ENERGY ABSORPTION OF POLYURETHANE-BASED POLYMER ALLOYS

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**Abstract**: Interpenetrating polymer networks (IPNs), elastomers and foams composed of polyurethanes and epoxies were prepared by the simultaneous technique. The effect of platelet geometry fillers (e.g. graphite, mica, glass) and cure time on the morphology of the IPNs was investigated. Fillers were incorporated by random batch mixing and the rubber roll mill technique. It was determined that low level of filler resulted in a small increase of tan δ and as the filler level increased, tan δ decreased. Samples with low post-curing times showed higher magnitudes of tan δ than samples with longer post-curing time. IPN foams...
with different ratios of polyurethane and epoxy were prepared and their sound absorption properties were measured by the impedance tube method. Different PU/Epoxy ratios showed different maximum sound absorption frequency ranges. In general, the IPN foams prepared showed higher sound absorption properties than the GM polyurethane foam reference.
Interim Technical Report

ENERGY ABSORPTION OF POLYURETHANE BASED POLYMER ALLOYS

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Polymer Institute
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TABLE OF CONTENTS

I. INTRODUCTION AND OBJECTIVES 1

II. EXPERIMENTAL 1
   A. Materials 1
   B. Preparation of Samples 1
      1. IPN Elastomers 1
      2. IPN Foams 2
   C. Testing 2
      1. Dynamic Mechanical Spectroscopy 2
      2. Standing Wave Apparatus (Bruel & Kjaer, Type 4002) For Sound Absorption Measurement 3
      3. Density 5

III. RESULTS AND DISCUSSION 5
    A. Effect of Fillers on IPN Elastomers 5
    B. Effect of Post-Curing Time 7
    C. Foams 8

IV. FUTURE WORK 8

Tables 10-12
Figures 13-44
I. INTRODUCTION AND OBJECTIVES

The objectives of this study are to develop interpenetrating polymer network (IPN) elastomers and foams which exhibit good sound attenuation characteristics over a broad frequency and temperature range. In this study the effect of various fillers with platelet geometry, such as graphite and mica, were investigated. The platelet geometry of the fillers is believed to encourage energy dissipation via shear contact of the planar particles. The fillers were incorporated into IPN elastomers by random batch-mixing and rubber milling. The effect of post-curing on the dynamic mechanical properties (tanδ) of IPN elastomers was also studied. IPN foams with different polyurethane/epoxy ratios were prepared and the sound absorption was measured by the impedance tube method.

II. EXPERIMENTAL

A. Materials

The materials used in this study are summarized in Table I. The polyols, chain extender, and epoxy resin were degassed under vacuum at 70°C for 24 hours. The other chemicals were used as received from the manufacturers.

B. Preparation of Samples

1. IPN Elastomers

The IPN elastomers based on polyurethane and epoxy were prepared by the simultaneous polymerization technique. One component contained the isocyanate (Isonate 143L) and epoxy resin (DER 330). The other component contained polyols (Niax 31-28), chain extender (Isonol 100), urethane catalyst...
(T-12), epoxy catalyst (BF$_3$-etherate) and various fillers.

The two components were mixed together for one minute at room temperature using a high speed mechanical stirrer. The mixture was then poured into a pre-heated mold and pressed on a laboratory platen press at 100°C. The samples were then removed from the press (after curing for 30 min.) and then post cured in an oven at 100°C for 16 hours. Samples were conditioned at 25°C and 50% relative humidity for at least three days prior to testing.

2. IPN Foams

IPN foams were prepared by the one shot, free rise method. Since difficulties were encountered in previous studies due to the slow reaction of the epoxy component at ambient temperature, the mixing temperature in this study was raised to 80°C. The Niax polyol 31-28 and epoxy resin DER 330 were first preheated to 80°C. The surfactants (DC-193, L-540), blowing agents (water, Freon 11A), chain extender (Isonol 100), polyurethane catalyst (T-12, A-1), and catalysts for epoxy resin (XU-213, DMP-30) were added and thoroughly mixed with a high speed stirrer for two minutes. The cream time, rise time and tack-free time were recorded. The foams were cured at 90°C for 16 hours and conditioned at 25°C and 50% relative humidity for at least three days prior to testing.

C. Testing

1. Dynamic Mechanical Spectroscopy

All dynamic mechanical measurements were conducted on a Rheovibron dynamic viscoelastometer, DDV II (Toyo Manufacturing Co.) at a scanning rate of 1 to 2°C per minute in the glass transition region or every 3 to 5°C per minute in the non-transition region. The specimens were in the form of rectangular films with dimensions of 2 cm in length, 0.1 cm in width, and
0.05 cm in thickness. The specimens were inserted into the chamber and cooled to -50°C where the measurement began. All tests were carried out at a frequency of 110 Hz.

