in elastomers with larger PAO content.

The specific values of mechanical properties are listed in Table I. It can be seen that the tensile strength and elongation of the elastomer prepared with only GAP were only 0.662 MPa and 212.4%, respectively. The mechanical properties reached 0.986 MPa, 296.1%, and 3.521 MPa, 435.4%, respectively, when the content of P(EO-*co*-THF) and PAO was 30 wt %. Meanwhile,

Table I. Mechanical Properties of Copolyurethane Elastomers

Content of flexible polyether (wt %)		0	5	10	15	20	25	30
δ_m (MPa)	GAP/P(EO- <i>co</i> -THF)	0.662	0.694	0.824	0.857	0.933	0.953	0.986
	GAP/PAO	0.662	0.753	0.885	1.633	1.917	2.528	3.512
ϵ_b (%)	GAP/P(EO- <i>co</i> -THF)	212.4	237.4	279.6	280.7	284.1	289.9	296.1
	GAP/PAO	212.4	256.7	260.4	271.4	276.1	330.1	435.4

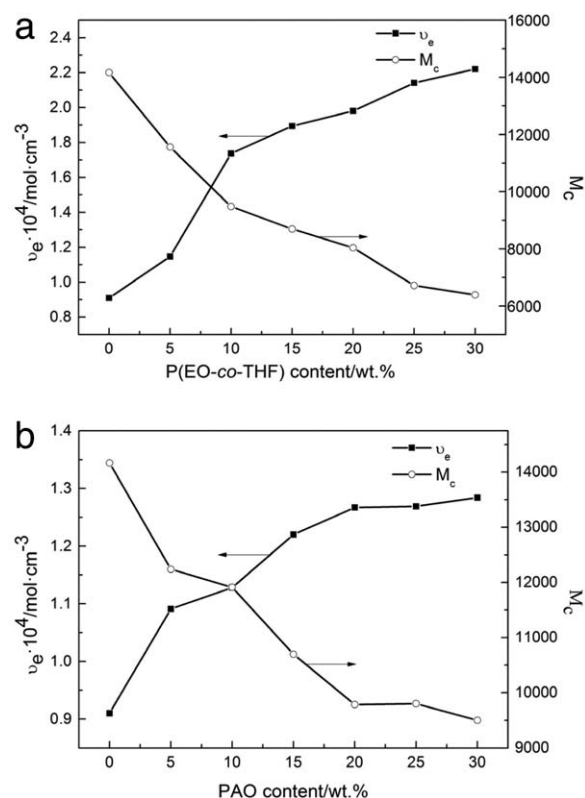


Figure 3. Values for v_e and M_c of copolyurethane elastomers: (a) GAP/P(EO-*co*-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers.

the tensile strength of GAP/PAO elastomers was always larger than that of GAP/P(EO-*co*-THF) elastomers at the same flexible polyether content. It can also be observed that the elastomers prepared incorporating PAO with GAP exhibited relatively higher mechanical properties with a PAO content larger than 15 wt %: the tensile strength and elongation were increased by 0.85 to 2.9 times and 10–175%, respectively, compared to that for the 10 wt % content of PAO. Meanwhile, no similar tendency can be seen for the elastomers prepared with GAP and P(EO-*co*-THF).

Effect of P(EO-*co*-THF) and PAO on Network Parameters of Copolyurethane Elastomers

The crosslinking density and average molecular weight between crosslinks were first determined through a swelling experiment, which can be used to account for the change in mechanical properties of polymeric networks. Figure 3 shows the variation of the values of v_e and M_c for copolyurethane prepared with

