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Test Methods for the Dynamic Mechanical Properties of Polymeric Materials

By G. K. Baker

Published June 1980

Final Report

Prepared for the United States Department of Energy
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MECHANICAL PROPERTIES OF
POLYMERIC MATERIALS

By G. K. Baker

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Final Report
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TEST METHODS FOR THE DYNAMIC MECHANICAL PROPERTIES OF POLYMERIC MATERIALS

BDX-613-2405, Final Report, Published June 1980

Prepared by G. K. Baker

Various test geometries and procedures for the dynamic mechanical analysis of polymers employing a mechanical spectrometer have been evaluated. The methods and materials included in this work are forced torsional pendulum testing of Kevlar/epoxy laminates and rigid urethane foams, oscillatory parallel plate testing to determine the kinetics of the cure of VCE with Hylene MP, oscillatory compressive testing of B-3223 cellular silicone, and oscillatory tensile testing of Silastic E and single Kevlar filaments. Fundamental dynamic mechanical properties, including the storage and loss moduli and loss tangent of the materials tested, were determined as a function of temperature and sometimes of frequency.

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CONTENTS

Section	Page
SUMMARY.	8
DISCUSSION	10
SCOPE AND PURPOSE.	10
ACTIVITY	10
<u>Dynamic Mechanical Testing of Kevlar/Epoxy</u> <u>Laminates.</u>	10
<u>Dynamic Mechanical Testing of Rigid Urethane</u> <u>Foams.</u>	34
<u>Determination of the Kinetics of the Cure of VCE</u> <u>With Hylene MP Using Dynamic Mechanical Testing.</u> .	43
<u>Dynamic Compressive Testing of Flexible Foams.</u> . . .	50
<u>Dynamic Tensile Testing of Elastomers.</u>	57
<u>Dynamic Mechanical Testing of Fibers</u>	64
ACCOMPLISHMENTS.	67

ILLUSTRATIONS

Figure		Page
1	Orientation and Identification of Test Specimens Taken From the Six-Ply Kevlar/Epoxy Laminates With Respect to the Fabric Warp.	12
2	Storage Shear Modulus (G') of Six-Ply Kevlar/Epoxy Laminate as a Function of Temperature for Specimens Cured 16 Hours at 85°C.	15
3	Two Orientations of Kevlar Fabric Layers in the Test Specimens for Dynamic Mechanical Analysis	16
4	Storage Shear Modulus (G') of Six-Ply Kevlar/Epoxy Laminate as a Function of Temperature for Specimens Cured 2 Hours at 85°C.	17
5	Storage Shear Modulus of Kevlar/Epoxy Laminate With Two Resin/Cure Agent Weight Ratios.	21
6	Orientation of Test Specimens Taken From the 17-Ply Kevlar/Epoxy Laminate for the Reproducibility Study.	22
7	Storage Shear Modulus (G') of 17-Ply Kevlar/Epoxy Laminate.	27
8	Loss Shear Modulus (G'') of 17-Ply Kevlar/Epoxy Laminate.	29
9	Loss Tangent of 17-Ply Kevlar/Epoxy Laminate	31
10	Storage Shear Modulus of Kevlar Laminates Made From Various Numbers of Plies of Kevlar Fabric and DER 332/T-403 Resin System	33
11	Storage Shear Modulus of Kevlar/DER 332/T-403 Laminates Exposed to Various Relative Humidities.	35

12	Storage Shear Modulus Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures	39
13	Loss Shear Modulus Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures	40
14	Loss Tangent Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures. .	41
15	Shear Stress Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures. .	42
16	Change of Storage Shear Modulus of Unfilled (Left) and Filled (Right) VCE/Hylene MP Elastomer During Cure at Various Temperatures	44
17	First Order Plot of Cure of Unfilled (Left) and Filled (Right) VCE/Hylene MP at Various Temperatures	46
18	Arrhenius Plot for the Cure of Unfilled and Filled VCE/Hylene MP	49
19	Dynamic Mechanical Properties of B-3223 Cellular Silicone as a Function of Temperature and at a Frequency of 0.1 Hz.	56
20	Load Deflection Curves of B-3223 at Two Temperature Ranges	58
21	Load Deflection Properties of B-3223 Cellular Silicone as a Function of Temperature. . . .	61
22	Relationship of Storage Tensile Modulus and Toluene Swell Ratio for Silastic E	65
23	Tensile Stress of a Single Kevlar Filament as a Function of Vibrational Elongation in a Previously Elongated State	68

TABLES

Number		Page
1	Dynamic Mechanical Properties of Six-Ply Kevlar/Epoxy Laminate, Cured 16 Hours at 85°C	13
2	Dynamic Mechanical Properties of Six-Ply Kevlar/Epoxy Laminate, Cured 2 Hours at 85°C	18
3	Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 0° Orientation, Seven Test Specimens	23
4	Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 22.5° Orientation, Three Test Specimens.	24
5	Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 45° Orientation, Four Test Specimens	25
6	Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 90° Orientation, Two Test Specimens	26
7	Dynamic Mechanical Properties of Kevlar/Epoxy Laminates Stored at Various Relative Humidities	36
8	VCE Elastomer Formulations	45
9	Storage Shear Modulus Change During Cure of Unfilled and Filled VCE With Hylene MP	47
10	Rate Constants for the VCE/Hylene MP Reaction and Associated Data for the Arrhenius Plots in Figure 18	51
11	Dynamic Storage and Loss Compressive Modulus of B-3223 Cellular Silicone at 25°C and Three Frequencies.	53
12	Dynamic Compressive Properties of B-3223 Cellular Silicone at 0.1 and 1.0 Hz and Temperature Range of -120 to 160°C	55

13	Load Deflection Properties of B-3223 Over a Temperature Range of -120 to 200°C	60
14	Dynamic Tensile Properties of Silastic E With Varying Initial Sample Height, L_0	63
15	Dynamic Tensile Properties and Toluene Swell Ratio of Silastic E Silicone Rubber With Various Cure Conditions.	64
16	Maximum Dynamic Tensile Stress of Kevlar-49 Single Filament as a Function of Frequency .	67
17	Maximum Dynamic Tensile Stress of Kevlar-49 Single Filament as a Function of Elonga- tion	67

SUMMARY

The mechanical spectrometer provides a new capability at Bendix for the characterization and diagnostic analysis of polymeric materials. A project was initiated to develop procedures for the dynamic mechanical testing of a wide variety of polymeric materials used at Bendix in the manufacture of plastic products. Some of the fundamental dynamic mechanical properties of the materials tested also were determined.

The forced torsional pendulum geometry was used to determine the storage and loss shear moduli and loss tangent of Kevlar/epoxy laminates. The reproducibility of the method for storage shear modulus testing is very good, with a coefficient of variation of 3 to 4 percent. The coefficient of variation for the loss shear modulus is about 10 to 11 percent.

The orientation of the direction of warp of the Kevlar fabric in the laminates was found to have a small but measurable effect on the dynamic mechanical properties of the laminates. Processing variables such as cure time and mix ratio of epoxy resin and cure agent were found to be readily measured by dynamic mechanical testing. Above the glass transition temperature, the shear modulus of the laminates was found to increase as the number of plies of Kevlar fabric was increased from 12 to 20 plies.

Laminates made from Kevlar fabric with moisture contents varying from 0.2 to 3 percent were tested. With the exception of the sample containing 3 percent moisture, the laminates below the glass transition temperature exhibited the same shear modulus. Above the glass transition, shear modulus decreased slightly with increasing moisture content; moisture may be competing for epoxy groups during cure, resulting in a slightly lower crosslink density.

Low-density rigid urethane foams cured at three different temperatures were tested by the forced torsional pendulum geometry. Differences in the dynamic mechanical properties of the foam specimens were readily observed, and the data correlate well with thermal mechanical analysis (TMA) data. The mechanical spectrometer offers an advantage over TMA, in that a larger, more representative test specimen is used than with TMA.

The oscillatory parallel plate test geometry was found to be very useful for evaluating the cure behavior of low-modulus elastomers. The kinetics of the cure of VCE elastomer with Hylene MP were determined at four temperatures using this method. From these data, rate constants and activation energies for filled and unfilled elastomer were calculated.

The oscillatory compressive properties of flexible foams may be determined using a tension/compression fixture as an attachment to the mechanical spectrometer. This fixture translates the rotational motion of the mechanical spectrometer into an up and down motion. Specimens of B-3223 cellular silicone with formulation differences were tested. A definite decrease was observed in the dynamic compressive modulus in the foams with decreasing ratios of silica filler to silicone gum; unexpectedly, however, very little change was observed in the specimens with varying peroxide crosslinking agent. The dynamic mechanical properties of B-3223 cellular silicone were determined over a temperature range of -120 to 160°C. The compressive modulus of this material is nearly constant from -80 to 160°C.

Load deflection testing may be carried out using the tension/compression fixture if the mechanical spectrometer is operated in a steady rotation mode. Load deflection testing was performed on B-3223 cellular silicone over a temperature range of -120 to 200°C. As found in the dynamic testing, the load-bearing properties of B-3223 cellular silicone are nearly constant from -80 to 200°C. From -120 to -54°C, the silicone foam exhibits increasing elasticity (higher percent compression for a given load). Above -54°C, the elasticity decreases slightly and becomes constant from 120 to 200°C.

