

Correlation Between Mechanical Damping and Interphase Content in Interpenetrating Polymer Networks

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ABSTRACT: Recently, some interpenetrating polymer networks with good mechanical damping properties have been synthesized. However, the effect of morphology on this property has not yet been clearly elucidated. Herein, two polystyrene–polyurethane interpenetrating polymer networks, which were grafted using TMI [benzene-1-(1-isocyanato-1-methyl ethyl)-3-(1-methylenyl)] and HEMA (2-hydroxyethyl methacrylate), respectively, have been investigated, as model samples, by modulated-temperature differential scanning calorimetry and by dynamical mechanical thermal analysis. The results indicate that there is a correlation between mechanical damping and both interphase content and the distribution of composition in the interphase region. The findings should provide valuable information for the design of future damping materials. © 2001 John Wiley & Sons, Inc. *J Appl Polym Sci* 81: 2439–2442, 2001

Key words: interpenetrating polymer networks; modulated DSC; interphase; mechanical damping

INTRODUCTION

Interpenetrating polymer networks (IPNs) as damping materials have been studied for more than 30 years.^{1,2} A fundamental phenomenon associated with nearly all IPNs is phase separation. Although a huge effort has been made,^{3–8} the effect of morphology on the mechanical damping of IPNs has not yet been clearly understood. Until recently,^{9,10} IPN interphase content could not be analyzed quantitatively. Now, the ability to determine the interphase content in IPNs quantitatively provides a new opportunity for understanding the effect of interphase content on IPN damping properties.

The interphase content and mechanical damping of polystyrene (PS)–polyurethane (PU) IPNs

grafted using benzene-1-(1-isocyanato-1-methyl ethyl)-3-(1-methylenyl) (TMI) and 2-hydroxyethyl methacrylate (HEMA) IPNs were investigated by modulated-temperature differential scanning calorimetry (M-TDSC) and dynamical mechanical thermal analysis (DMTA), respectively. The correlation between mechanical damping and interphase content was then analyzed.

EXPERIMENTAL

PU–PS IPN Preparation

The preparation details for the two PU–PS (60:40 by weight) IPN samples with TMI and HEMA as inter-network grafting agent, respectively, are as follows.

The PU components consisted of 1,1,3,3-tetramethylxylene diisocyanate (m-TMXDI, kindly donated by Cytec Industries, UK), a polyoxypropylene glycol with a molar mass of 1025

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(PPG1025, BDH, UK) and the crosslinker, trimethylol propane (Aldrich, UK). TMI (Aldrich, UK) and HEMA (Aldrich, UK) were incorporated singly as grafting agents. Stannous octoate (Sigma, UK) was used as the PU catalyst. The other monomer used was styrene (Aldrich, UK). The free radical reaction was initiated with azoisobutyronitrile (Aldrich, UK). The required amount of azoisobutyronitrile was dissolved in the monomer (styrene). In a separate vessel, the triol (trimethylol propane) was dissolved in the PPG1025 at 60°C. Both components were combined at room temperature and the polyurethane catalyst and TMI (or HEMA) were added. A nitrogen blanket was applied. On addition of the TMXDI, the components were mixed for 5 min at high speed. Degassing for 1 min under vacuum was conducted to remove any entrapped air. The mixture was cast into stainless-steel spring-loaded O-ring molds, which had been pretreated with CIL Release 1771 E release agent. The curing cycle consisted of three stages of 24 h each at 60, 80, and 90°C.

M-TDSC

A TA Instruments M-TDSC (2920) calorimeter was used. An oscillation amplitude of $\pm 1.0^\circ\text{C}$, an oscillation period of 60 s, and a heating rate of 3°C/min were used. The calorimeter was calibrated as for a conventional differential scanning calorimeter with a standard indium sample.