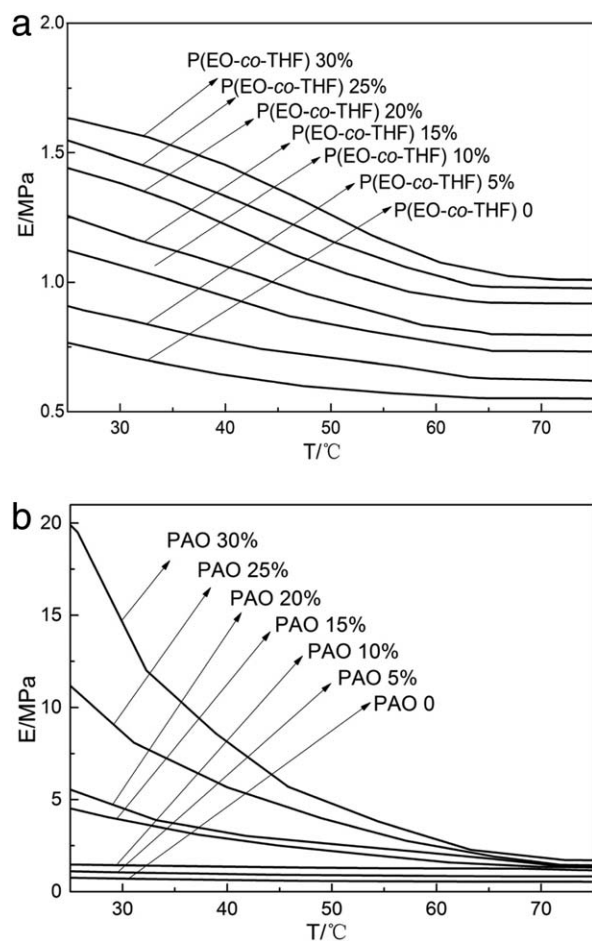


Figure 4. Change of elastic modulus of copolyurethane elastomers along with the temperature: (a) GAP/P(EO-*co*-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers.

different P(EO-*co*-THF) and PAO contents. The same trend can be seen in Figure 3(a) and Figure 3(b): the values of v_e increased with increasing content of P(EO-*co*-THF) and PAO.

Two reasons can be responsible for this tendency. First, bulk side groups exist in the GAP molecular structure, which increases the distance between molecular chains, resulting in an increase in free volume of the networks.¹¹ The incorporation of P(EO-*co*-THF) or PAO with GAP reduces the free volume and leads to the formation of tighter networks with higher values of v_e . Secondly, the hydrogen bond effect in GAP polyurethane is much weaker than in polyurethane which is consisted of polymers that have no side groups.¹⁷ Therefore, the physical crosslinking effect was enhanced by the introduction of P(EO-*co*-THF) or PAO in GAP networks, which also leads to a higher value of v_e and a lower value of M_c .

A higher value of v_e leads to better tensile strength; meanwhile, the decreasing hindrance effect of the bulk side groups in GAP enhances the mobility of molecular chains in the network. As a consequence, both tensile strength and elongation of the elastomers were improved with the introduction of flexible polyether P(EO-*co*-THF) and PAO. However, the elastomers prepared with PAO and GAP always showed a higher tensile strength

than that of GAP/P(EO-*co*-THF) elastomers at the same GAP content, while the values of v_e were lower. This can be due to the number of defects in the networks that are reduced by the introduction of the regular structure polymer PAO.

To study the mechanism of the enhancement effect, it is necessary to figure out the effects of chemical crosslinking and physical crosslinking in copolyurethane elastomers. The physical crosslinking effect includes hydrogen bonding, crystallization, chain entanglement, and phase separation effects, and it is hard to separate.

According to the statistical theory of crosslinked rubber,¹⁸ the crosslinking density can be determined from the elastic modulus, which is the effective sum of both chemical and physical crosslinking effects through the following equation:

$$E = 3v_e RT = 3(v_c + v_p) RT \quad (1)$$

where E is the elastic modulus, v_p is the physical crosslinking density, v_c is the chemical crosslinking density, R is the general gas constant, and T is the absolute temperature.

Upon an increase in temperature, the extent of the hydrogen bond and chain entanglement effect in polyurethane diminishes,¹⁷ and at the same time the crystallization in the elastomers melts. In this circumstance, the physical crosslinking effect seldom affects the elastic modulus. In this way, we are able to calculate the chemical and physical crosslinking density in elastomers semiquantitatively.