The use of the tension/compression fixture for oscillatory tension testing also was evaluated. The applicability of the method was demonstrated by determining the change in Silastic E, a RTV silicone rubber, with varying postcure conditions. The dynamic tensile modulus was observed to increase with increasing cure time and cure temperature. Toluene swell tests also were performed on the test specimens, and an excellent inverse linear relationship between storage tensile modulus and toluene swell ration was found.

Dynamic mechanical testing of single filaments of Kevlar was attempted. The method has good potential for the analysis of fibers; however, improvements need to be made in clamping fibers to the mechanical spectrometer.

The test procedures evaluated in this project are applicable to a wide variety of polymeric materials, including very high modulus materials such as the Kevlar/epoxy laminates, elastomers, rigid foams, flexible foams, and, with additional development, fibers. The methods developed will be useful in future activities, such as the preparation of materials and process specifications, diagnostic analysis of production problems, and plastic material and process development activities.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project was to develop new test methods for determining the dynamic mechanical properties of polymeric materials used to produce parts for various weapon programs. Fundamental dynamic mechanical property data of the materials used in the test method development also were determined.

ACTIVITY

In June, 1973, Bendix procured a mechanical spectrometer for measuring the dynamic mechanical properties of polymeric material. This instrument provided a new capability for characterization and diagnostic analysis of polymers and plastic parts in production.

Because the instrument was relatively new and because procedures for the type of testing required at Bendix had not been established, this development project was initiated. In addition to needing test methods for new materials, better test methods than those currently used were needed for older materials, such as silicone foams. Moreover, materials such as Kevlar fibers and Kevlar/epoxy laminates were new to the plastics industry, and many of their mechanical properties had not yet been determined. Consequently, a secondary objective of the project was to determine basic property data of selected materials.

Dynamic Mechanical Testing of Kevlar/Epoxy Laminates

Test Method

The dynamic mechanical properties of Kevlar laminates, which include the storage shear modulus (G'), loss shear modulus (G''), and loss tangent (G''/G'), were determined using the forced torsional pendulum test geometry. In this test, a rectangular test specimen is clamped at the top and bottom. The bottom clamp is stationary and is coupled to the mechanical spectrometer stress detection transducer. The top clamp is connected to the drive unit which, with a low frequency sinusoidal function generator, can apply an oscillatory torsional strain on the specimen. The maximum stress and phase angle (difference between the oscillatory stress and strain) is measured with a phase analyzer. From these data, the G' , G'' , and loss tangent can be calculated.

The specimens used were nominally 3.8 by 1.27 by 0.3 cm and were cut from the Kevlar/epoxy laminates with a carbon dioxide laser welding machine. The length between the clamps was approximately 2.54 cm.

Effect of Fabric Orientation

An experiment was performed to determine the effect of the orientation of Kevlar fabric in the laminate test specimen on the dynamic mechanical properties of the specimen.

A single, six-ply laminate was made of Kevlar-49 fabric from merge number 6-G004 and DER 332 epoxy resin cured with Jeffamine T-403. The weight ratio of epoxy resin to cure agent was 100/35. The Kevlar cloth was laid up with the warp of the two center layers oriented at 0 degrees. The next two layers were oriented at 45 degrees on one side of the two center layers and at 315 degrees on the other side. The orientation of the warp of the two outermost layers was 90 degrees. The resin laminate was heated between the platens of a laboratory press to 85°C and under a pressure of 103 kPa for 16 hours to cure the resin. The resulting laminate was 0.13 cm thick and had a resin content of 41 percent.

Rectangular test specimens, nominally 3.8 by 1.3 cm were cut from the cured laminate for dynamic mechanical testing with the mechanical spectrometer. The test specimens were cut using a CO₂ laser welding machine, and were taken such that the orientation of the fabric warp was varied as shown in Figure 1.

The storage shear modulus (G'), loss shear modulus (G''), and loss tangent of the various specimens taken from the laminate were determined using the mechanical spectrometer and the forced torsional pendulum test mode. Stress measurements were made over a temperature range of -60 to 220°C and at a single frequency of 0.2 Hz.

The results are tabulated in Table 1; plots of the storage shear modulus, G' , as a function of temperature are given in Figure 2. The data show this six-ply laminate is anisotropic with respect to shear modulus. Two distinct sets of curves are observed. One set, representing those specimens cut at a 45° angle from the direction of the warp of the center two layers (specimens C and D, as shown in Figure 1), have a distinctly higher modulus over the entire temperature range, as compared to the other set.

The other set of curves is from specimens cut either in the direction of the warp of the center two layers or at right angles to it (specimens A and B, respectively, in Figure 1).

Figure 1. Orientation and Identification of Test Specimens Taken From the Six-Ply Kevlar/Epoxy Laminates With Respect to the Fabric Warp

An examination of the procedure used to lay up the laminate shows those specimens cut at 45° to the warp of the center two layers (specimens C and D) contain four layers in which the warp of the fabric is oriented at 45° (or 315°) relative to the long portion of the rectangle. That is, specimens C and D have four layers in which the fabric is oriented as shown in Figure 3a, and two layers oriented as shown in Figure 3b.

On the other hand, specimens A and B contain four layers of Kevlar fabric which are oriented as shown in Figure 3b and two layers as shown in Figure 3a. The conclusion is the shear modulus is higher in laminates having more plies in which the bias of the fabric is in the same direction as the torsional axis.

Effect of Processing Conditions

A useful application of dynamic mechanical analysis should be evaluation of the processing conditions used to manufacture plastic parts. In the case of Kevlar/epoxy laminates for example,

Table 1. Dynamic Mechanical Properties of Six-Ply Kevlar/Epoxy Laminate, Cured 16 Hours at 85°C

Temperature (°C)	Loss Tangent (G''/G')	Shear Modulus (MPa)	
		Storage (G')	Loss (G'')
Specimen A			
- 60	0.020	6,495	132
- 40	0.020	6,311	128
- 20	0.018	6,081	106
0	0.014	5,943	86
20	0.014	5,759	83
40	0.014	5,621	81
60	0.061	4,921	301
80	0.132	1,078	142
100	0.047	939	43
120	0.035	871	30
140	0.026	844	32
160	0.023	837	19
180	0.020	830	16
200	0.014	829	12
220	0.020	843	17
Specimen B			
- 60	0.020	6,170	125
- 40	0.023	5,923	137
- 20	0.006	5,677	32
0	0.020	5,528	112
20	0.014	5,282	76
40	0.020	5,133	104
60	0.076	4,430	335
80	0.146	1,219	178
100	0.052	993	52
120	0.047	936	43
140	0.038	901	34
160	0.029	865	25
180	0.026	866	22
200	0.026	844	22
220	0.026	830	21
240	0.026	837	21
260	0.026	830	

Table 1 Continued. Dynamic Mechanical Properties
of Six-Ply Kevlar/Epoxy
Laminate, Cured 16 Hours at
85°C

Temperature (°C)	Loss Tangent (G''/G')	Shear Modulus (MPa)	
		Storage (G')	Loss (G'')
Specimen C			
- 60	0.009	10,319	89
- 40	0.003	10,206	29
- 20	0.018	9,965	174
0	0.015	9,647	140
20	0.012	9,567	111
40	0.015	9,409	136
60	0.023	8,537	198
70	0.302	4,597	1390
80	0.206	1,922	396
90	0.091	1,659	150
100	0.061	1,624	99
120	0.049	1,451	71
160	0.038	1,318	50
210	0.029	1,215	35
Specimen D			
- 60	0.018	10,916	191
- 40	0.018	10,850	190
- 20	0.012	10,601	124
0	0.015	10,350	150
20	0.012	10,183	119
40	0.012	10,016	117
60	0.135	7,777	1048
80	0.141	1,978	278
100	0.061	1,684	103
120	0.047	1,599	74
140	0.038	1,509	57
180	0.032	1,454	47
220	0.023	1,366	32

Figure 2. Storage Shear Modulus (G') of Six-Ply Kevlar/Epoxy Laminate as a Function of Temperature for Specimens Cured 16 Hours at 85°C

Figure 3. Two Orientations of Kevlar Fabric Layers in the Test Specimens for Dynamic Mechanical Analysis

the processing engineer needs to know the appropriate cure time and temperature to obtain fully-cured laminates. Consequently, several experiments were performed to determine if changes in processing conditions could be detected by dynamic mechanical analysis.

The effect of cure time was examined by preparing a six-ply laminate in the same manner as previously, except that this second laminate was cured for 2 hours at 85°C rather than 16 hours. Test specimens for dynamic mechanical testing were taken from this laminate in a manner similar to the laminate cured for 16 hours (shown in Figure 1). The shear modulus data for these specimens are given in Figure 4 and Table 2.

The specimens from the laminate cured for 2 hours have a pattern of anisotropy with respect to shear modulus similar to the laminate cured for 16 hours. The specimens with four plies of fabric oriented as shown in Figure 3a have a higher shear modulus over the temperature range of -60 to 220°C than those specimens with four plies of fabric oriented as shown in Figure 3b.