DMTA

Dynamic mechanical measurements were performed with a Polymer Laboratories MK II Dy-

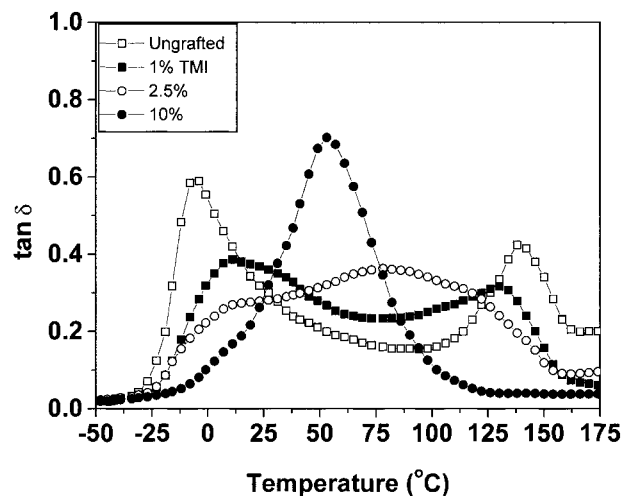


Figure 1 Tan δ versus temperature plots for the PU-PS IPNs with different TMI contents.

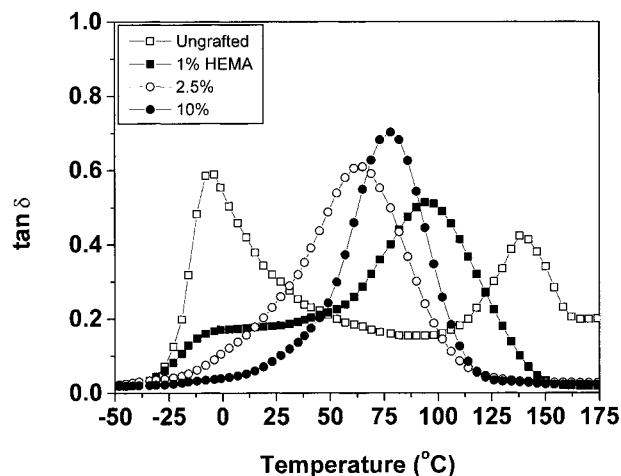


Figure 2 Tan δ versus temperature plots for the PU-PS IPNs with different HEMA contents.

amic Mechanical Analyser. Three samples were measured from -60 to 200°C in the bending mode (single cantilever) at a fixed frequency of 10 Hz using a heating ramp of 3°C/min. The test specimens were cut to a rectangular shape of about 50 mm in length, 10 mm in width, and 3 mm in thickness. The applied strain setting was $\times 4$.

RESULTS AND DISCUSSION

Figures 1 and 2 show tan δ versus temperature plots for the PS-PU (40:60 by weight) ungrafted and grafted (TMI and HEMA, respectively) IPNs. For the PS-PU IPNs with 10 wt % of TMI and with 2.5 wt % of HEMA, a broad single tan δ transition peak was shown in both cases. The PS-PU IPNs with 10 wt % of HEMA showed a relatively narrow tan δ peak. These DMTA results indicated that with increasing degree of grafting, the miscibility increased. The results also indicated that the effect of TMI on morphology of the PS-PU IPN was different from that of HEMA. The PS-PU IPN with 2.5 wt % of TMI showed good mechanical damping over a wide temperature range.

Recently, the application of the differential of heat capacity, dC_p/dT , versus temperature signal from M-TDSC to the characterization of the phase structure of IPNs⁹⁻¹¹ has been developed. Herein, this dC_p/dT signal was again used to analyze these PU-PS IPNs.

Figure 3 shows the dC_p/dT versus temperature signals for the PS-PU IPNs grafted using 1 wt %

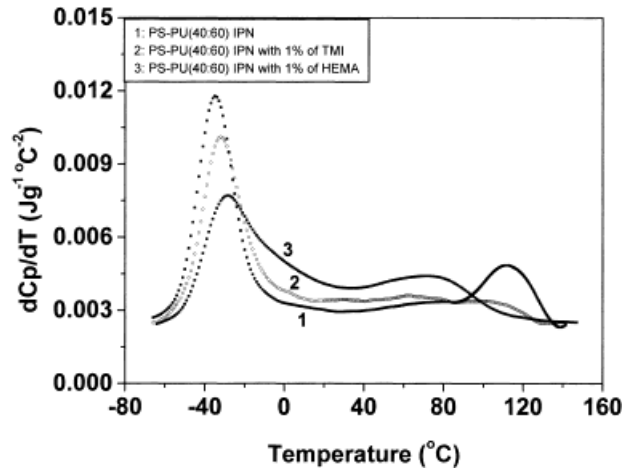


Figure 3 dC_p/dT versus temperature plots for the PU-PS IPN with 1 wt % of HEMA and with 1 wt % of TMI and the ungrafted PU-PS IPN.