Figure 4 shows the change of elastic modulus of copolyurethane elastomers along with the temperature rise from 25 to 75 °C. It can be observed in Figure 4 that the values of E decrease as the temperatures increase from 25 to 75 °C for all elastomers, which indicates that the physical crosslinking effect diminishes. For copolyurethane elastomers prepared with 15%, 20%, 25%, and 30% PAO weight content, the values of E decrease sharply as the temperature rises.

The calculated values of chemical and physical crosslinking density of copolyurethane elastomers are shown in Table II. Both chemical and physical crosslinking density increased with increasing content of P(EO-*co*-THF) and PAO in the elastomers, which means the formation of tighter networks. The values of crosslinking density calculated by elastic modulus in GAP/PAO elastomers were always higher than that in GAP/P(EO-*co*-THF) elastomers at the same GAP content. While this result is in contrast with what was determined by the swelling experiment, this is due to the inherent nature of these two methods.

Meanwhile, for copolyurethane elastomers prepared with larger PAO content (larger than 15 wt %), the value of the physical crosslinking density is much larger than the chemical crosslinking density. The same tendency did not exist for the GAP/P(EO-*co*-THF) copolyurethane elastomers. The higher values of physical crosslinking density may be attributed to the crystallization of PAO segments in elastomers.

Effect of the Content of P(EO-*co*-THF) and PAO on the Hydrogen Bonding Effect of Copolyurethane Elastomers

Hydrogen bonding is the most important physical crosslinking effect in polyurethane elastomers; it exists between urethane

Table II. Values of Chemical and Physical Crosslinking Density of Copolyurethane Elastomers

		Content of flexible polyether (wt %)						
		0	5	10	15	20	25	30
$\nu_c \times 10^4$ (mol cm ⁻³)	GAP/P(EO-co-THF)	0.63	0.71	0.84	0.92	1.05	1.12	1.16
	GAP/PAO	0.63	0.96	1.34	1.53	1.59	1.65	1.96
$\nu_p \times 10^4$ (mol cm ⁻³)	GAP/P(EO-co-THF)	0.41	0.48	0.60	0.76	0.92	0.97	1.03
	GAP/PAO	0.41	0.57	0.70	3.85	6.04	15.27	24.22

—NH groups and possible proton acceptors.¹⁹ At room temperature, most of the hydrogen bonding forms between —NH and carbonyl groups, while for the carbonyl groups that participate in hydrogen bonding formation, the FTIR absorption peaks shift to lower wave numbers. Therefore, the effect of P(EO-co-THF) and PAO on the hydrogen bonding effect of copolyurethane elastomers can be estimated by FTIR.

Figure 5 presents the FTIR spectra of the carbonyl stretching region of GAP/P(EO-co-THF) and GAP/PAO copolyurethane elastomers. The carbonyl bands at around 1725 cm⁻¹ and 1690 cm⁻¹ can be assigned to free carbonyl and hydrogen-bonded carbonyl, respectively. Through a comparison of the area below the two bands, the ratio between the area below the

hydrogen-bonded carbonyl band and the sum of the area below the hydrogen-bonded and free carbonyl bands can be calculated as the fraction of hydrogen-bonded carbonyl groups *R*. The results are shown in Table III. As expected, the degree of hydrogen bonding is heightened by the addition of P(EO-co-THF) and PAO in the GAP elastomer, which indicates the formation of more lightly crosslinked networks, which scatter input mechanical energy more readily and exhibit greater resistance to tension.²⁰

Effect of P(EO-co-THF) and PAO on the Crystallinity of Copolyurethane Elastomers

DSC was used to study the crystallinity of the two kinds of copolyurethane elastomers. Figure 6 shows the DSC curves of elastomers prepared with different contents of P(EO-co-THF) and PAO. In Figure 6(a), no melting peak can be observed in the DSC curves, which means no crystallization exists in the GAP/P(EO-co-THF) elastomers. In Figure 6(b), melting peaks can be found at around 39 °C only in the DSC curves of elastomers prepared with higher PAO weight content (20 wt %, 25 wt %, and 30 wt %).

