Oriented Distribution Structure, Interaction, and Performance of Thermoplastic Polyurethane/Selective Hindered Amine Hybrids

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ABSTRACT: By employing the melt blending method, selective hindered amines were oriented to disperse in the hard phase of thermoplastic polyurethane (TPU). The microstructure and performance of organic hybrids consisting of TPU and poly-[(1-hydroxyethyl -2,2,6,6- tetramethyl-4-hydroxyl piperidine)]-glycolsuccinate (GW-622) and poly-{[6-(1,1,3,3-tetramethylbutyl) -imino]-[1,3,5-triazine-2,4- dimethyl] [2-(2,2,6,6- tetramethyl piperidinyl)- imino]-cyclohexane-[4-(2,2,6,6-tetramethyl piperidinyl)-imino]} (GW-944) were investigated by dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC), scanning electric microscopy (SEM), and tensile measurement. The neat TPU matrix displayed only one glass transition peak in the DMA curve, whereas the TPU/GW-622 hybrids exhibited two overlapping loss peaks, and the TPU/GW-944 hybrids demonstrated two separated relaxation peaks. The second excited relaxation peak of the hybrids was attributed to the oriented distribution of hindered amine in TPU and strong intermolecular interactions between the hindered amine and the hard segments of TPU. The complex and interesting evolution in structure and properties of the hybrids with hindered amine incorporation was interpreted. The loss peak areas (TA) of the hybrids increased greatly, and the high temperature damping properties was expected to improve. This kind of novel hybrid presented a promising future as high performance damping material. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 120: 906–913, 2011

Key words: TPU; hindered amine; dynamic property; intermolecular interaction

INTRODUCTION

During recent years, some studies have been focused on the organic hybrids consisting of polymers and small molecules because of their unique physical properties.[1–7] Many applications of organic hybrids have been reported, for example, the use of these hybrids as damping materials[2] and shape memory rubber.[8] In these cases, forming strong intermolecular interactions (such as hydrogen bond) between the polymer and small molecule was of importance in improving the dynamic mechanical properties of the hybrids. This is because strong interactions would result in both fine dispersion of polar small molecules in the matrix and increase of intermolecular friction. The strong interactions further caused large energy consumption during dynamic deformations. Obviously, selecting the suitable small molecules and polymers is the key to construct the hybrids with strong intermolecular interactions. Wu and coworkers disclosed that thermoplastic chlorinated polyethylene (CPE)/hindered phenol (AO-80, AO-60, AO-70) hybrids exhibited remarkable damping properties.[8–10] A crosslinked rubber hybrid with both excellent damping properties and mechanical performance consisting of hindered phenol (AO-80, AO-60) and nitrile butadiene rubber was prepared in our laboratory.[3–5]

As is well known, TPUs are linear block copolymers having hard segments and soft segments in one system. The hard segments provide multifunctional tie points that act as both physical crosslinking points and reinforcing fillers. The soft segments form an elastomer matrix that accounts for the elastic properties of TPU.[11–13] Owing to its excellent mechanical performance, adhesion properties, and processing properties, TPU was applied as damping materials in many engineering fields. However, its
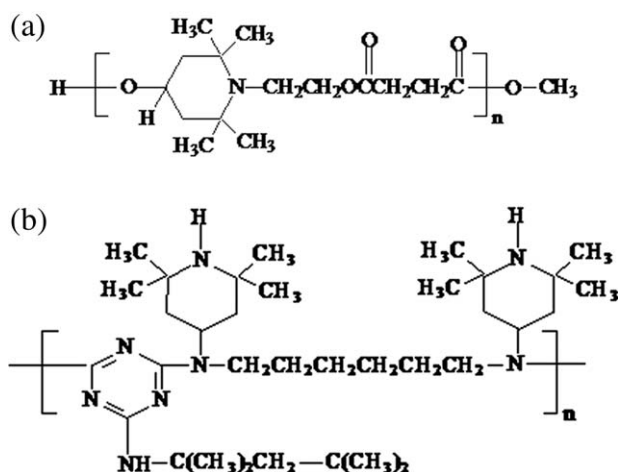
Figure 1 Chemical structures of (a) GW-622 and (b) GW-944.