2. Standing Wave Apparatus (Bruel & Kjaer, Type 4002) For Sound Absorption Measurement

This apparatus (Type 4002) is designed for easy and quick determination of the absorption coefficient of acoustic materials by the standing wave method. The advantages of the method are that only small circular samples, about 10 cm in diameter, are needed. The principal of the measurement method is shown in Figure A. The loudspeaker at one end of the tube is operated at the desired test frequency from an audio-frequency oscillator with 6 ohms output impedance and a distortion of less than 1% (B.F. Oscillator Type 1022). The sound waves move through the tube and strike the sample which is placed in a sample holder with a thick back plate, to avoid all sound absorption by the apparatus itself. The sound waves are then partly reflected at the sample. The resultant of the incident wave with amplitude 1 and reflected wave with amplitude r is a standing wave pattern with alternate sound maxima 1+r and minima 1-r in the tube. From the ratio n of these sound pressure maxima and minima the reflection coefficient r (see equation below) follows directly.

\[ r = \frac{n-1}{n+1} \]  

(1)

However, we are interested in the absorption coefficient \( \alpha \), i.e. the ratio of the energy absorbed by the sample to the incident energy. In other words, \( \alpha = 1 - r^2 \), from which, with the aid of the relation (1):

\[ \alpha = \frac{4}{n + \frac{1}{n} + 2} \]  

(2)
Figure A. The Standing Wave Apparatus Type 4002 mounted for low frequency in Deluxe Analyzer 2112.

Figure B. Relation between absorption coefficient of large rooms and tube apparatus.
The sound field is explored by means of a probe microphone, movable on a track equipped with a scale on which the exact distance between the probe entrance and test sample can be read. The microphone voltage is amplified by a selective amplifier to reduce the influence of hum and noise and higher harmonics, which are inevitably generated by the speaker in the tube. Particularly suitable for this purpose is the 1/3 Octave Analyzer 2112 with 33 fixed filters from 22 Hz to 45 kHz and three scales, 0-100%, 0-70%, and 0-30%.

The absorption coefficient is determined by the tube measurement method only at normal incidence, which is why the measured coefficients are generally somewhat smaller than those determined by the reverberation room method according to W.C. Sabine's formula. In Figure B a curve is shown which indicates the relationship between the results of the tube method and the reverberation room method.

3. Density

The density of foam samples was measured according to ASTM D-1622.

III. RESULTS AND DISCUSSION

A. Effect of Fillers on IPN Elastomers

The effect of various fillers on the morphology (\(\tan \delta\)) of IPN elastomers was investigated. The polyurethane/epoxy IPN (formulation #43, previous Report No. 1), 60/40 with 2% Isonol 100 as chain extender, was selected as a model formulation (labelled as formulation #1 in this report). This formulation was repeated to check the reproducibility and consistency of the preparation. The results are shown in Figures 1 and 2. Comparison of both figures indicates the reproducibility of previous work.
Since it is postulated that fillers with platelet geometry shall encourage energy dissipation via shear contacts of the planar particles, fillers with platelet structure such as graphite flake and mica flake were incorporated in various proportions in the PU/epoxy IPN.

Three different particle sizes of graphite flake (#1 -20 mesh to +80 mesh, #2 -50 mesh to +200 mesh, and #3 -80 mesh down) were investigated. Initially, the graphite filler (platelets) was incorporated into the PU/epoxy IPN by random batch mixing with a high speed stirrer and then pressing on a platen press. The dynamic mechanical spectra of three graphite filled IPNs (10%) are shown in Figures 3-5 and the comparison of tanδ of unfilled and graphite filled elastomers is shown in Figure 6. The result in Figure 6 shows a little increase in tanδ and a shift of Tg to about $10^0$C lower. When the amount of graphite filler was increased to 20% and 40% as shown in Figures 7 and 8, it was found that the tanδ magnitude decreased (Figure 9). An increase as well as a broadening was expected with this filler (studies at the University of Dayton showed these results, i.e. increase and broadening of tanδ with addition of graphite platelets).