The data show the cure time has a significant effect on the dynamic mechanical properties. The samples having the longer cure time of 16 hours have a higher viscoelastic transition temperature (which is related to the glass transition temperature) than the samples cured for 2 hours. That is, the inflection point of the G' curve occurs at about 70°C for the sample cured 16 hours and at about 50°C for the sample cured for 2 hours.

Figure 4. Storage Shear Modulus (G') of Six-Ply Kevlar/Epoxy Laminate as a Function of Temperature for Specimens Cured 2 Hours at 85°C

Table 2. Dynamic Mechanical Properties of Six-Ply Kevlar/Epoxy Laminate, Cured 2 Hours at 85°C

Temperature (°C)	Loss Tangent (G''/G')	Shear Modulus (MPa)	
		Storage (G')	Loss (G'')
Specimen A			
- 60	0.018	5,503	97
- 40	0.014	5,298	77
- 20	0.020	5,145	91
0	0.014	4,993	72
20	0.018	4,840	85
40	0.044	4,456	194
60	0.488	1,128	550
70	0.200	570	114
80	0.099	517	51
90	0.070	506	35
100	0.058	502	29
120	0.044	494	21
140	0.035	478	16
160	0.029	454	13
180	0.026	446	11
Specimen B			
- 60	0.027	6,126	165
- 40	0.023	5,881	135
- 20	0.020	5,784	116
0	0.020	5,588	112
20	0.023	5,432	125
30	0.032	5,267	168
40	0.073	4,645	339
50	0.216	3,141	678
60	0.384	1,196	459
70	0.188	763	143
80	0.114	702	80
100	0.076	670	51
120	0.061	651	40
140	0.050	631	32
200	0.038	631	24

Table 2 Continued. Dynamic Mechanical Properties
of Six-Ply Kevlar/Epoxy
Laminate, Cured 2 Hours at 85°C

Temperature (°C)	Loss Tangent (G''/G')	Shear Modulus (MPa)	
		Storage (G')	Loss (G'')
Specimen C			
- 60	0.009	9,710	84
- 40	0.009	10,230	89
- 20	0.012	9,969	116
0	0.012	9,584	112
20	0.003	9,351	28
30	0.015	9,273	134
40	0.035	8,718	304
50	0.185	6,112	1132
60	0.351	2,631	723
70	0.234	1,388	324
80	0.114	1,178	128
100	0.067	1,143	76
120	0.055	1,110	61
140	0.050	1,091	54
160	0.044	1,091	47
200	0.035	1,091	38
220	0.032	1,052	33
Specimen D			
- 60	0.018	10,953	192
- 40	0.020	10,664	217
- 20	0.018	9,893	173
0	0.015	9,660	140
20	0.015	9,504	138
40	0.023	9,035	210
60	0.284	2,918	828
70	0.144	1,794	258
80	0.090	1,600	145
100	0.058	1,425	83
120	0.041	1,341	55
140	0.034	1,341	47
180	0.032	1,311	42
200	0.032	1,301	42
220	0.026	1,262	33

The effect of cure time is reflected also in the temperature at which the maximum of the loss tangent occurs. The loss tangent is the ratio of the loss shear modulus to the storage shear modulus, or G''/G' . The maxima occur at 60°C and 70 to 80°C for the laminates cured for 2 hours and 16 hours, respectively. The greater degree of cure for the specimen cured for 16 hours is also reflected in the higher storage shear modulus observed above the transition temperature ($\sim 80^\circ\text{C}$). The laminates cured for 16 hours had moduli ranging from 900 to 1500 MPa and the laminates cured for 2 hours had moduli ranging from about 450 to 1200 MPa over the same temperature range (80 to 220°C). These data show that the laminates cured 16 hours have a higher cross-link density than the laminates cured for 2 hours and that such differences can readily be detected by dynamic mechanical analysis.

The effect of varying the weight ratio of DER 332 epoxy resin to Jeffamine T-403 cure agent was evaluated by testing specimens of the resin system alone. In most of the Kevlar/epoxy laminates tested in this project, the weight ratio of DER 332 to T-403 was 100 to 35.

Calculations indicate a weight ratio of 45 parts of T-403 to 100 parts of DER 332 would be a 1:1 ratio of epoxy groups to active amine hydrogen groups. The G' and G'' between -140 and 140°C of test specimens made from the two weight ratios are given in Figure 5. The increased amount of T-403 cure agent provides a greater degree of crosslinking which is exhibited in Figure 5 by three characteristics. One is the higher modulus above the viscoelastic transition temperature ($\sim 90^\circ\text{C}$). The second is the shift of the viscoelastic transition temperature from 70 to 90°C, and the third is the shift of the G'' maximum from 60 to 70°C.

Reproducibility of the Method

A single 17-ply laminate was made of Kevlar-49 fabric and DER 332 epoxy resin cured with Jeffamine T-403. The weight ratio of epoxy resin to cure agent was 100:35. The laminate was prepared by laying up the 17 plies of Kevlar cloth so that the orientation of the warp of the cloth alternated between 0° and 45°. That is, the center ply was oriented at 0°, the next two plies oriented at 45°, and so forth. The outermost plies of the laminate were oriented at 45°.

The laminate was cured between the platens of a press at 99°C for 1 hour. The resulting thickness and resin content of the laminate was 0.340 cm and 35 weight percent (59 volume percent), respectively.

To evaluate the reproducibility of the test, the following number of test specimens having the four orientations shown in Figure 6 were tested: seven at 0°, three at 22.5°, four at 45°, and two at 90°. The average and standard deviation of G' , G'' , and loss tangent for the various orientations was calculated and are given

Figure 5. Storage Shear Modulus of Kevlar/Epoxy Laminate With Two Resin/Cure Agent Weight Ratios

Figure 6. Orientation of Test Specimens Taken From the 17-Ply Kevlar/Epoxy Laminate for the Reproducibility Study

in Tables 3 through 6. The standard deviation was not calculated for the 90° specimens because only two were tested. The data in Tables 3 through 6 are shown graphically in Figures 7 through 9.

The results indicate the forced torsional pendulum test mode for determining the dynamic mechanical properties of Kevlar/epoxy laminates is very reproducible. This is especially true in the case of the storage shear modulus, G' . The coefficient of variation of G' was usually less than 5 percent.

The anisotropy of the dynamic mechanical properties as a result of fabric orientation reported in a previous section also was observed in the 17-ply laminate used in the reproducibility study. The 0- and 90-degree test specimens yielded slightly higher shear moduli than the 22.5- and 45-degree specimens. The 0- and 90-degree specimens have eight layers as depicted in Figure 3a and seven layers of the orientation given in Figure 3b; the reverse is the case for the specimens cut at 45°. The specimens cut at 22.5° have a fabric orientation somewhere between that of 0 and 45°.

Table 3. Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 0° Orientation, Seven Test Specimens

Temperature (°C)	Storage Shear Modulus, G'			Loss Shear Modulus, G''			Loss Tangent, G''/G'	
	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average	Standard Deviation
-140	4920	150	3.0	15	0.6	4.0	0.003	0
-120	4830	150	3.1	20	7.3	46.5	0.004	0.002
-100	4720	130	2.8	41	1.2	2.9	0.009	0
- 80	4510	150	3.3	64	3.3	5.2	0.014	0.001
- 60	4330	150	3.5	63	8.4	3.3	0.014	0.002
- 40	4210	150	3.6	63	4.9	7.8	0.015	0.001
- 20	4100	140	3.4	49	4.5	9.9	0.012	0.001
0	4020	135	3.4	34	1.1	3.2	0.009	0.001
40	3850	110	2.9	33	0.6	1.8	0.010	0.002
60	3550	70	2.0	97	26.0	26.8	0.028	0.006
70	2730	120	4.3	430	72.0	16.7	0.157	0.031
80	990	75	7.6	350	38.0	10.9	0.348	0.017
90	560	25	4.5	107	10.6	9.9	0.197	0.016
100	480	7	2.2	63	4.5	7.1	0.130	0.010
120	440	9	2.0	45	3.5	7.8	0.101	0.007
160	430	15	3.5	34	3.1	9.1	0.077	0.007
200	420	15	3.6	27	2.8	10.3	0.064	0.007

Table 4. Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 22.5° Orientation, Three Test Specimens

Temperature (°C)	Storage Shear Modulus, G'			Loss Shear Modulus, G''			Loss Tangent, G''/G'	
	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average	Standard Deviation
-140	4250	85	2.0	13	0.7	5.4	0.006	0.008
-120	4190	65	5.5	25	0.7	2.8	0.008	0.003
-100	4100	40	1.0	42	7.8	18.6	0.009	0.003
- 80	3940	25	0.6	63	8.5	13.5	0.016	0.002
- 60	3800	45	1.1	62	7.8	12.6	0.013	0.006
- 40	3740	100	2.7	61	6.4	10.5	0.016	0.002
- 20	3640	95	2.6	60	9.2	15.3	0.013	0.002
0	3550	80	1.2	32	1.2	3.8	0.009	0.001
40	3410	100	1.9	36	7.8	21.7	0.008	0.004
60	3220	215	6.7	125	9.2	7.4	0.033	0.006
70	2220	105	4.7	390	21.0	5.4	0.175	0.002
80	780	40	5.1	250	18.0	7.2	0.322	0.006
90	480	22	0.4	90	1.7	1.9	0.184	0.003
100	440	12	2.7	55	1.7	3.1	0.123	0.006
120	390	9	2.3	35	1.5	4.3	0.096	0.005
160	370	10	2.7	20	12.7	63.5	0.070	0.002
200	360	12	3.3	21	1.0	4.7	0.058	-