damping performance was limited by its special two-phase structure and the chemical linking between the hard phase and soft phase. TPU usually exhibits a small loss peak at low temperatures corresponding to the glass transition of the soft phase. In our recent study,[14] the damping properties of thermoplastic polyurethane (TPU) elastomers/hindered phenol (AO-80) hybrids were investigated in detail. TPU/AO-80 hybrids had high tanδ values and a wide range of glass transition temperatures. More interestingly, AO-80 molecules formed strong intermolecular interactions with only the soft segments of TPU, therefore leading to the great evolution of glass transition peak of TPU at low temperatures. So it was believed that AO-80 could improve the low temperature damping properties of the TPU. In the viewpoints of application and science, it is very interesting to know if we can construct a TPU hybrid in which small molecules strongly interact with the hard segments of TPU and if the weak transition corresponding to the glass relaxation of the hard phase could be excited and further greatly enhance the damping properties of TPU at high temperatures.

Considering that the hard segments of TPU contains mainly -OOCNH- groups, two low-molecular-weight hindered amines were selected in this study to blend with TPU to first prepare hindered amine/polymer hybrids, as shown in Figure 1. These two hindered amines were commercially available as light stabilizers. There are some polar amine groups on the molecular chain of the hindered amines, which are expected to markedly increase the compatibility between the hindered amine and hard segments of TPU. Of the two hindered amines GW-622 and GW-944, the latter contains more amine groups.

By employing the melt blending method, the selected hindered amines were oriented to disperse in the hard phase of TPU. In this study, we characterized the additive effects of GW-622 and GW-944 on the dynamic mechanical properties and static mechanical properties of TPU. The results showed that TPU/GW-622 hybrids had relatively high loss tangent value and double overlapping transition peaks, properties that greatly broadened the application areas of damping materials. However, TPU/GW-944 hybrids showed two transitions peaks far from each other coupled with an effective loss tangent value. Besides, the static mechanical properties decreased with increasing content of hindered amine in both hybrids. Some interesting interpretations were made based on these phenomena.

EXPERIMENTAL

Materials

The TPU (Desmopan DP9380A) used in this research was provided by Bayer Co. Ltd. (Germany). GW-622 and GW-944, in powder form, were purchased from Beijing Additive Research Institute (Beijing, China). All materials were used without further purification.

Preparation of hybrids

The TPU/GW-622 and TPU/GW-944 hybrids were prepared by using the following procedure and conditions: (1) The as-received TPU was dried in a cabinet dryer at 110°C for 2 h. (2) The dried TPU was kneaded on a two roll mill at 150°C for 3 min. Then GW-622 or GW-944 powders was added in the TPU/GW-622 or TPU/GW-944 mass ratios of 100/0, 100/30, 100/50, 100/70, and 100/100, respectively. These mixtures were then kneaded on the two roll mill at 150°C for 5 min to prepare the binary hybrids. (3) To get samples for characterization, the hybrids were melted for 3 min, hot-pressed at 170°C for 10 min under the pressure of 15 MPa, and then cool-pressed at room temperature for 15 min under the pressure of 15 MPa.

Characterization

Differential scanning calorimetry (DSC) measurements were performed on a DSC 204F1 calorimeter made by NETZSCH Co., Germany. The DSC curves were recorded from −100 to +220°C at a heating rate of 10°C/min.

Scanning electron microscopy (SEM) images were taken of the representative fractured surfaces of TPU/GW-622 and TPU/GW-944 hybrids by using an XL-30 field emission scanning electron microscope made by FEI Co., USA. The SEM specimens were prepared by brittle fracturing of hybrid specimens in liquid nitrogen.

Dynamic viscoelasticity measurements were carried out on a dynamic mechanical analyzer (DMA VA3000) made by 01dB-METRAVIB Co., France. The specimens were 20 mm long, 6 mm wide, and around 1 mm thick. The temperature dependence of the loss factor tanδ was measured in the range −90 to +150°C at a frequency of 1 Hz and a heating rate of 3°C/min.

Tensile tests of the TPU/GW-622 and TPU/GW-944 hybrids were conducted according to ASTM standard (D412; Dumbbell-shaped), and the specimens were tested on an LRX Plus Tensile Tester made by Lloyd Instruments, Ltd., UK.

