2nd
12/27/78
25 cy. to NTIS

BDX-613-1913 (Rev.)

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM

By H. M. McIlroy

Published November 1978

Topical Report
J. E. Anderson, Project Leader

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

MASTER

Bendix Kansas City Division

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price: Microfiche $3.00
Paper Copy $4.50

BDX-613-1913 (Rev.)
Distribution Category UC-25

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM

By H. M. McIlroy

NOTICE
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Published November 1978

Topical Report
J. E. Anderson, Project Leader

Project Team:
G. T. Crow
R. J. McWhirter
F. E. Meisner
C. L. Long
B. G. Parker

Communications Services

Bendix Kansas City Division

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM

BDX-613-1913 (Rev.), Topical Report, Published November 1978

Prepared by H. M. McIlroy

The effects of cure and postcure conditions on the mechanical and thermal properties of a syntactic foam made from a polyimide resin and glass microbubbles were measured. Compressive properties at 25 and 150°C and tensile properties at 25°C are given for foams cured from 1.5 to 24 hours at 175 to 250°C. Thermal properties were measured using differential scanning calorimetry, thermomechanical analysis, dielectric spectroscopy, and thermogravimetric analysis.

WPC-jap

This report was prepared as an account of work sponsored by the United States Government. Neither the United States, nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, expressed or implied or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights.

The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141

A prime contractor with the United States Department of Energy under Contract Number DE-AC04-76-DP00613

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

CONTENTS

ILLUSTRATIONS

TABLES

SUMMARY

The syntactic foam described is a blend of polyimide resin and glass microbubbles. Syntactic foams were molded from 45/55 and 50/50 weight blends of resin/glass microbubbles (GMB) and given various cures and postcures. The goal was to measure the effects of cure time, cure temperature, postcure time, postcure temperature, and postcure atmosphere on mechanical and thermal properties.

The actual mold cures tested were from 1.5 to 8 hours at 190 to 250°C while the postcures were from 4 to 24 hours at 220 to 250°C in a retort with either air or argon atmospheres. Changing the cure and postcure conditions did not change the specimen density. The test specimens postcured in an air atmosphere did oxidize and darken, but any shrinkage or weight loss was not measurable.

The compressive and tensile properties of the foam are more a function of the foam density than of cure and postcure conditions. When tested at room temperature, even the green foams with a minimum cure of 1 hour at 190°C have the same crush strengths as the postcured foams. The 150°C compressive and the 25°C tensile tests did show the postcured foams to be stronger than green cured foam. However, the different postcure temperatures and times did not significantly influence the compressive or tensile properties.

Thermal properties were measured using Differential Scanning Calorimetery (DSC), Thermomechanical Analysis (TMA), Dielectric Spectroscopy (DS), and Thermogravimetric Analysis (TGA). By the TGA weight loss method, no change in foams was measured as a function of postcure. Dielectric spectroscopy was not effective with the foams because of the high GMB content. With the resin only, the cure times by DS measurements ranged from 1 hour at 250°C to 7 hours at 175°C. The TMA and DSC methods both show that some time at 245°C is necessary to completely cure the resins. Cure times longer than 5 hours at 220°C probably would cure the resin completely, but no tests were made.

Syntactic foams made from Kerimid 601 resin develop strength and thermal resistance with only minimum cures. In order for the foam to develop full properties within reasonable times, a postcure is necessary. For the 150°C maximum use temperature of the intended application, a mold cure of 5 hours at 190°C without any postcure is adequate but does not take into account slight variations because of changes in resin lots. A cure of 1 hour at 190°C in the mold followed by a 1 hour minimum postcure at 245°C (foam time and temperature) is conservative but will allow for variations in raw materials.

DISCUSSION

SCOPE AND PURPOSE

This effort was recommended by the resin manufacturer to determine if the time required for the present cure cycle could be reduced without adversely affecting the foam properties.

PRIOR WORK

The polyimide resin binder for this foam is also the binder resin used for another thermally conductive foam. Early compressive test data and weight loss data from foams made with resin and carbon microbubbles indicated that the foam achieves high strengths and thermal stability with only abbreviated cures.

The manufacturer's recommended cure is a melting step at 120°C plus an initial cure at 180°C for at least 1 hour. Postcures of 24 hours at 250°C or 48 hours at 200°C are recommended to achieve full properties.