Table 5. Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 45° Orientation, Four Test Specimens

Temperature (°C)	Storage Shear Modulus, G'			Loss Shear Modulus, G''			Loss Tangent, G''/G'	
	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average (MPa)	Standard Deviation (MPa)	Coefficient of Variation (Percent)	Average	Standard Deviation
-140	4180	105	2.5	13	0.8	6.1	0.003	0
-120	4140	105	2.5	25	1.5	6.0	0.005	0.001
-100	4040	110	2.7	45	2.4	5.3	0.011	0.003
- 80	3860	140	3.6	62	6.1	9.8	0.015	0.002
- 60	3710	135	3.6	56	4.0	7.1	0.014	0
- 40	3590	130	3.6	67	5.6	9.8	0.016	0.002
- 20	3500	130	3.7	57	7.1	15.1	0.013	0.002
0	3420	120	3.5	28	5.1	18.2	0.009	0
40	3350	225	6.7	26	4.2	16.2	0.008	0.002
60	3160	200	6.3	100	12.5	12.5	0.028	0.010
70	2450	530	21.6	360	44.0	12.5	0.152	0.024
80	790	110	13.9	285	25.0	8.8	0.248	0.088
90	470	35	7.4	85	10.0	11.8	0.186	0.006
100	410	35	8.5	44	2.0	4.5	0.110	0.013
120	380	25	6.6	33	3.3	10.0	0.085	0.010
160	370	25	6.8	24	2.6	10.8	0.061	0.006
200	350	25	7.1	18	1.0	5.5	0.053	0.006

Table 6. Dynamic Mechanical Properties of Kevlar/Epoxy Laminate, 90° Orientation, Two Test Specimens

Temperature, (°C)	Storage Shear Modulus, G' (MPa)		Loss Shear Modulus, G'' (MPa)		Loss Tangent, G''/G'	
	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
-140	4950	4730	-	14	-	0.003
-120	4870	4620	42	-	0.009	-
-100	4790	4490	28	26	0.006	0.006
- 80	4630	4320	27	51	0.006	0.012
- 60	4310	4220	25	49	0.006	0.014
- 40	4140	4051	36	47	0.009	0.012
- 20	4070	3950	24	46	0.006	0.012
0	4000	3840	47	33	0.012	0.009
40	3900	3600	23	21	0.006	0.006
60	3580	3430	115	60	0.032	0.018
70	2920	2660	385	360	0.132	0.135
80	1160	1110	415	380	0.358	0.341
90	620	470	130	110	0.210	0.236
100	520	385	65	45	0.129	0.117
120	485	360	50	33	0.102	0.091
160	470	355	37	23	0.079	0.064
200	440	355	27	19	0.061	0.052

Effect of Number of Plies of Kevlar Fabric

An evaluation of the change in G' with number of plies of Kevlar fabric was made. Laminates having 12, 14, 16, 18, 20, and 22 plies of fabric were molded at 85°C for 2 hours and using the DER 332/T-403 resin system; the weight ratio of DER 332 to T-403 was 100 to 45.

The storage shear modulus data for each of the laminates are summarized in Figure 10. The modulus is observed to increase as the number of plies increases from 12 to 18. This behavior is observed both below and above the transition temperature, which begins at about 70°C. The 20-ply laminate has about the same modulus as that of the 14-ply laminate below the transition temperature, but the modulus is higher than that of the 18-ply laminate between 80 and 220°C. The 22-ply laminate was found to have the lowest modulus of all the laminates below the transition temperature and about the same modulus as that of the 20-ply

Figure 7. Storage Shear Modulus (G') of 17-Ply Kevlar/Epoxy Laminate

Figure 7 Continued. Storage Shear Modulus (G') of 17-Ply Kevlar/Epoxy Laminate

Figure 8. Loss Shear Modulus (G'') of 17-Ply Kevlar/Epoxy Laminate

Figure 8 Continued. Loss Shear Modulus (G'') of 17-Ply Kevlar/Epoxy Laminate

Figure 9. Loss Tangent of 17-Ply Kevlar/Epoxy Laminate

Figure 9 Continued. Loss Tangent of 17-Ply Kevlar/Epoxy Laminate

Figure 10. Storage Shear Modulus of Kevlar Laminates Made From Various Numbers of Plies of Kevlar Fabric and DER 332/T-403 Resin System

laminate above the transition temperature. The reason that the higher ply laminates exhibit a low modulus below the transition temperature and yet a moderate-to-high modulus above the transition temperature is not immediately apparent.

Effect of Moisture on Kevlar/Epoxy Laminates

Kevlar fabric squares, (20.3 by 20.3 cm), were cut and placed in four desiccators made to have relative humidities (RH) of 0, 20, 40, and 60 percent. The samples were stored in the desiccators for 3 to 5 weeks, which is sufficient time for the fabric to reach equilibrium moisture content. From a previous moisture analysis of Kevlar, the fabric stored at 0, 20, 40, and 60 percent RH had moisture contents of 0.2, 1.6, 2.4, and 2.9 percent, respectively.

Three laminates, (20.3 by 20.3 by 0.32 cm) were prepared from fabric exposed to each of the four moisture conditions. The resin system used was 100 parts DER 332 to 45 parts Jeffamine T-403. Each laminate contained 16 plies of fabric and was molded at 85°C for 2.5 hours. Rectangular test specimens (3.8 by 1.27 by 0.32 cm) were cut from the laminates with a CO₂ laser welding machine. The specimens were tested using the forced torsional pendulum geometry at a frequency of 1 Hz and a maximum strain of 0.0135 rad.

Duplicate determinations of storage and loss shear modulus and loss tangent for each relative humidity condition were made over a temperature range of -140 to 300°C. Plots of the storage shear modulus are shown in Figure 11, and the other dynamic mechanical property data are given in Table 7.

The data indicate moisture in the Kevlar fabric prior to molding has only a slight effect on the dynamic mechanical properties of laminates. From -140 to 100°C, the modulus of the laminates are essentially the same, with the exception of the 60-percent RH sample. From 100 to 300°C, the plateau region, there is a slight trend of decreasing modulus with increasing relative humidity. Moisture may be competing for epoxy groups during cure, resulting in a slightly lower crosslink density.

Dynamic Mechanical Testing of Rigid Urethane Foams

Two procedures for the dynamic mechanical testing of rigid urethane foams were evaluated. The first method was the use of the forced torsional pendulum test mode to obtain the storage shear modulus (G'), loss shear modulus (G''), and loss tangent (G''/G') of a rigid urethane foam.

The foam system is a sucrose-based polyether cured with a polymeric aryl isocyanate (PAPI). Three test blocks of the foam (15.24 by 15.24 by 2.54 cm) were prepared and cured at three

Figure 11. Storage Shear Modulus of Kevlar/DER 332/T-403 Laminates Exposed to Various Relative Humidities

Table 7. Dynamic Mechanical Properties of Kevlar/Epoxy Laminates Stored at Various Relative Humidities

Temperature (°C)	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent
0 Percent RH	Specimen 10-10			Specimen 11-10		
-140	7460	132	0.018	7400	161	0.022
-120	7400	126	0.017	7330	161	0.022
-100	7270	152	0.021	7220	172	0.024
- 80	7050	196	0.028	7050	202	0.029
- 60	6810	225	0.033	6810	231	0.034
- 40	6600	223	0.034	6570	229	0.035
- 20	6430	212	0.033	6410	211	0.033
0	6300	187	0.030	6290	180	0.029
20	6200	161	0.026	6140	196	0.032
40	6100	132	0.022	6080	169	0.028
60	5910	148	0.025	5930	147	0.025
80	5000	406	0.081	5040	403	0.080
90				2390	800	0.336
100	1310	293	0.224	1260	231	0.184
120	1020	73	0.071	1050	78	0.074
140	980	54	0.055	990	55	0.056
180	930	30	0.033	930	30	0.032
220	880	18	0.021	920	18	0.020
260				890	15	0.017
300				880	14	0.016
20 Percent RH	Specimen 1-10			Specimen 2-10		
-140	7540	134	0.018	7020	252	0.036
-120	7480	127	0.017	6850	226	0.033
-100	7370	138	0.019	6850	226	0.033
- 80	7190	186	0.026	6720	255	0.038
- 60	6930	213	0.031	6490	266	0.041
- 40	6670	213	0.032	6290	257	0.041
- 20	6490	193	0.028	6130	226	0.037
0	6360	163	0.026	6020	199	0.033
20	6260	136	0.022	5940	165	0.028
40	6160	109	0.018	5820	156	0.027
60	5930	129	0.022	5650	145	0.026
80	4980	417	0.084	4860	380	0.078
90	2100	705	0.336	2260	768	0.340
100	1260	227	0.180	1240	251	0.203
120	1050	72	0.068	1010	75	0.074