RESULTS AND DISCUSSION

TPU/GW-622 system

The representative fractured surfaces of the prepared TPU/GW-622 (100/50) hybrids are shown in Figure 2. The fractured surfaces of the hybrids are quite sleek, and no small molecule particle is exhibited. In TEM micrographs (not shown), a homogeneous structure with no dispersed particles was seen. This result suggests that the melt blending and hot-pressing above the melting temperature of GW-622 have well dispersed GW-622 into TPU, and that GW-622 exist in TPU mainly as molecules. Intriguingly, subsequent cooling did not cause any evident phase separation, probably because of the strong intermolecular interactions between GW-622 and TPU. Subsequent DSC and DMA results confirmed this viewpoint.

Figure 3 displays the DSC curves of the TPU/GW-622 hybrids with different TPU/GW-622 mass ratios. The curves for GW-622 and pure TPU are also included for comparison purposes. As shown in Figure 3, the as-received GW-622 powder was amor-
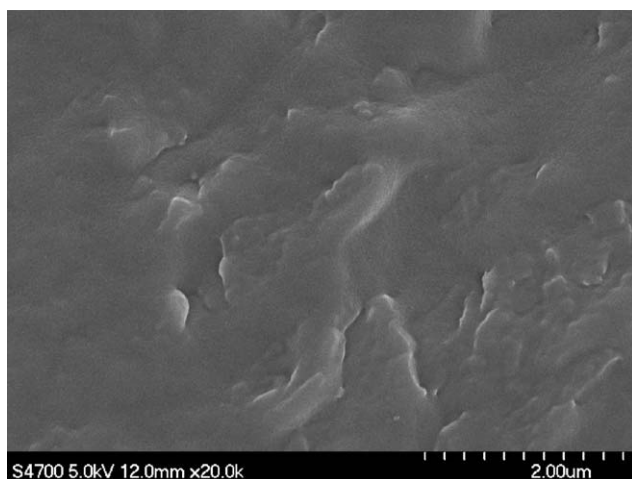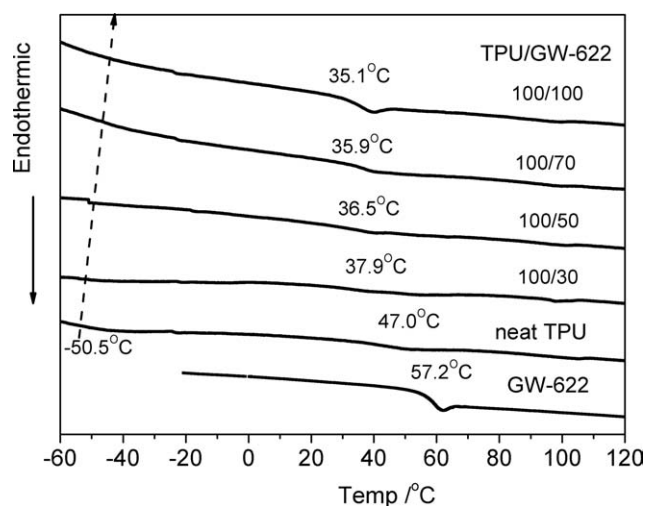
Figure 3 DSC curves of GW-622, neat TPU, and TPU/GW-622 hybrids with various TPU/GW-622 mass ratios.

phous and had the glass transition temperature at 57.2°C. The as-received TPU had two glass transition temperatures at around −50.5°C and 47.0°C, corresponding to the glass transition of the soft segments (denoted by T_{gs}) and hard segments (denoted by T_{gh}) in the TPU. Like the as-received TPU, TPU/GW-622 hybrids also show two glass transition temperatures, corresponding to T_{gs} and T_{gh} of the TPU. However, with increasing content of GW-622 in the hybrids, the T_{gs} of TPU/GW-622 hybrids increased slightly, whereas the T_{gh} of TPU/GW-622 prominently shifted from 47.0 to 35.1°C. Moreover, the glass transition peak of amorphous GW-622 did not appear in the curves. DSC results suggest that GW-622 molecules are mainly dispersed (more accurately, dissolved at a molecular level) into the hard phase of TPU through melt blending and formed strong intramolecular interactions with the hard segments, just as expected. This was probably because GW-622 containing ─OH, ─COO─, and amido groups possessed higher polarity and produced strong interactions with ─NHCOO─ groups of the hard segments of TPU.