The difference in molecular structure between P(EO-co-THF) and PAO can be responsible for this phenomenon. It can be seen in Figure 1 that P(EO-co-THF) is a copolymer of EO and THF. The regularity of the molecular chain of P(EO-co-THF) is destroyed by random copolymerization, which makes it difficult to form crystals. While PAO consists of regular C—O—C segments, which easily form crystals, in GAP/PAO copolyurethane networks, the molecular motion is suppressed by the hindrance effect caused by the methylene azido group of GAP, which makes it difficult for the PAO segment to crystallize. With the decreasing content of GAP, the hindrance effect of the bulk side groups diminished and crystallization formed in the PAO segments; accordingly, the mechanical properties were enhanced markedly. No melting peak was observed in the DSC curves of the elastomer prepared with 15 wt % PAO content, whereas it exhibited a relatively large value of physical crosslinking density; it can be assumed that stress-induced crystallization occurred during the process of tension.

Low-Temperature Mechanical Properties of Copolyurethane Elastomers

As the low-temperature characteristics are a critical issue for the GAP binder system, the mechanical properties of different samples at -40 °C were tested, and the stress-strain curves are shown in Figure 7. It can be seen in Figure 7 that the GAP elastomer has a relatively higher initial modulus, while the elongation is poor. This is attributed to the higher glass-transition

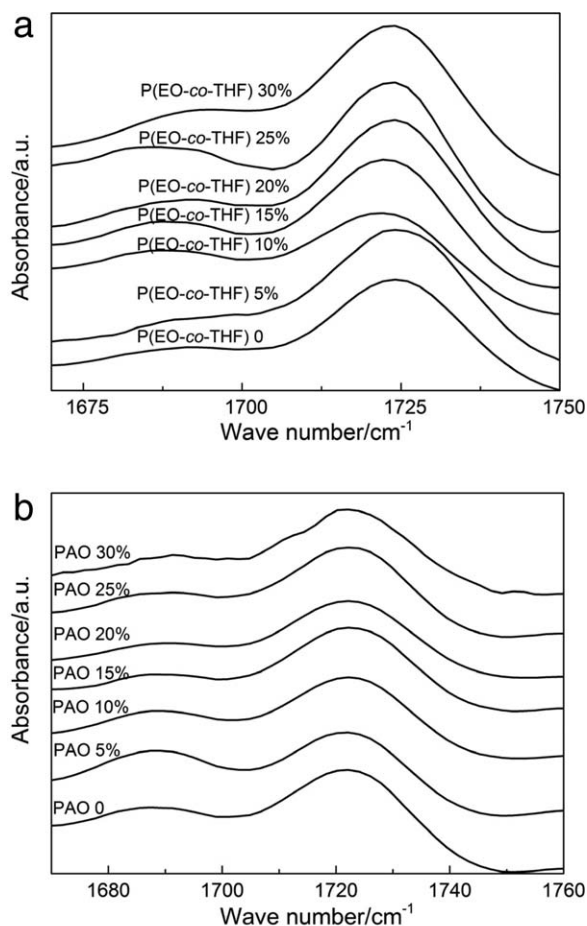


Figure 5. FTIR spectra of carbonyl stretching region for copolyurethane elastomers: (a) GAP/P(EO-co-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers.

Table III. Fraction of Hydrogen-Bonded Carbonyl Groups in Copolyurethane Elastomers

		Content of flexible polyether (wt %)						
		0	5	10	15	20	25	30
R (%)	GAP/P(EO-co-THF)	30.05	39.11	44.54	45.73	49.78	51.17	52.66
	GAP/PAO	30.05	33.52	37.01	43.07	49.53	51.35	54.03

temperature of the GAP elastomer, which was about -41°C and can be seen in Figure 6. With the addition of flexible polyether, the motility of the molecular chains was enhanced; the elongation of elastomers increased accordingly, and the initial modulus diminished. Meanwhile, the GAP segment in the elastomers kept the tensile strength at a high value. It also can be seen that the typical tensile curve of a crystalline polymer appears on the stress–strain curves of elastomers prepared with higher PAO weight content (20 wt % and 30 wt %), so the

initial modulus, tensile strength, and elongation of the elastomers was enhanced with the increasing content of PAO.