The graphite filler was also incorporated into the IPN elastomer on a rubber mill. This technique was used so that the shear force between the rolls will align the platelets of the graphite filler in the same direction. This should facilitate energy dissipation. Twenty and forty percent of #2 graphite filler were incorporated into the IPN by this technique and their dynamic mechanical spectroscopy were measured and are shown in Figures 10 and 11. At 20% graphite filler there was no change of tanδ observed, however, at the 40% level the tanδ breadth decreased.

In the case of mica filler, the filler was incorporated into the IPN system with and without Isonol-100 chain extender. The same phenomenon was
was observed. As the amount of filler increased, the tanδ magnitude decreased (Figures 12 and 13). In the system with Isonol-100 chain extender, Figure 14, the tanδ did not show significant change.

Figures 15 and 16 show the dynamic mechanical spectra of the Dicaprl FP-1010 (hollow glass bubble filler) and Sundex 740T plasticizer filled IPN elastomers. At the 10% level, in both cases, there was no change of tanδ observed. The amount of Dicaprl filler and plasticizer will be increased in the next study.

B. The Effect of Post-Curing Time

The effect of post-curing was investigated by measuring the dynamic mechanical properties of the IPN elastomers after the samples were post-cured for different periods of time (e.g. 0, 2, 8, and 16 hours). The results are shown in Figures 17-20 and the tanδ's are compared in Figure 21. It was found that with no post-curing, the sample showed a high tanδ. However, as the post-curing time increased to 2, 8 and 16 hours, the tanδ magnitude decreased with the Tg shifted to higher temperatures as expected (\(\approx 100^\circ C\)). The high tanδ of the sample without post-curing or low post-curing time was probably due to the uncured epoxy as well as the polyurethane. The molecular chains are partially loosening and the small chain segments can move. An attempt was made to stabilize the uncured condition by immersing the uncured sample in methanol (to "kill" the reactive epoxy groups) for 3 hours and drying the sample under vacuum for 24 hours. The dynamic mechanical spectroscopy was taken and is shown in Figure 22. The tanδ was found to decrease to the same level as the sample post-cured for 8 hours. This area will be further investigated.
C. Foams

The IPN foams were prepared by mixing the preheated polyol and epoxy resin at 80°C. The high temperature was required in order to speed up the epoxy reaction. However, at this temperature, the evaporation rate of the blowing agent, Freon 11A, was too rapid and thus a second blowing agent, water, was necessary in order to obtain low density foams. Using water in the formulations required adjusting the amount of the catalysts, since problems with collapsing and shrinkage arose. It was observed that this foam system was sensitive to the amount of catalyst used. Efforts were made to formulate some samples by changing the percent water used and adjusting the amount of the catalysts for different polyurethane/epoxy (PU/epoxy) ratios.

IPN foams were prepared with PU/epoxy ratios of 70/30, 60/40, 50/50, 40/60, and 30/70. An isocyanate index of 105 was used in preparing these samples. Figures 23-28 show the percentage of sound absorption of foam containing 2% Isonol 100 as chain extender. In these formulations the urethane component contained excess Isonate 143L to react with the pendant hydroxyl group of the epoxy DER 330. Figures 29-31 represent the sound absorption of the foams without Isonol 100 and excluding the excess Isonate 143L to react with the epoxy. Figures 23 and 24 show the tested specimens cut out from the top and bottom of the prepared samples with a PU/epoxy ratio of 70/30. This was done to investigate how homogeneous the samples were in the direction of foam rise. It was suspected that concentration of the epoxy may tend to be more at the bottom of the prepared samples, since the unreacted epoxy may descend due to gravity. This phenomenon is more likely to happen while the foam is rising and gelling. Therefore, it is important to have balanced rates of reactions of both the polyurethane and the epoxy systems. The lower portion of the sample, Figure 24, shows about 10-15% lower sound absorption than the upper portion, Figure 23, at higher frequencies.
This, as mentioned above, could be due to more available hard segments (epoxy crosslinks) in the lower portion of the sample. In Figure 32, the polyurethane reaction was slowed down by applying less amounts of catalysts, A-1 and T-12. The curing effect on the percent sound absorption of a sample with a PU/epoxy ratio of 40/60 is shown in Figures 33 and 34. As can be seen from these figures, the cured specimen has a lower sound absorption capability over the test frequencies. This is most likely due to uncured epoxy containing a distribution of MW and therefore broader energy absorbing abilities (wider Temp. and frequency range). Figures 35 and 36 represent foams without the blowing agent, Freon 11A, at different levels of catalyst, DMP-30. The excess amount of DMP-30 promoted trimerization of Isonate 143L and gave a harder structure to the foam which resulted in lower percent sound absorption. Foams were prepared with a different type of blowing agent (dichloromethane) which has a higher boiling point than Freon 11A. Samples made with blowing agent from preheated (also not preheated) epoxy and Niax polyol show lower percent sound absorption. The test results for both preheated and not preheated samples are shown in Figures 37 and 38. The sample tested in Figure 28, due to closed and tight cell structure, showed poor sound absorbing properties. This demonstrates the important effect of cell structure of a foam on dissipation of energy and sound.