As is well known, the hard phase of TPU consists of both ordered and disordered structures.[15] Naturally and reasonably, the ordered structure of the hard phase will elevate T_{gh}. The hard phase will likewise increase the T_{gs} of the soft phase of TPU because the soft segments and hard segments in the two phases are chemically linked together. With the increase of GW-622 loading, the hard phase of TPU is extended because GW-622 is mainly dispersed in the hard phase. However, the intermolecular interactions between GW-622 and the hard segments of TPU might destroy the ordered region of the hard phase to some extent and transform some segments from the ordered region into a disordered
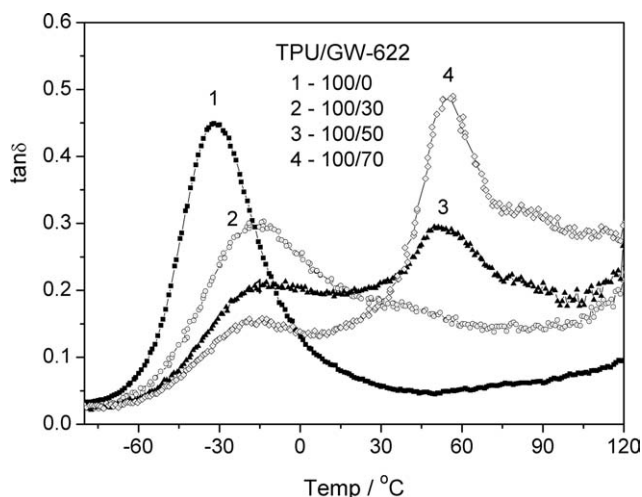
Figure 2 Representative SEM images of fractured surfaces of TPU/GW-622 (100/50) hybrids.

Figure 4 Temperature dependence of loss tangent (tanδ) values for TPU/GW-622 hybrids with various mass ratios.

aggregating state because of the disturbance effect (or impurity effect) of GW-622. Hence, the disordered structure in hard phase would expand. As a result, the T_{gh} of TPU/GW-622 hybrids decreased by about 12°C and the T_{gs} of TPU/GW-622 slightly rose. To further clarify the interaction between hindered amine and TPU and its effect on phase structure, DMA was employed.

Figure 4 shows the temperature dependence of the loss tangent (tanδ) values of the prepared TPU/GW-622 hybrids with various mass ratios of TPU/GW-622. It can be seen that the neat TPU matrix exhibited only one relaxation transition at about −30°C. This relaxation was the glass transition of the soft segments. However, no apparent peak corresponding to the glass transition of the hard phase appeared in the curve as a result of the predominant effect of the ordered structure in the hard phase. In fact, tanδ of the neat TPU slightly and gradually increases as the temperature increases from 50°C. This result could be ascribed to the glass transition of hard phase. There was an evident uptrend in the curve when the temperature approaches 100°C owing to the destruction of the ordered structure of the hard phase. Compared with the neat TPU, all the TPU/GW-622 hybrids showed very broad overlapping double tanδ peaks (about 120°C wide). The low temperatures peak was related to the glass transition of the soft region and the high temperature peak (novel peak) corresponded to the glass transition of the hard phase. Moreover, with the increase of GW-622 loading, the T_g of the soft segments increased slightly and the peak value of the soft phase transition was lowered. At the same time, the glass transition of the hard phase became more and more distinct and strong. DMA results were consistent with DSC results. When GW-622 was incorporated into the TPU matrix, it was selectively dis-

persed in the hard phase of TPU at the molecular level and strongly interacted with the hard segments of TPU. As a result, the ordered structure was destroyed, the ratio of disordered structure to ordered structure in the hard phase remarkably increased, and the total amount of the hard phase also markedly increased. Therefore, the glass transition peak became stronger and stronger. However, the volume content of the soft phase decreased and the content of the hard phase increased, upon which the loss peaks value of the low temperature peak of the TPU/GW-622 hybrids decreased, but the T_g of the soft segments increased slightly. Thus, such a design of TPU/hindered amine hybrid greatly expands the effective temperature range for damping of material.

As is well known, the area under the peak from the tanδ versus temperature curve, which is abbreviated as TA, could be considered to be a damping index. It is a measure of the energy dissipation of a transition process.[16] The data shown in Table I are the loss peak area in Figure 4 from −80°C to +120°C. It can be seen that the TA value increased from 19.0 to 36.4 when the GW-622 amount in the hybrids increased from 0 phr to 70 phr. In other words, the friction in the hybrids system was increased and the damping properties of TPU hybrids were improved greatly with increasing GW-622 amount.