Table 7 Continued. Dynamic Mechanical Properties of Kevlar/Epoxy Laminates Stored at Various Relative Humidities

Temperature (°C)	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent
140	1010	53	0.053	940	53	0.056
180	970	28	0.029	880	27	0.031
220	930	19	0.021	840	17	0.020
260	880	14	0.016	830	12	0.015
300	870	13	0.015	830	12	0.014
<hr/>						
40 Percent RH	Specimen 5-10			Specimen 4-10		
-140	7700	191	0.025	7320	196	0.027
-120	7630	182	0.024	7270	196	0.027
-100	7470	209	0.028	7120	227	0.032
- 80	7220	251	0.035	6880	274	0.040
- 60	6870	267	0.039	6600	281	0.043
- 40	6600	249	0.038	6290	263	0.042
- 20	6390	229	0.036	6110	225	0.037
0	6240	173	0.028	5980	189	0.032
20	6150	140	0.023	5870	158	0.027
40	6030	124	0.021	5770	149	0.026
60	5780	138	0.024	5460	174	0.032
80	4920	729	0.186	4160	526	0.126
90	1630	499	0.307	1960	637	0.325
100	1100	164	0.149	1170	208	0.179
140	920	46	0.050	920	53	0.058
180	880	24	0.028	870	30	0.035
220	830	17	0.021	830	22	0.027
260	750	14	0.019	770	19	0.025
300	760	14	0.019	760	17	0.023
<hr/>						
60 Percent RH	Specimen 7-10			Specimen 8-10		
-140	7400	190	0.026	7270	289	0.040
-120	7330	188	0.026	7230	280	0.039
-100	7170	214	0.030	7130	291	0.041
- 80	6880	267	0.039	7000	325	0.047
- 60	6560	274	0.042	6600	322	0.049
- 40	6250	268	0.043	6330	296	0.047
- 20	6030	230	0.038	6140	269	0.044
0	5890	193	0.033	6000	222	0.037
20	5800	160	0.028	5910	195	0.030
40	5570	128	0.023	5810	186	0.032

Table 7 Continued. Dynamic Mechanical Properties of Kevlar/Epoxy Laminates Stored at Various Relative Humidities

Temperature (°C)	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent	Storage Shear Modulus (MPa)	Loss Shear Modulus (MPa)	Loss Tangent
60	5370	133	0.025	5540	181	0.033
80	3990	582	0.156	4030	629	0.156
90	1980	650	0.328	1840	601	0.326
100	1190	232	0.194	1180	212	0.180
140	940	55	0.058	940	51	0.054
180	880	30	0.034	900	28	0.031
220	840	20	0.023	850	20	0.023
260	730	16	0.022	780	16	0.021
300	710	14	0.019	770	15	0.020

different temperatures (121°C, 163°C, and 204°C) for 8 hours. The density of the foam of each test block was 320 kg/m³. Rectangular test specimens (4.45 by 1.27 by 0.32 cm) were machined from the larger test blocks. Dynamic mechanical testing of the specimens was performed at a frequency of 0.2 Hz and over a temperature range of 30 to 250°C.

Plots of the G' , G'' , and loss tangent data as a function of temperature are given in Figures 12, 13, and 14, respectively. The foam specimens were cured at different temperatures; therefore, a difference in their mechanical behavior would be expected. Urethane foams undergo more extensive crosslinking at higher temperatures, and other crosslinking mechanisms can occur, such as the formation of allophanate and biuret groups. As Figures 12, 13, and 14 show, the dynamic mechanical properties of the three foams were found to vary as expected. The transition shifts to higher temperatures with increasing cure temperature.

Thermal mechanical analysis (TMA), using an expansion probe and a programmed heating rate of 5°C/minute, was performed on each foam specimen. The foams cured at 121°C, 163°C, and 204°C underwent transitions at 140°C, 167°C, and 170°C, respectively. These temperatures correlate approximately with the maxima of the G'' curves (Figure 13) and with the point at which the loss tangent rapidly increases (Figure 14).

The second technique evaluated involved the continuous measurement of the stress resulting from the sinusoidally varying strain, and while heating the sample at a programmed rate of 5°C/minute. The

Figure 12. Storage Shear Modulus Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures

Figure 13. Loss Shear Modulus Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures

Figure 14. Loss Tangent Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures

temperature range was 30°C to about 200°C, and the strain was always ± 0.136 rad. A two-channel recorder was used to simultaneously measure the sample temperature and the stress. The results of this testing are illustrated in Figure 15. Again, the transition, referred to as the softening point of the foam caused by segmental motion of some portion of the polymer backbone, is readily observed. The temperatures at the transitions, 137°C, 158°C, and 165°C for the foams cured at 121, 163, and 204°C, respectively, correlate reasonably well with the TMA and G' data.

This technique may be more applicable to some rigid cellular materials, compared to thermal mechanical analysis using a penetration or expansion probe. Abnormal TMA curves are sometimes obtained because of penetration into a cell caused by fracture of the cell, as opposed to being caused by molecular motion. Also, the area of the sample being tested by TMA is quite small ($1.1 \times 10^{-1} \text{ cm}^2$ for the expansion probe and $6.4 \times 10^{-3} \text{ cm}^2$ for the penetration probe). Consequently, inhomogeneities in foams can

Figure 15. Shear Stress Versus Temperature of Rigid Urethane Foam Cured at Three Temperatures

give misleading results. The test specimens used in this new method are considerably larger than those used in TMA and are, therefore, more representative of the actual cellular product.

Determination of the Kinetics of the Cure of VCE With Hylene MP Using Dynamic Mechanical Testing

Use of the oscillatory parallel plate geometry with the mechanical spectrometer should be especially useful for determining the cure behavior of low modulus elastomers. Therefore, an experiment was performed to evaluate this technique for such an application.

The binder used in the manufacture of filled elastomer parts is VCE crosslinked with Hylene MP. VCE is a terpolymer of vinyl ethylene/vinyl acetate/vinyl alcohol in which the weight ratio of the mers is about 57 percent ethylene, 37 percent vinyl acetate and 6 percent vinyl alcohol. Hylene MP (a registered trademark of Du Pont) is diphenyl-4,4'-methylenebis (phenylcarbamate) and is commonly called phenol blocked MDI. MDI is the acronym for 4,4'-methylenebis (phenyl-isocyanate). Samples of unfilled and filled VCE were prepared by blending the components of the formulation on a rubber mill; the formulations are given in Table 8.

Lithium stearate is used as an internal lubricant and is chemically inert in this formulation. Leached natural boron (less than 0.02 percent boric acid) was used for the filled formulation. The ratio of VCE/Hylene MP/lithium stearate is the same in both formulations, and the ratio of isocyanate groups from the Hylene MP to hydroxyl groups from the VCE is about 1:2.

The cure rates of the two formulations were determined at temperatures of 135, 150, 165, and 178°C, according to the following procedure. The 25-mm-diameter serrated parallel plate discs were connected to the mechanical spectrometer and heated to the appropriate temperature in the environmental chamber.

A sample of the VCE formulation was placed on the lower plate, and the upper plate was quickly lowered until the gap between the two plates was 2.6 mm. A clock was started to monitor the cure time. The excess material which flowed out between the plates was trimmed off with a razor blade while rotating both plates, and then the lower plate was clamped to remain stationary during the tests. The sample loading operation was accomplished in less than 1 minute.

Measurements of the storage shear modulus (G') as a function of time were made while the upper plate was oscillated in a dynamic sinusoidal manner at a frequency of 1 Hz. The maximum angular oscillation amplitude was 0.0026 rad, which represents a maximum strain amplitude of 0.13. Plots of the change in G' with time for the filled and unfilled VCE formulations are given in Figure 16.

Figure 16. Change of Storage Shear Modulus of Unfilled (Left) and Filled (Right) VCE/Hylene MP Elastomer During Cure at Various Temperatures

Table 8. VCE Elastomer Formulations

Component	Unfilled (Percent)	Filled (Percent)
VCE	90.0	27.0
Hylene MP	8.3	2.5
Lithium Stearate	1.7	0.5
Boron		70.0

The shear modulus data were examined to determine the kinetics of the reaction between VCE and Hylene MP. The reaction was found to be first order over the entire temperature range examined (135 to 178°C). The rate constants at each temperature were determined from Equation 1 by plotting $\ln (G_f'/G_t' - G_t')$ versus time. The rate constant, k , is the slope of the line.

$$\ln \frac{G_f'}{G_f' - G_t'} = kt \quad (1)$$

where

G_f' = final shear modulus,

G_t' = shear modulus at time t ,

k = rate constant, and

t = time.

As observed in the plots for unfilled VCE and filled VCE (Figure 17), the curves are linear over the entire reaction, which is proof the reaction is first order. The data from which the plots were prepared are given in Table 9.