As a consequence, two enhancement mechanisms can be determined. The incorporation of P(EO-co-THF) or PAO with GAP leads to the formation of tighter networks and enhances both the chemical crosslinking density and the physical interaction such as hydrogen bonding in the copolyurethane network, which enhances the tensile strength. Meanwhile, the reduction of bulk side groups in GAP makes the molecular chains easy to move and improved the elongation. The extremely high physical crosslinking density in copolyurethane networks containing more than 15 wt % PAO was caused by the crystallization of the PAO segments. The diagrammatic structure of the three kinds of networks are shown in Figure 8.

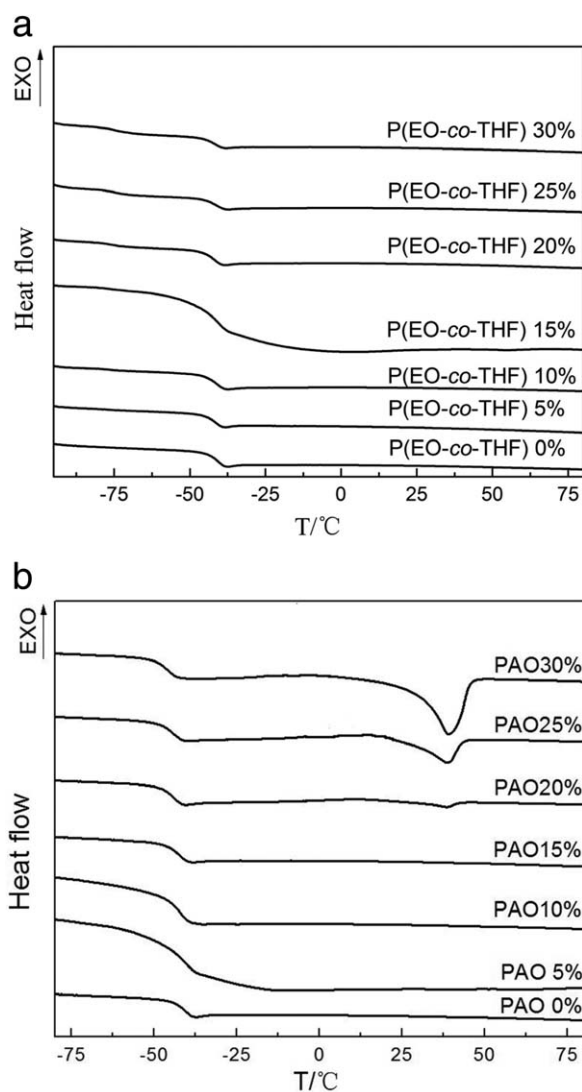


Figure 6. DSC curves of copolyurethane elastomers: (a) GAP/P(EO-co-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers.