Figure 5 shows the temperature dependence of storage modulus (E') for TPU/GW-622 hybrids and the neat TPU. All of the storage modulus curves display two transitions. In the glass transition region, every hybrid had a storage modulus higher than that of the matrix because the low-molecular-weight molecules (GW-622) behaved as rigid inclusions when the temperature was lower than the T_g of GW-622. In the rubbery region, the E' of the hybrids kept on declining from the glass transition region, and the typical plateau region didn't exhibit in Figure 5 because of the overlapping of the two transitions. As discussed before, the small molecules GW-622 mainly dispersed into the hard phase and formed the strong intermolecular interactions with the hard segments of the matrix. In other words, the total amount of hard phase increased with increasing amount of GW-622 in the hybrids.

TABLE I
Peak Area of TPU/GW-622 Hybrids

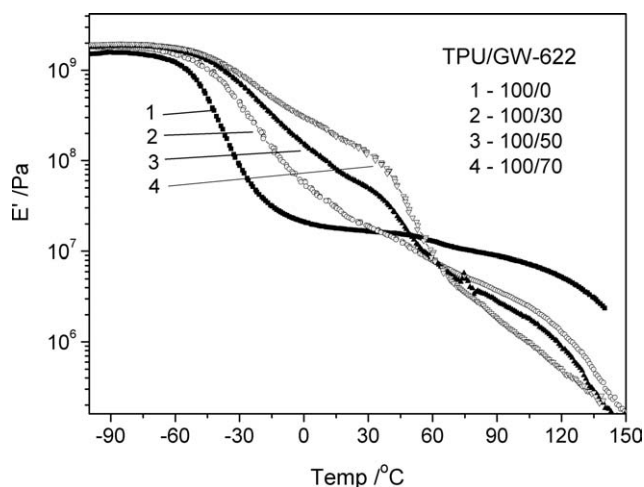| Samples	Location of first peak/°C	Location of second peak/°C	Temp. range/°C	TA
100/0	−31.4	–	−80–120	19.0
100/30	−14.6	37.6	−80–120	28.9
100/50	−13.9	50.9	−80–120	31.6
100/70	−13.9	54.7	−80–120	36.4

Figure 5 Temperature dependence of storage modulus (E') for TPU/GW-622 hybrids with various mass ratios.

Hence, at the low temperature end of the rubbery region, every TPU/GW-622 hybrid had a higher E' than that of the neat TPU', and E' increased gradually with increasing amount of GW-622 in the hybrid. However, at the high temperature end of the rubbery region, the E' decreased significantly with increasing amount of GW-622 in the hybrid with the beginning of the glass transition determined by the interaction between GW-622 and the hard segments.

As mentioned above, while the addition of GW-622 increased the total amount of hard phase of TPU, the ordered structure of hard segments was destroyed to a certain extent. Hence, the thermal stability of the hard phase, which decided the thermal stability of TPU, became poorer and poorer. In addition, at temperatures above the T_g of GW-622, the interaction between GW-622 and hard segments was destroyed, and GW-622 behaved as a plasticizer for TPU. Consequently, the dynamic modulus of TPU was lowered by GW-622.

Table II summarizes the mechanical properties of TPU/GW-622 hybrids. TPU is a good self-assembly block copolymer with self-reinforcing capability: the hard phase provides reinforcement to the soft phase. Therefore, TPUs usually have excellent tensile strength. However, the tensile stress and tear resistance of the hybrids were much lower than those of

TABLE II
Mechanical Properties of TPU/GW-622 Hybrids

Properties	Loading of GW-622/phr			
	0	30	50	70
Hardness (Shore A)	85	87	93	95
Tensile strength/MPa	36.7	15.2	11.9	9.2
Modulus at 100%/MPa	5.7	5.6	6.4	8.2
Elongation at break/%	550	720	754	430
Tear resistance/kN·m^{-1}	94.5	76.5	79.9	86.1

the neat TPU. These results further support the interpretation that GW-622 is mainly dispersed in the hard phase of TPU and forms intermolecular interactions with the hard segments of the matrix. When GW-622 is mainly dispersed in the hard segments, the ordered structure of the hard phase is destroyed to some extent, and the volume content of the ordered structure in the hybrids is reduced. As a result, the tensile strength and tear resistance of the hybrids decreased clearly with increasing GW-622 amount in the hybrids.