IV. FUTURE WORK

A. Different fillers and amounts of fillers and plasticizer will be further investigated.

B. Various types of fillers will be incorporated into the IPN foam and the sound attenuation properties will be studied.

C. Three-component IPNs will be studied.
Table I Materials

<table>
<thead>
<tr>
<th>Materials</th>
<th>Chemical Composition</th>
<th>Eq.Wt.</th>
<th>Supplier</th>
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<tr>
<td>Isonate 143L</td>
<td>Carbodiimide modified diphenylmethane diisocyanate</td>
<td>143</td>
<td>Dow Chem. Co.</td>
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<tr>
<td>Niax 31-28</td>
<td>Graft copolymer of poly(oxy-propylene)(oxyethylene) adduct of glycerol</td>
<td>2004.5</td>
<td>Union Carbide</td>
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<tr>
<td>Mondur MR</td>
<td>Polymeric isocyanate</td>
<td>133.3</td>
<td>Mobay</td>
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<tr>
<td>Isonol 100</td>
<td>N,N'-Bis(2-hydroxypropyl)aniline</td>
<td>104.5</td>
<td>Dow Chem. Co.</td>
</tr>
<tr>
<td>T-12</td>
<td>Dibutyltin dilaurate</td>
<td>-</td>
<td>M &amp; T Chem.</td>
</tr>
<tr>
<td>Niax A-1</td>
<td>70% Bis(2-dimethylaminoethyl) ether solution in dipropylene glycol</td>
<td></td>
<td>Union Carbide</td>
</tr>
<tr>
<td>BF&lt;sub&gt;3&lt;/sub&gt;(O&lt;sub&gt;2&lt;/sub&gt;CH&lt;sub&gt;3&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;</td>
<td>Boron triflorurine etherate</td>
<td></td>
<td>Eastman Chem.</td>
</tr>
<tr>
<td>DMP-30</td>
<td>2,4,6-Tris (dimethylaminomethyl) phenol</td>
<td></td>
<td>Rohm &amp; Haas</td>
</tr>
<tr>
<td>XU-213</td>
<td>BCl&lt;sub&gt;3&lt;/sub&gt;-Amine complex</td>
<td></td>
<td>Ciba-Geigy</td>
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<tr>
<td>Freon 11A</td>
<td>Trichlorofluoromethane</td>
<td></td>
<td>E.I. duPont</td>
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<td>DC 193</td>
<td>Silicone copolymer surfactant</td>
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<td>Hollow glass bubble filler</td>
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<td>Grefco Inc.</td>
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<tr>
<td>Sundex 740T</td>
<td>Plasticizer</td>
<td></td>
<td>Sun Products</td>
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<td>Graphite flake #1</td>
<td>Size: -20 Mesh + 80 Mesh</td>
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<td>Graphite flake #2</td>
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<td>Graphite flake #3</td>
<td>Size: -80 Mesh down</td>
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<td>Asbury Graphite Mills, Inc.</td>
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**Figures 7-22 have the same formulation**

**Fillers are incorporated by rubber mill technique**

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**Formulations of Polystyrene/Epoxy 1N Elastomers**

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<td>4</td>
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<td>6</td>
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</table>

**Notes:**
- Dimension 740L, 9
- Mica, 9
- Graphite, 9
- Graphite, 9

**Fillers**

- Pu/Epox ratio
- DEG 300%, 9
Figure 1. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN
Fig. 2  Dynamic Mechanical Spectroscopy: PU (143L Niax 31-28 (2% Isomol 100) (DER 330 BF3) -60/40

\[ E' \]

\[ E'' \]

\[ \tan \delta \]

\[ 10^8 \]

\[ 10^7 \]

\[ 10^6 \]

\[ 10^5 \]

\[ 10^4 \]

\[ 10^3 \]

\[ 10^2 \]

\[ 10^1 \]

\[ 10^0 \]

\[ -60 \]

\[ -20 \]

\[ 20 \]

\[ 60 \]

\[ 100 \]

\[ 140 \]