of TMI and 1 wt % HEMA and the corresponding ungrafted PS-PU IPN. Figure 4 shows the same results for the PS-PU IPNs grafted using 2.5 wt % of TMI and 2.5 wt % HEMA and ungrafted PS-PU IPN. Figure 5 shows the equivalent data for the PS-PU IPNs grafted using 10 wt % of TMI, and 10 wt % HEMA, and again, the ungrafted PS-PU IPN.

Comparing the dC_p/dT signals of the PU-PS IPNs with 1, 2.5, and 10 wt % of TMI, with 1, 2.5, and 10 wt % of HEMA, and with the PU-PS IPN, it was found that the dC_p/dT signals for these grafted IPNs were quite different from those of the ungrafted PS-PU IPN. Between the glass

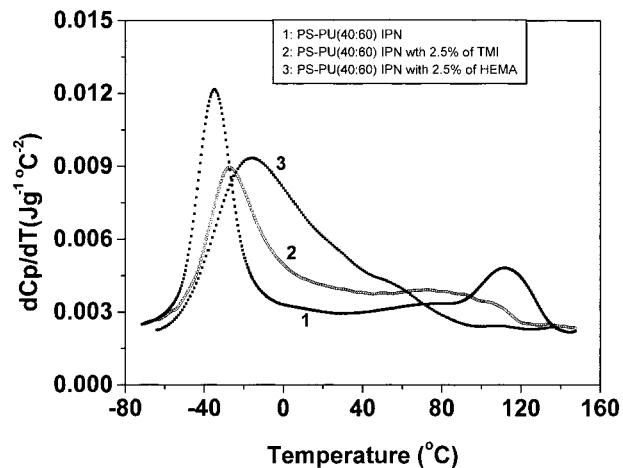


Figure 4 dC_p/dT versus temperature plots for the PU-PS IPN with 2.5 wt % of HEMA and with 2.5 wt % of TMI and the ungrafted PU-PS IPN.

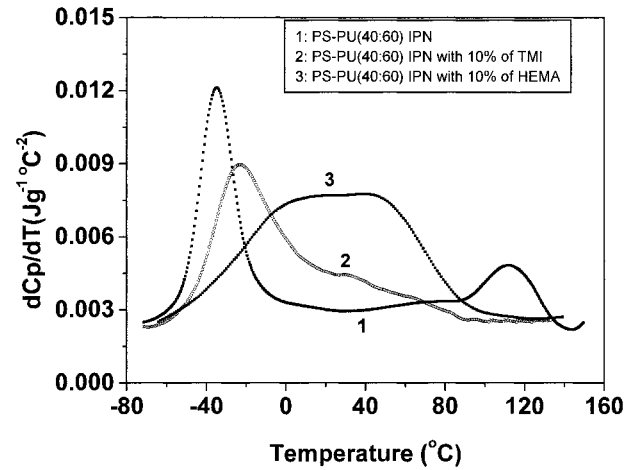


Figure 5 dC_p/dT versus temperature plots for the PU-PS IPN with 10 wt % of HEMA and with 10 wt % of TMI and the PU-PS IPN.

transition temperatures of the PU and PS phases, the values of dC_p/dT signals for these grafted IPNs are larger than those of the ungrafted case. This indicates there is higher interphase content in these grafted IPNs than is the case for the ungrafted materials.

From Figures 3 to 5, it is clear that the interphase content in the PU-PS IPNs with HEMA is higher than in the TMI-grafted IPNs. Grafting with HEMA results in higher degrees of mixing between the PS and the PU components than does TMI grafting.