TPU/GW-944 system

As shown in Figure 1, GW-944 contains more amine groups than GW-622, and has a stronger interaction with the hard phase of TPU.

Figure 6 shows the SEM photographs of the representative surfaces of TPU/GW-944 (100/50) hybrids. The TPU/GW-944 hybrids system is similar to the TPU/GW-622 hybrids system. Very few voids/holes can be identified by the SEM photographs and no GW-944 particle is observed from Figure 5. Moreover, the TPU/GW-944 hybrids samples are quite transparent, suggesting that GW-944 might well have dissolved in the matrix, just like GW-622.

Figure 7 shows the DSC curves for the TPU/GW-944 hybrids with various TPU/GW-944 mass ratios. The curves for GW-944 and pure TPU are also included for comparison purposes. As shown in Figure 7, the as-received GW-944 powder is amorphous and has a glass transition temperature of 87.8°C. Compared with TPU/GW-622 hybrids, TPU/GW-944 hybrids do not show distinct information on the glass transition of the two phases of TPU. Moreover, the glass transition peak of amorphous GW-944 does not appear in any of the curves, implying that GW-944 might have dissolved in the TPU matrix to form
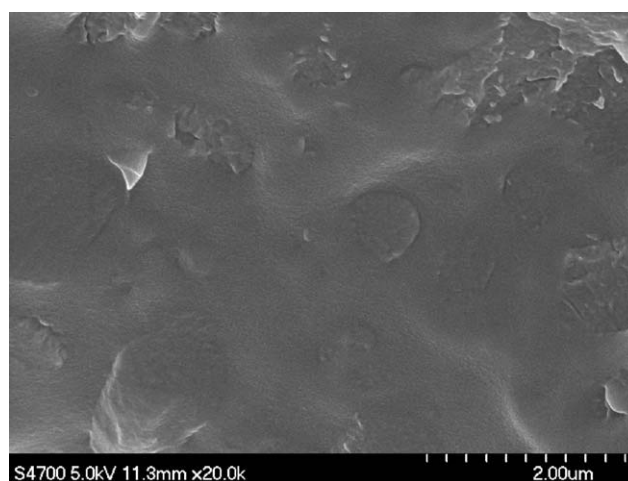

Figure 6 Representative SEM images of fractured surfaces of TPU/GW-944 (100/50) hybrids.
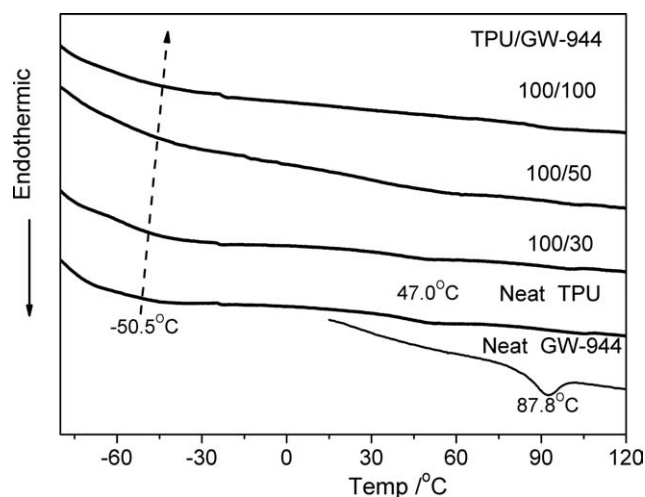
Figure 7 DSC curves of GW-944, neat TPU, and TPU/GW-944 hybrids with various TPU/GW-944 mass ratios.

a molecular hybrid, which markedly weakens the phase characteristics of TPU. This assumption is consistent with the interpretation of a high interaction between GW-944 and TPU. To clarify the phase structure and intermolecular interaction, DMA was employed and the results are shown in Figure 8.