Temperature (°C)
Figure 3. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 10% #1 graphite filler
Figure 4. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 10% #2 graphite filler.
Figure 5. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 10% #3 graphite filler.
Figure 6. Comparison of the tanδ of unfilled and graphite filled PU/Epoxy (60/40) IPN

60/40 PU/Epoxy
10% #1 Graphite (-20 Mesh +80 Mesh)
10% #2 Graphite (-20 Mesh +200 Mesh)
10% #3 Graphite (-80 Mesh xDown)

Tanδ

Temperature, °C
Figure 7. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 20% #2 graphite.
Figure 8. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 40% #2 graphite
Figure 9. Comparison of the tanδ of graphite filled PU/Epoxy (60/40) IPN
Figure 10. Dynamic mechanical spectroscopy of PU/Epoxy 60/40 with 20% #2 graphite, roll milled technique.
Figure 11. Dynamic mechanical Spectroscopy of PU/Epoxy (60/40) IPN with 40% #2 graphite, roll milled technique.
Figure 12. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 20% Suzorite mica filler, no Isonol-100 chain extender.
Figure 13. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 40% suzorite mica, no Isonol-100 chain extender

- $E'$
- $E''$
- $\tan\delta$

Temperature ($^\circ$C)
Figure 14. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 20% Suzorite mica filler.
Figure 15. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 10% Dicaprol filler.

Temperature (°C)

E'

E''

\( \tan \delta \)
Figure 16. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN with 10% Sundex 750T plasticizer
Figure 17. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN without post-curing
Figure 18. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN after 2 hrs. post curing at 100°C
Figure 19. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN after 8 hrs. post-curing at 100°C.
Figure 20. Dynamic mechanical spectroscopy of PU/Epoxy (60/40) IPN after 16 hrs. post-curing at 100°C.
Figure 21. Comparison of the tanδ of samples with different post-curing times

- -------- no post-cured
- -- 2 hrs. post-cured at 100°C
- --- 8 hrs. post-cured at 100°C
- ---- 16 hrs. post-cured at 100°C
Figure 22. Dynamic mechanical spectroscopy of non-post-cured PU/Epoxy (60/40) IPN stabilized with CH₃OH.
Figure 23. Top portion of 70/30 PU/Epoxy IPN foam

Figure 24. Bottom portion of 70/30 PU/Epoxy IPN foam
Figure 25. Sound absorption properties of 60/40 PU/Epoxy IPN foam

Figure 26. Sound absorption property of 50/50 PU/Epoxy IPN foam
Figure 27. Sound absorption property of 40/60 PU/Epoxy IPN foam

Figure 28. Sound absorption property of 30/70 PU/Epoxy IPN foam
Figure 29. Sound absorption property of 70/30 PU/Epoxy IPN foam excluded
Isonol 100 chain extended and excess Isonate 143L

Figure 30. Sound absorption property of 60/40 PU/Epoxy IPN foam excluded
Isonol 100 chain extender and excess Isonate 143L
Figure 31. Sound absorption property of 50/50 PU/Epoxy IPN foam excluded Isonol 100 chain extender and excess Isonate 143L.

Figure 32. Sound absorption property of 60/40 PU/Epoxy IPN foam with low amount of A-1 catalyst (0.01) and T-12 catalyst (0.01%).
Figure 33. Sound absorption property of 40/60 PU/Epoxy IPN foam cured at 90°C for 16 hours

Figure 34. Sound absorption property of 40/60 PU/Epoxy IPN foam without post-curing
Figure 35. Sound absorption property of 50/50 PU/Epoxy IPN foam (no Freon 11A, DMP-30=0.4%)

Figure 36. Sound absorption property of 50/50 PU/Epoxy IPN foam (no Freon 11A, DMP-30=1.0%)
Figure 37. Sound absorption property of 50/50 PU/Epoxy IPN foam using CH$_2$Cl$_2$ as blowing agent and room temperature mixing.

Figure 38. Sound absorption property of 50/50 PU/Epoxy IPN foam using CH$_2$Cl$_2$ as blowing agent, mixing temperature at 80°C.
Figure 39. Sound absorption of GM polyurethane foam (Control)
Figure 40. Sound absorption property of University of Detroit's polyurethane foam (Control)

Formulation

143L, g 25
Niax 31-28, g 100
Water, g 1.05
A-1, g 0.01
T-12, g 0.06
DC-193, g 0.5
L-540, g 0.5
Freon 11A, g 15
PU/Epoxy 100/0
Water, % 1
DTIC
END
4-86