Comparing DMTA data with M-TDSC results for the PU-PS IPNs with 1 and 2.5 wt % of TMI (Figs. 1 and 6), it is observed that the interphase content influences the mechanical damping. The interphase content in the PU-PS IPN with 2.5 wt % of TMI is higher than in the 1 wt % equivalent sample. This higher interphase content results in better mechanical damping in that it has a wider range of temperature over which $\tan \delta$ exceeds 0.3. This is a value often taken as an index of good damping performance. However, from the results shown in Figures 3 and 4, although there is high interphase content in the PU-PS IPNs with HEMA, this did not result in as good damping. Therefore, it seems that a high interphase content alone does not guarantee good mechanical damping.

We now reconsider the M-TDSC results shown in Figure 4. Although there is a high interphase content in the PU-PS IPN with 2.5 wt % of HEMA, the interphase region spanned a much narrower temperature range than was the case

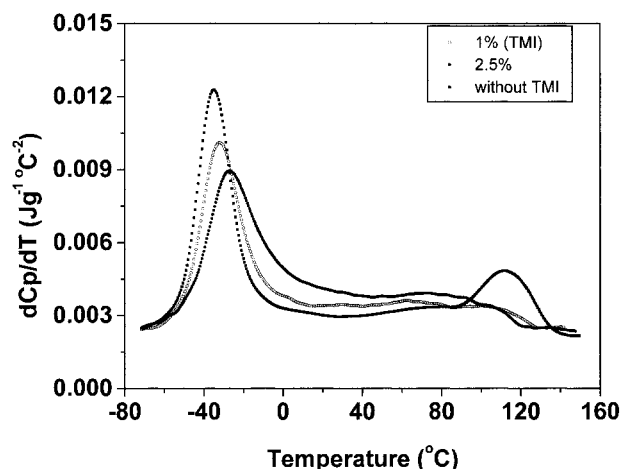


Figure 6 dC_p/dT versus temperature plots for the PU-PS IPN with 1 and 2.5 wt % of TMI and the ungrafted PU-PS IPN.

for the 2.5 wt % TMI grafted IPN. It is concluded that a broad interphase region results in good mechanical damping and that the interphase content alone does not control mechanical damping in IPNs.

CONCLUSIONS

There is a correlation between mechanical damping and both the interphase content and the broadness of the transition region ascribed to the interphase in IPNs. Neither high nor low interphase content resulted in good mechanical damp-

ing. Only an intermediate interphase content and a broad interphase transition region resulted in good mechanical damping. Thus, the design of IPNs for damping applications not only needs to consider interphase content, but also must consider the interphase composition distribution.

REFERENCES

1. Kim, S. C.; Sperling, L. H. *Polym Adv Technol* 1996, 7, 195.
2. Hu, R.; Dimonie, V. L.; ElAasser, M. S.; Pearson, R. A.; Hiltner, A.; Mylonakis, S. G.; Sperling, L. H. *J Polym Sci Polym Phys* 1997, 35, 1501.
3. Sperling, L. H.; Mishra, V. *Polym Adv Technol* 1996, 7, 197.
4. Lipatov, Y. S.; Alekseeva, T. T. *Polym Adv Technol* 1996, 7, 234.
5. Nemirovski, N.; Silverstein, M. S.; Narkis, M. *Polym Adv Technol* 1996, 7, 247.
6. Hourston, D. J.; Schafer, F. *Polym Adv Technol* 1996, 7, 273.
7. Han, X.; Chen, B.; Guo, F. *Polym Adv Technol* 1996, 7, 315.
8. Klempler, D.; Sperling, L. H.; Utracki, L. A. *Interpenetrating Polymer Networks*; American Chemical Society: Washington, DC, 1994.
9. Song, M.; Hourston, D. J.; Schafer, F.-U.; Pollock, H. M.; Hammiche, A. *Thermochim Acta* 1998, 315, 25.
10. Hourston, D. J.; Song, M.; Schafer, F.-U.; Pollock, H. M.; Hammiche, A. *Polymer* 1999, 40, 4769.
11. Hourston, D. J.; Song, M. *J Appl Polym Sci* 2000, 76, 1791.1. Kim, S. C.; Sperling, L. H. *Polym Adv Technol* 1996, 7, 195.