The temperature dependence of tanδ of TPU/GW-944 hybrids with various TPU/GW-944 mass ratios clearly demonstrates two transition peaks. As the content of GW-944 increases, the first peak diminishes gradually and shifts to a higher temperature. On the opposite, the second peak rises sharply and shifts to a lower temperature. The first peak could be attributed to the glass transition of the soft segments of TPU/GW-944 hybrids. The second peak should mainly correspond to the thermal decay of the hard phase, which is affected by the thermal stability of the ordered structure, glass transition of the disordered hard segments, and the degree of phase
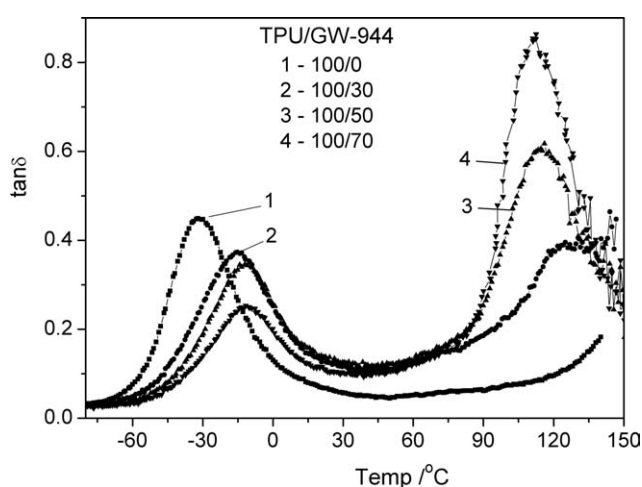

Figure 8 Temperature dependence of loss tangent (tanδ) values for TPU/GW-944 hybrids with various mass ratios.

separation of the soft phase and hard phase of TPU. Comparing Figure 4 with Figure 8 and the chemical structure of GW-622 with that of GW-944, we can draw the conclusion that GW-944 molecules have stronger interactions with the hard segments of TPU. As a result, GW-944 molecules are more concentrated in the hard phase, and disturb the ordered structure of the hard phase and lower the thermal stability of the hard phase to a larger extent. Since the glass transition temperature of GW-944 is 87.8°C, which is much higher than T_{gh} of TPU and near the dissociation temperature of TPU, the second peak of TPU/GW-944 hybrids is probably the comprehensive reflection of increased T_{gh} and decreased dissociation temperature, both caused by the incorporation of GW-944. With the increase of GW-944 loading, more and more ordered structure was destroyed, the ratio of disordered structure in the hard phase increased significantly, and the total amount of the hard phase also markedly increased. Therefore, the second transition peak became stronger and stronger. However, the volume content of the soft phase decreased and the volume content of the hard phase increased. Consequently, the first loss peak value of TPU/GW-944 hybrids decreased because of the volume effect, but the T_g of the soft segments moved to a slightly higher temperature.

The TA values of these TPU/GW-944 samples were also calculated and the data were summarized in Table III. The damping index for TPU/GW-622 hybrids, TPU/GW-944 hybrids was 49.6, a big improvement from the value of 19.0 for TPU/GW-622 hybrids, and was three times higher than that for the TPU matrix. Thus, the GW-944 molecules also improved the damping property of the TPU matrix, especially at high temperatures. However, the first transition peak and second transition peak were farther apart for TPU/GW-944 hybrids than for TPU/GW-622 hybrids, an unsatisfactory damping characteristic for application purposes.

The temperature dependence of E' for TPU/GW-944 hybrids and the neat TPU is shown in Figure 9. All the storage modulus curves basically display two transitions. The first transition is associated with the glass transition of the soft segments of TPU and

TABLE III
Peak Area of TPU/GW-944 Hybrids

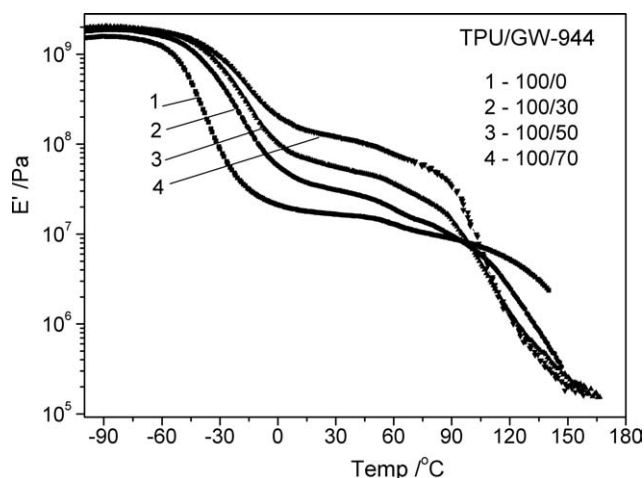| TPU/GW-944	First transition		Second transition		
	$T_g/°C$	TA	$T_g/°C$	TA	ΣTA
100/0	−32.1	19.0	–	–	19.0
100/30	−15.1	17.1	123.2	14.1	31.2
100/50	−12.4	15.3	114.3	29.1	44.4
100/70	−11.8	11.5	112.1	38.1	49.6