To determine if the data obey the Arrhenius law, expressed in Equations 2 and 3, plots of $\ln k$ versus $1/T$ were made and are given in Figure 18.

$$k = Ae^{-E_a/RT} \quad (2)$$

Figure 17. First Order Plot of Cure of Unfilled (Left) and Filled (Right) VCE/Hylene MP at Various Temperatures

Table 9. Storage Shear Modulus Change During
Cure of Unfilled and Filled VCE With
Hylene MP

Temperature (°C)	Cure Time (Min)	Storage Shear Modulus, G' (MPa)	$\ln \frac{G'_f}{G'_f - G'_t}$
Unfilled			
178	1	1.18	0.36
	2	3.41	2.06
	3	3.86	4.36
	4	3.90	5.99
	5	3.91	
165	1	0.19	0.05
	2	1.83	0.68
	3	2.97	1.61
	4	3.42	2.54
	5	3.59	3.41
	6	3.66	4.25
	10	3.70	5.65
150	15	3.71	
	1	0.31	0.01
	3	0.57	0.16
	5	1.94	0.71
	10	3.14	1.75
	15	3.57	2.80
	20	3.72	3.86
	25	3.77	4.84
	30	3.79	5.94
	35	3.80	
135	5	0.17	0.03
	10	1.01	0.11
	15	2.14	0.49
	20	3.07	0.82
	30	4.22	1.46
	40	4.80	2.06
	50	5.12	2.67
	55	5.22	2.98
	65	5.36	3.67
	70	5.40	4.00
	90	5.49	6.31
	100	5.50	

Table 9 Continued. Storage Shear Modulus Change
During Cure of Unfilled and
Filled VCE With Hylene MP

Temperature (°C)	Cure Time (Min)	Storage Shear Modulus, G' (MPa)	$\ln \frac{G'_f}{G'_f - G'_t}$
Filled			
178	1	0.133	0.24
	2	0.573	2.10
	3	0.617	3.56
	4	0.633	5.76
	5	0.635	
165	1	0.013	0.02
	2	0.323	0.65
	3	0.532	1.56
	4	0.616	2.45
	5	0.648	3.26
	6	0.662	4.03
	9	0.674	
150	3	0.058	0.07
	5	0.409	0.65
	10	0.761	2.20
	15	0.835	3.70
	20	0.853	5.66
	25	0.856	
135	10	0.117	0.13
	15	0.344	0.46
	35	0.798	1.86
	40	0.839	2.22
	50	0.894	3.06
	60	0.926	4.14
	70	0.931	4.54
	80	0.941	

$$\ln k = \ln A - E_a/RT$$

(3)

where

k = rate constant,

A = Arrhenius constant (frequency factor),

Figure 18. Arrhenius Plot for the Cure of Unfilled and Filled VCE/Hylene MP

E_a = activation energy,
 R = gas constant, and
 T = absolute temperature.

As seen in the Arrhenius plots, an excellent linear relationship is observed. The rate constants at each temperature and the data for the Arrhenius plots are given in Table 10. The activation energy for the unfilled and filled VCE formulations were calculated to be 27.1 and 29.3 kcal/mol, respectively; the frequency factors for the unfilled and filled formulations were calculated to be 2.84×10^{13} and 3.02×10^{14} , respectively. These are reasonable values for this type of reaction, and the difference in the values for the two systems is probably not significant.

The reproducibility of the technique was evaluated by making four replicate runs on the unfilled VCE formulation at 150°C and six replicate runs at 178°C. The latter should be the least reproducible because of the VCE/Hylene MP reaction, which is completed in about 3 minutes. The standard deviation of these replicate determinations is given in Table 10.

Replicate determinations were not made on the other specimens. Previous work has indicated good reproducibility of the method for these formulations; the highest coefficient of variation was 10 percent. The earlier work is not reported because of errors in some of the VCE/Hylene MP formulations.

The method developed will be applicable to cure-behavior studies of low modulus, crosslinked systems.

Dynamic Compressive Testing of Flexible Foams

Test Method

The dynamic mechanical properties of flexible foams, including storage compressive modulus (E'), loss compressive modulus (E''), and loss tangent (E''/E'), may be determined using the tension/compression fixture.

The tension/compression fixture is connected to the upper quill of the mechanical spectrometer. One 25-mm flat parallel plate is attached to the tension/compression fixture, and the other is attached to the air-bearing transducer assembly.

The upper plate is lowered until it just touches the bottom plate, and the sample height gage is set at zero. The test specimen and the free height of the specimen is determined by lowering the upper quill until the load on the specimen is 20 g, corresponding to a stress of 0.4 kPa.

Table 10. Rate Constants for the VCE/Hylene MP Reaction and Associated Data for the Arrhenius Plots in Figure 18

Formulation	Temperature (K)	Rate Constant k (min ⁻¹)	ln k
Unfilled	408	0.076	-2.57
	423	0.307 ±0.005	-1.18 ±0.02
	438	0.844	-0.17
	451	1.900 ±0.190	0.64 ±0.10
Filled	408	0.064	-2.75
	423	0.207	-1.57
	438	0.633	-0.46
	451	2.006	0.70

The upper quill is then lowered until some specific load or percent compression is achieved. Locking the upper quill changes the position of the quill slightly, and, consequently, the load changes. Not locking the upper quill markedly improved reproducibility of the method.

A small sinusoidal oscillatory deflection is then applied to the sample. Usually the oscillatory deflection ranged from ±0.003 to ±0.03 mm, which represents a corresponding range of ±0.15 to ±1.5 percent compression for a specimen with an initial height of 4 mm.

Equations 4 and 5 are used to calculate the compressive moduli.

$$E' = a \times \frac{H}{R^2} \times 1.545 \times 10^3 \quad (4)$$

$$E'' = b \times \frac{H}{R^2} \times 1.545 \times 10^3 \quad (5)$$

where

E' = storage compressive modulus (kPa),

E'' = loss compressive modulus (kPa),

a = "A" reading from the phase analyzer when mode is Y/X,
b = "B" reading from the phase analyzer when mode is Y/X,
H = specimen compressed height, and
R = radius of parallel plates.

Reproducibility of the Test Method and Effect of Varying the Formulation of B-3223 Cellular Silicone on the Dynamic Mechanical Properties

The reproducibility of the dynamic compressive test method was determined by repetitive testing of a single specimen and of several specimens from one slab of B-3223 cellular silicone.

Seven slabs of B-3223 were used in this study. Five discs, 28.6 mm in diameter, were cut from various locations on each of the seven slabs. The dynamic mechanical properties of each disc were measured at least three times.

The seven slabs of B-3223 were manufactured at Bendix from Y-1668, Lot 1100, which had been purchased from Rhodia. The slabs were prepared using differing amount of W-97 silicone gum blended into the Y-1668 to reduce its hardness, and differing amounts of peroxide crosslinking agent, TS-50. The slabs were designated numerically to indicate these two variables as follows. The first numbers (1, 2, 3, and 4) indicate 0, 5, 10, and 15 parts per hundred resin (phr) of W-97, respectively, had been added to the Y-1668. The second numbers (3.0, 3.5, 4.0, and 4.5) indicate the phr of TS-50 added. For example, slab 2-3.0 was made using 5 phr W-97 and 3.0 phr TS-50.

The reproducibility of measurements of storage compressive modulus, E' , and loss compressive modulus, E'' , on a given disc was determined first. At least three determinations were made on each disc (105 determinations total). The coefficient of variation of the determinations ranged from 1 to 2 percent. Next, the reproducibility of measurements of E' and E'' of five discs taken from the same slab was determined. The coefficient of variation in this case ranged from 2 to 4 percent, indicating good homogeneity in the slabs. These data are summarized in Table 11.

Very little change in E' and E'' was noted in B-3223 slabs with increasing amounts of TS-50 peroxide. An increase in E' between 3.0 and 3.5 phr TS-50 was seen, but the E' values for 3.5, 4.0, and 4.5 phr TS-50 are virtually the same. The number of vinyl groups in the silicone polymer is constant in this series. Consequently, there should be a concentration of TS-50 beyond which no further crosslinking (via vinyl groups) occurs. These data indicate 3.5 phr TS-50 is the optimum amount.

Table 11. Dynamic Storage and Loss Compressive Modulus of B-3223 Cellular Silicone at 25°C and Three Frequencies

	0.1 Hz		1.0 Hz		10.1 Hz	
	E' (kPa)	E'' (kPa)	E' (kPa)	E'' (kPa)	E' (kPa)	E'' (kPa)
B-3223 Slab						
1-3.0	847 ±21	35 ±5	906 ±13	42 ±12	970 ±15	114 ±20
2-3.0	712 ±20	26 ±1	752 ±16	31 ±8	812 ±20	74 ±9
3-3.0	685 ±14	22 ±1	714 ±16	25 ±7	772 ±38	63 ±1
4-3.0	515 ±7	20 ±3	552 ±13	39 ±11	628 ±13	47 ±13
1-3.5	898 ±26	30 ±1	934 ±12	29 ±6	984 ±27	119 ±11
1-4.0	919 ±18	28 ±1	934 ±36	23 ±2	981 ±38	116 ±6
1-4.5	910 ±1	30 ±2	934 ±11	25 ±2	983 ±10	119 ±4

A definite decrease in E' and a less definite decrease in E'' was seen in the slabs containing added W-97. This result is expected, because the addition of W-97 to Y-1668 changes the ratio of silicone polymer to silica reinforcing filler, and material with more W-97 would be softer.