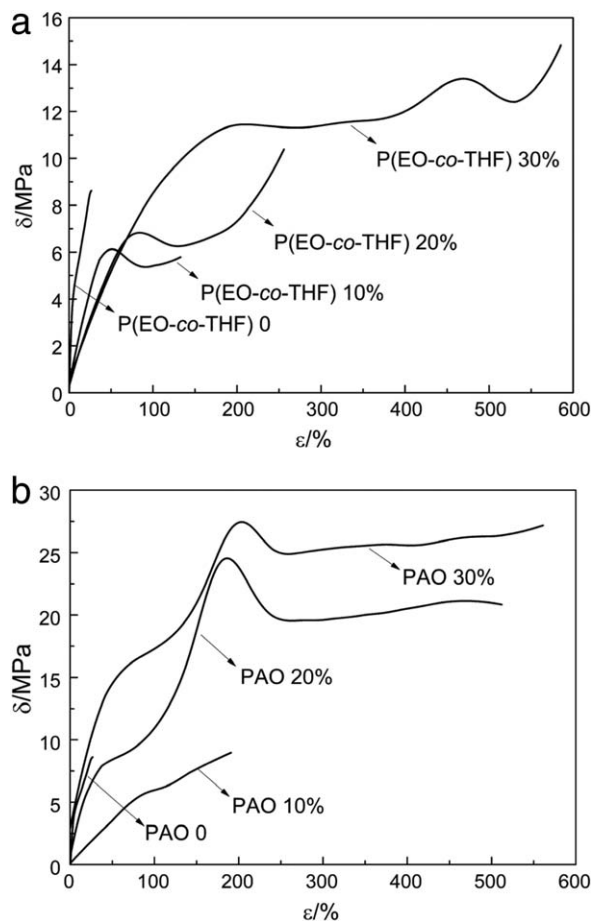


Figure 7. Stress–strain curves of copolyurethane elastomers at -40°C : (a) GAP/P(EO-co-THF) copolyurethane elastomers; (b) GAP/PAO copolyurethane elastomers.

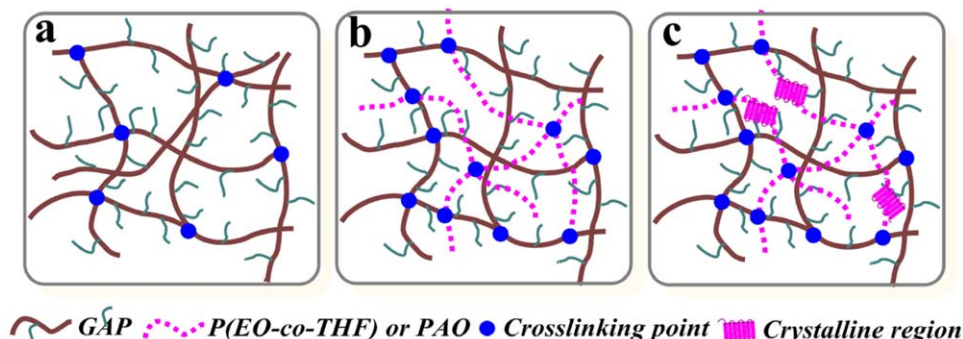


Figure 8. Description of elastomeric networks: (a) GAP polyurethane network; (b) copolyurethane network of GAP/P(EO-co-THF) and GAP/PAO (PAO < 15 wt %); (c) copolyurethane network of GAP/PAO (PAO ≥ 15 wt %). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSIONS

The addition of P(EO-co-THF) and PAO enhanced physical interactions like hydrogen bonding in the copolyurethane network. At the same time, it leads to the formation of tighter networks, which results in an increase in both chemical and physical crosslinking density. For copolyurethane elastomers prepared with larger PAO content (PAO ≥ 15 wt %), the values of physical crosslinking density of the elastomers was much larger than that of other elastomers. The DSC curves of elastomers prepared with PAO content larger than 15 wt % exhibited a crystal melting peak, while no melting peak could be seen in the DSC curves of other elastomers. On account of the increasing crosslinking density, the tensile strength at 25 °C was enhanced by 0.04 to 0.19 times, and 0.14 to 2.9 times with the added content of P(EO-co-THF) and PAO increasing from 5 to 30 wt %, and the corresponding elongations were improved by 25–83% and 44–223%, respectively. Meanwhile, the mechanical properties at –40 °C were also enhanced by the introduction of a flexible polyether.

The mechanism of the enhancement of mechanical properties was determined. For elastomers prepared with P(EO-co-THF) and lower PAO content (PAO < 15 wt %), the mechanical properties were enhanced by the reduction of bulk side groups in GAP, which improved the chemical crosslinking density, the hydrogen bonding effect of the elastomers, and the motility of the molecular chains, resulting in improvements in both tensile strength and elongation accordingly. And because of the regular structure of PAO, fewer defects existed in the networks, the result of which was that the tensile strengths of GAP/PAO elastomers were always larger than that of GAP/P(EO-co-THF) elastomers at the same flexible polyether content. For elastomers prepared with more than 15 wt % PAO, the crystallization of PAO segments played a major role in the improvement of the mechanical properties of the GAP binder system.

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