Figure 9 Temperature dependence of storage modulus (E') for TPU/GW-944 hybrids with various mass ratios.

the T_g increases with increasing GW-944 content. The second transition is assigned to the dissociation of the intermolecular interactions between GW-944 molecules and the hard segments of TPU. At temperatures lower than about 100°C, the E' of TPU/ GW-944 hybrids increases gradually when the addition amount of GW-944 increases from 0 phr to 70 phr. However, at temperatures higher than 100°C, the E' of TPU/GW-944 hybrids decreases greatly with the increasing temperature and GW-944 content in the hybrids. Similar to the case of TPU/GW-622 hybrids, the GW-944 molecules in TPU/GW-944 are mainly dispersed in and form strong intermolecular interactions with the hard segments of TPU. Hence, the E' of TPU/GW-944 hybrids increases gradually at low temperature. But at the high temperatures of the rubbery region, the addition of GW-944 molecules affects both the order of TPU's hard segments and the thermal stability of the hybrids, and the E' decreases significantly with increasing amount of GW-944 in the hybrids. Because GW-944 has a higher T_g than GW-622, the rubbery plateau of TPU/GW-944 hybrid is longer than that of TPU/ GW-622.

Table IV summarizes the mechanical properties of the TPU/GW-944 hybrids. As expected, GW-944 has a more significant effect on the tensile strength of the

hybrids than GW-622. The values of tensile strength, elongation at break, and modulus at 100% of the TPU/GW-944 hybrids are much lower than the corresponding values of the neat TPU. Table IV also lists the hardness and tear resistance of the TPU/GW-944 hybrids. It is clear that the mechanical properties decrease sharply with the addition of GW-944 molecules. The ordered structure of the hard phase in TPU is a highly effective source of strength of the material. In other words, crosslinks slows down chain relaxation during extension.[16] However, GW-944, which mainly stays in the hard phase, destroys the ordered structure of the hard segments. So the volume of the hard phase increases, whereas the relative volume of ordered structure decreases. The interaction effect is much stronger than the volume effect. The evolution of mechanical performance of TPU with GW-944 incorporation confirms the interpretation that GW-944 selectively interacts with and is dispersed in the hard phase of TPU.

CONCLUSIONS

Novel TPU/hindered amine organic hybrids were prepared by blending TPU with two hindered amines: GW-622 and GW-944. The structure and dynamic mechanical properties of the organic hybrids were investigated. The results showed that GW-622 and GW-944 mainly interacted with the hard segments of TPU and were selectively concentrated in the hard phase of TPU. GW-944 had stronger interaction with the hard segments of TPU than GW-622. As a result, the neat TPU matrix had only one glass transition peak, whereas the TPU/GW-622 hybrids exhibited two overlapping glass transition loss peaks, which were corresponding to the glass transition of the soft phase and hard phase, respectively. The TPU/GW-944 hybrids also demonstrated two evident loss peaks. However, the second peak of TPU/GW-944 hybrids was believed to be the comprehensive reflection of increased T_{gh} and decreased dissociation temperature, both caused by the incorporation of GW-944. The TA values of two hybrids greatly increased when the GW-622 or GW-944 amount in the hybrids increased from 0 phr to

TABLE IV
Mechanical Properties of TPU/GW-944 Hybrids

Properties	Loadings of GW-944/phr			
	0	30	50	70
Hardness (Shore A)	85	89	92	94
Tensile stress/MPa	36.7	11.3	6.5	5.4
Modulus at 100%/MPa	5.7	7.4	6.4	4.9
Elongation at break/%	550	699	249	103
Tear resistance/kN·m^{-1}	94.5	90.5	45.9	34.1

70 phr. This increase demonstrated their advantages in wide frequency range damping. The static mechanical properties of these two hybrids were greatly decreased because the addition of GW-622 and GW-944 affected the ordered structure of the hard phase of TPU. However, the properties were still good enough for many engineering applications. Therefore, the TPU/hindered amine organic hybrid is a potentially new damping material.

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