With increasing frequency, E' and E'' increased slightly. Materials normally exhibit a greater frequency dependency than observed here; however, this result may be characteristic of the particular dynamic mechanical test used in this experiment.

Dynamic Compressive Moduli of Cellular Silicone as a Function of Temperature

A 29-mm-diameter disc of cellular silicone was placed under a compressive force of about 10 kPa (~2 percent compression). Then, a small sinusoidal oscillatory deflection of about ±0.15 percent was applied, and the maximum compressive stress and phase angle were measured. Dynamic mechanical test data were determined for B-3223 cellular silicone over a temperature range of -120 to 160°C.

Because of expansion and contraction of the test fixtures with change in temperature, it was necessary to re-zero the parallel plates at each test temperature used. The values of E', E'', and

loss tangent for B-3223 over a temperature range of -120 to 160°C and at frequencies of 0.1 and 1.0 Hz are given in Table 12, and a plot of this data for 0.1 Hz is given in Figure 19.

Determinations of E' and E'' were made at temperatures as low as -165°C; however, the values obtained were not realistic. For example, the E' from -135 to -165°C was about 3000 kPa, or half the value of that at -120°C. Above -120°C, it is believed the modulus of B-3223 is too high to be compressed enough to be greater than the small oscillatory compression applied to obtain dynamic properties. In other words, it is believed the upper platen breaks contact with the sample on the upward stroke.

As observed from the data, the loss modulus, E'' , is a maximum at -110°C and gradually decreases with increasing temperature. The storage modulus is nearly independent of temperature from -80 to 160°C, indicating the mechanical properties of B-3223 cellular silicone should be reasonably constant over the normal temperature excursions to which weapons may be subjected.

Load Deflection Testing of Cellular Silicone

Load deflection testing can be achieved using the tension/compression fixture if the mechanical spectrometer is operated in a steady rotation mode. This fixture translates counterclockwise rotational motion into a downward motion. The procedure is the following. The parallel plates are brought into contact with one another, and the height gage is set at zero. The sample, B-3223 cellular silicone in this case, is placed on the lower parallel plate, and the upper quill is lowered manually until the upper parallel plate just contacts the specimen (about 20 g force). The free height of the sample is determined from the height gage. The Z-axis signal conditioner is set at the maximum range (10,000 g full scale) and the recorder pen is set on the zero load position of the strip chart paper (Y-axis). The recorder is set at the desired speed, and, at an appropriate bench mark on the chart paper, the upper parallel plate is set in motion by manually switching the motor to the counterclockwise position. The speed at which the upper plate can be lowered can be varied from 4×10^{-5} to 4 cm/s. In this work, a speed of 1×10^{-3} cm/s was used. The motor can be immediately switched to the clockwise position when desired, resulting in an upward motion of the parallel plate, providing an unloading curve. In this work, the switch was made when the load reached 7000 g, which corresponds to a stress of 140 kPa and a compression of about 17 percent at 25°C for the sample used. If such testing is done at various temperatures, it is necessary to re-zero the parallel plates after they have equilibrated to the set temperature.

This technique was used to evaluate the load deflection properties of B-3223 cellular silicone over a temperature range of -120 to

Table 12. Dynamic Compressive Properties of B-3223 Cellular Silicone
at 0.1 and 1.0 Hz and Temperature Range of -120 to 160°C

Temperature (°C)	Storage Modulus, E' (kPa)		Loss Modulus, E'' (kPa)		Loss Tangent	
	0.1 Hz	1.0 Hz	0.1 Hz	1.0 Hz	0.1 Hz	1.0 Hz
-120	6010	5700	350	4	0.058	0.001
-110	4260	6630	1650	1750	0.385	0.264
-100	2140	3140	540	980	0.254	0.313
- 80	1270	1510	180	200	0.138	0.133
- 54	1060	1190	80	40	0.075	0.037
- 20	1040	1120	40	25	0.037	0.023
25	930	960	25	20	0.028	0.018
74	870	900	10	12	0.011	0.013
120	960	980	15	6	0.018	0.006
160	980	980	15	2	0.015	0.002

Figure 19. Dynamic Mechanical Properties of B-3223 Cellular Silicone as a Function of Temperature and at a Frequency of 0.1 Hz

200°C. The load deflection curves are given in Figure 20. A summary of the loads at the various temperatures and degrees of deflection is given in Table 13 and is plotted in Figure 21.

Several observations can be made from these data. One, the B-3223 cushion becomes more elastic (has a higher percent compression for a given load) as the temperature increases from -120 to -54°C. Then, as the temperature increases from -54 to 120°C the cushion becomes slightly less elastic. The load deflection curves at 160 and 200°C are identical to the curve at 120°C. The percentage compression at 140 kPa and at the various temperatures is plotted as a dashed line in Figure 21. The curve is a maximum at -54°C, and ranges from 18.4 to 13.7 percent deflection from -54 to 200°C.

Another observation is that the difference between the loading and unloading curve increases as the temperature is lowered from 200 to -110°C. Below -110°C, the loading and unloading curves are similar. The cellular material is presumably becoming a rigid, glassy polymer below -110°C. This behavior is consistent with the dynamic compressive moduli data presented in Table 12.

The loss tangent, sometimes referred to as the damping factor, is the ratio of E'' to E' and is proportional to the ratio of the energy lost to the energy stored during a mechanical action on a test specimen. The increase of the loss tangent with decreasing temperature, to -110°C, which is near the viscoelastic transition temperature, at least qualitatively explains why the difference in the loading and unloading curves increases with decreasing temperature.

A third observation from the data presented in Figure 21 is that the load-bearing properties of B-3223 at a given percent compression do not change a great deal over a very broad temperature range. In fact, from -80 to 200°C at 5 percent compression, the load varies only from 33 to 45 kPa. The slope of the plot of load with temperature increases slightly with increasing percent compression. For example, the load variation at 15 percent compression from -80 to 200°C varies from 125 to 155 kPa. The values at 200°C were estimated by extrapolation.

Dynamic Tensile Testing of Elastomers

Test Method

Dynamic tensile analysis can be performed on elastomeric materials using the tension/compression fixture. The technique is similar to dynamic compression testing, except that the sample is placed under a given tension before applying the sinusoidal oscillatory deflection.

Figure 20. Load Deflection Curves of B-3223 at Two Temperature Ranges

Figure 20 Continued. Load Deflection Curves of B-3223 at Two Temperature Ranges

Table 13. Load Deflection Properties of
B-3223 Over a Temperature Range
of -120 to 200°C

Temperature (°C)	Load (kPa) at Given Deflection (Percent)			Deflection at 140 kPa (Percent)
	5	10	15	
-120	-	-	-	1.7
-110	60	-	-	6.7
-100	52	123	-	11.5
- 80	33	81	125	16.7
- 55	39	82	117	18.4
- 20	38	79	118	17.9
25	37	84	125	16.9
74	41	89	133	15.8
120	40	95	-	14.0
160	44	98	-	14.1
200	45	100	-	13.7

A rectangular test specimen is clamped between fixtures similar to those used in the forced torsional pendulum geometry. In this particular case, the samples were approximately 2.5 by 1.3 by 0.2 cm. The initial length of the sample, L_0 (distance between upper and lower clamps), is measured with calipers after the sample is adjusted to be at zero stress by manually lowering or raising the upper quill. The height gage is set at zero, and then the sample is placed under tension to a given load (250 g in this case). The extended length, L , is determined by adding the height gage value to the initial length, L_0 . A small sinusoidal oscillatory deflection then is applied to the sample. In this work, the deflection was about ± 0.4 percent. Data were taken at 0.1 and 1.0 Hz. From the maximum tensile stress and the phase angle, the storage and loss tensile moduli and loss tangent are determined using the following equations.

Storage tensile modulus, E' , in kPa:

$$E' = \frac{aL}{WT} \times 4854. \quad (6)$$

Figure 21. Load Deflection Properties of B-3223 Cellular Silicone as a Function of Temperature

Loss Tensile Modulus, E'' , in kPa:

$$E'' = \frac{bL}{WT} \times 4854. \quad (7)$$

Loss Tangent, $\tan \delta$:

$$\tan \delta = E''/E'. \quad (8)$$

In Equations 6 through 8,

a = "A" reading from the phase analyzer,

b = "B" reading from the phase analyzer,
L = sample length in cm,
W = sample width in cm, and
T = sample thickness in cm.

It is somewhat difficult to assemble the test specimen in the tension/compression fixture to obtain an accurately reproducible initial sample length, L_0 . Consequently, an experiment was performed on a single sample of Silastic E, a solid silicone rubber formulation made by Dow Corning, in which L_0 was varied from 1.2 to 3.0 cm. The values of E' and E'' obtained (Table 14) are seen to be very nearly the same, regardless of the value of L_0 .

Reproducibility

The reproducibility of the test method was evaluated by testing five specimens from the same slab of Silastic E. Each specimen, approximately 2.5 by 1.27 by 0.2 cm, was mounted in the tension/compression fixture, was tested as described earlier, and the values of E' and E'' were determined. This process was repeated at least three times without removing the specimen from the fixture. The specimen was then removed from the fixture and remounted and the above process repeated. The specimen was removed, remounted, and retested as described for a third time.

The coefficient of variation for replicate runs in which the specimen was not remounted was found to be 0.6 percent, and that for replicate runs in which the specimen was remounted was 1.1 percent. The coefficient of variation of moduli values among the five test specimens from the same slab was 2.3 percent. These data indicate good reproducibility of the test method.

Dynamic Tensile Properties of Silastic E

To demonstrate the applicability of the dynamic tensile test method, the dynamic mechanical properties of samples of Silastic E with varying postcure conditions were determined. A single slab of Silastic E, 15.2 by 15.2 by 0.2 cm, was molded at 93°C for 1 hour. The slab was cut into squares, with sides approximately 2.5 cm in length. The square specimens were then subjected to various postcure conditions (both time and temperature). Five squares were subjected to each condition. Three of the squares were submerged in toluene for toluene swell ratio determinations. The two remaining squares were made available for dynamic tensile testing. Specimens, 1.27 cm wide, were cut from the squares for the testing, and were mounted so that the initial sample height

Table 14. Dynamic Tensile Properties of
Silastic E With Varying Initial
Sample Height, L_0

Initial Height (cm)	Extended Height* (cm)	Storage Modulus, E' , at 1.0 Hz (kPa)	Loss Modulus, E'' , at 1.0 Hz (kPa)
3.048	3.331	1000	45
2.522	2.744	1020	50
2.062	2.247	1012	48
1.219	1.327	1013	50

*Represents elongation of 109 percent.

was 1.0 to 1.5 cm. The various postcure conditions, dynamic mechanical test data, and toluene swell ratios of the test specimens are summarized in Table 15.

Toluene swell ratios were determined by weighing the square test specimen and then allowing it to swell in toluene at room temperature for 24 hours. The specimen was then removed from the toluene, and the excess toluene on the surface of the specimen quickly removed by blotting with a paper towel. The specimen was placed in a tared plastic, snap-cap bottle and the top snapped shut to minimize weight change in the specimen from evaporation of toluene. The time between removal of the specimen from the toluene and sealing in the tared plastic bottle was less than 10 s. The weight of the swollen specimen was determined, and then the weight of the specimen again was determined after removal of the solvent by heating at 120°C in a vacuum, 1 mm.

The toluene swell ratio (TSR) is the ratio of the volume of solvent absorbed to the volume of the test specimen and is calculated from Equation 9.

$$\text{TSR} = \frac{(W_2 - W_1)(D_t)}{W_2 D_s} \quad (9)$$

where

W_1 = weight of specimen after drying,

W_2 = weight of swollen specimen,

Table 15. Dynamic Tensile Properties and Toluene Swell Ratio of Silastic E Silicone Rubber With Various Cure Conditions

Post Cure Condition	Tensile Moduli at 1.0 Hz (MPa)		Toluene Swell Ratio*
	Storage, E'	Loss, E''	
None	950	47	1.449 $\pm$ 0.001
1 hr at 93°C	980	52	1.422 $\pm$ 0.001
5 hr at 93°C	1110 $\pm$ 16*	56 $\pm$ 3*	1.359 $\pm$ 0.001
1 hr at 149°C	1416 $\pm$ 18**	66 $\pm$ 1**	1.191 $\pm$ 0.006
3 hr at 149°C	1543	71	1.122 $\pm$ 0.004
6 hr at 149°C	1610	62	1.069 $\pm$ 0.001

*Average of three determinations

**Average of two determinations

D_t = density of toluene (0.866 g/cm³), and

D_s = density of specimen (1.12 g/cm³).

A toluene swell ratio can be calculated from Equation 9 in which W_1 is the initial weight of the specimen. Because 3 to 6 percent of unbound chemicals are extracted during the submersion in toluene, an accurate value for the volume of toluene absorbed can be obtained only from the weight of the specimen after removal of the toluene.

The toluene swell ratio is inversely proportional to the crosslink density of a polymer, and, according to the kinetic theory of rubber elasticity, the shear modulus (and tensile modulus) is directly proportional to the crosslink density. Consequently, the tensile modulus should be inversely proportional to the toluene swell ratio. A plot for the data in Table 15 is given in Figure 22. As observed, a very good inverse linear relationship exists, which lends support to the validity and utility of the dynamic tensile modulus test method developed.

Dynamic Mechanical Testing of Fibers

Dynamic tensile analysis can be performed on fibers using a method similar to that described for elastomeric materials. To

Figure 22. Relationship of Storage Tensile Modulus and Toluene Swell Ratio for Silastic E.

demonstrate the applicability of the method, several tests were performed on single filaments of Kevlar fiber.

Single filaments from Kevlar woven cloth were separated from strands and clamped between short sections of stainless steel. Initially, simple tensile strength tests were performed on the single filaments. Measurements of the ultimate tensile strength of single filaments were made at a strain rate of 2×10^{-3} cm/s. The initial height in each case, about 25 mm, was carefully measured with micrometers. The elongation at any point during the test was obtained from the predetermined relationship of extension and angular rotation.

Tensile strengths were obtained ranging from 2.6 to 3.6 GPa. The elongation at break was about 2 percent, and the diameter of the fibers was $10.2\text{ }\mu\text{m}$, as measured by a microscope. These values are in the proper range of those reported by others on bundles of filaments. The variation observed is probably caused by lack of a consistent procedure for handling and mounting the fibers. In fact, the mounting of the fibers for testing was found to be a major problem. In some cases, the fiber failure occurred at the clamping site, and in other cases failure occurred somewhere between clamping sites.

Although the emphasis in this project was not directed at traditional transient tests like tensile strength determinations, which can be done by other instruments, this work indicates such testing is feasible with the mechanical spectrometer.

Another experiment was run to evaluate dynamic testing of fibers under strain and at various frequencies. A single filament of Kevlar was clamped in the test fixture at an initial height of 2.965 cm. The fiber was then elongated to a height of 2.979 cm, which corresponds to 0.7 percent elongation. A sinusoidal vibrational strain was applied to the fiber at a selected frequency. The strain at the maximum of the amplitude was an additional 0.003 cm. Therefore, the fiber was elongated to a maximum of 2.982 cm (2.979 cm plus 0.003 cm) compared to the initial height. The minimum elongation was 2.976 cm (2.979 cm minus 0.003 cm). Frequencies were varied from 10^{-2} to 2 Hz. The results of this experiment (Table 16) indicate the stress behavior of the fiber increases very slightly with frequency.

A third experiment was run in which a single filament was mounted at an initial length of 2.837 cm. The fiber was elongated to 2.873 cm, which corresponds to 1.16 percent elongation, and then stretched in an oscillatory manner at a frequency of 0.2 Hz. The amplitude of the oscillation was varied, and the stress response was measured (Table 17). A plot of stress versus the additional elongation at the maximum of the oscillatory strain is given in Figure 23. As shown, Kevlar exhibits linear behavior over the range examined in testing of this type.

These experiments illustrate the type of dynamic mechanical testing which can be carried out on fibers. Of course, such testing can be done at any temperature between -160°C and 325°C . This capability, along with that of varying the frequency, can provide a good deal of fundamental dynamic mechanical property data on fibers.

Although the above testing was performed on single filaments with reasonable success, the problem of clamping filaments in the test fixture is significant. A better method of clamping is needed before this technique will be useful for testing fibers.

Table 16. Maximum Dynamic Tensile Stress of Kevlar-49 Single Filament as a Function of Frequency

Frequency (Hz)	Maximum Tensile Stress (MPa)
0.01	314
0.02	324
0.05	327
0.10	331
0.20	336
0.50	340
1.00	347
2.00	365

Table 17. Maximum Dynamic Tensile Stress of Kevlar-49 Single Filament as a Function of Elongation

Oscillatory Elongation (cm)	Additional Elongation at Maximum Amplitude (Percent)	Maximum Tensile Stress (MPa)
±0.003	0.16	208
±0.005	0.27	366
±0.008	0.43	551
±0.011	0.58	728

ACCOMPLISHMENTS

Test procedures, using the mechanical spectrometer in four different test geometries, have been evaluated for determining the dynamic mechanical properties of polymeric materials. The test geometries studied include forced torsional pendulum, oscillatory parallel plate, oscillatory compressive testing, and oscillatory tensile testing. These tests were demonstrated to be applicable to a wide variety of polymeric materials, such as very high modulus laminates; elastomers; rigid foams; flexible foams; and, with further development work, fibers. The test methods provide a new resource at Bendix for the preparation of material and process specifications, for diagnostic analysis of production problems, and for future plastics material and process development activities.

Figure 23. Tensile Stress of a Single Kevlar Filament as a Function of Vibrational Elongation in a Previously Elongated State

BDX-613-2405

TEST METHODS FOR THE DYNAMIC MECHANICAL
PROPERTIES OF POLYMERIC MATERIALS. G. K.
Baker, Final, June 1980

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PLASTICS: Dynamic Testing

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