ACTIVITY

Materials

The polyimide resin is a fully imidized powder that cures by an addition reaction without evolving volatiles during cure. The resin is well suited as a binder for a syntactic foam since the resin powder softens with some flow around the filler prior to cure. The physical and thermal properties have been previously reported.[1,2,3]

The glass microbubbles have a density of 0.30 to 0.35 g/cm^3 and a particle size of 10 to 100 μm. As part of the manufacturing process, the bubbles are floated to remove broken GMB and also coated with a coupling agent to improve adhesion with an organic resin. Other properties of the glass microbubbles have been reported.[4,5]

Test Specimens

Two blends of the resin and GMB were prepared. The formulations tested were 50/50 and 45/55 weight blends of Resin/GMB.

Test blocks with 215 by 215 by 38 mm and 127 by 127 by 38 mm dimensions were molded to a 0.38 g/cm^3 density from the 45/55 blend. Individual test specimens (25.4 by 25.4 by 25.4 mm) were machined from the blocks.

A standard billet was molded to 0.38 g/cm^3 from the 50/50 blend of materials. Individual test specimens were machined from the billet (28.7 mm diameter by 25.4 mm thick). These cylindrical test specimens were maintained at below 15 percent relative humidity (R.H.) prior to testing, but the cubic specimens were stored at ambient conditions with no control on the R.H.

Cure Schedule

The standard cure for this foam is a melt at 120°C for about 1 hour, an initial or "green" cure at 190°C for 1 hour, and a postcure in an inert gas of 16 hours at 250°C. For this study, the melt time and temperature were constant. The initial cure was varied from 1.5 to 8 hours at 190 to 250°C in the mold. The postcure times and temperatures were from 4 to 24 hours at 220 to 250°C in a retort with either an air or argon atmosphere.

Tests and Test Methods

The compressive properties of the foam were measured using both the cylindrical and cubic test specimens at 1.3 mm/min. The cubic test specimens were not maintained in a controlled relative humidity area but were exposed to air at about 50 percent R.H. These specimens were tested unrestrained with the compressive strength taken as the yield point or ultimate value. The cylindrical specimens were in equilibrium with 15 percent R.H. (maximum). A restraining fixture was used to obtain the crush strength between 13 and 20 percent strain.

Tensile strength was measured on the cylindrical specimens from the molded billet. Prior to testing, the specimens were bonded to aluminum test fixture plugs. The crosshead speed was 1.3 mm/min. The foam density and density distribution values were obtained from weights and measurements of the individual specimens. Block identification and location of individual specimens within a block were maintained.

A dielectric analyzer was used to monitor the changes in capacitance and dissipation factor during the cure. The dielectric analyzer is based upon the principle that the dipoles within dielectric material respond to an electric field. The ability of the dipoles to respond and align with an electric field depends

upon factors, such as molecular structure, viscosity, and temperature. Dielectric spectroscopy (DS) is widely used to monitor the cure of thermoset materials.[6,7,8] For these tests, 50 by 50 mm aluminum electrodes and a specimen thickness of 1.3 mm were used. The heating rate was 5°C/min. with hold times as required.

Another method used to measure the degree or extent of cure is Differential Scanning Calorimetry (DSC). A thermal analyzer with a DSC attachment was used. DSC equipment measures the difference in temperature between a reference and a sample as a function of temperature and time. Differences in temperature between the reference and sample indicate exothermic or endothermic reactions in the sample.[9,10] The sensitivity was 0.0329 mw/mm (0.2 mcal/s-in.) with a 10 to 20 mg sample at 10°C/min. heating rate in nitrogen.

The Thermal Mechanical Analyzer (TMA) attachment was used to measure a transition in the foam with different postcures. Both penetration and expansion modes were run at 5 and 10°C/min. Weight loss measurements were made with the Thermogravimetric Analysis (TGA) attachment at 10°C/min.

Discussion of Results

Density Distribution

One concern was the effect of postcure time, temperature, and atmosphere on the density of the individual test specimens. One group of test specimens was weighed and measured after machining and again after the specimens were postcured. The change in density with postcure is shown in Figure 1. The control samples did not receive a postcure but were weighed and measured twice and at the same time as the other test specimens. The postcuring had no effect on the density.

All the sets of specimens postcured in air were dark brown to black depending upon the postcure time as a result of oxidation. Each level in Figure 1 is the average of five test specimens. Each of the 70 test specimens including the control specimens had gained weight after the postcure. As mentioned previously this group of specimens was not maintained in a controlled environment and absorbed water in the higher relative humidity of the testing area.

The other set of specimens (cylinders) was under controlled relative humidity of 15 percent maximum from the time the billet was molded until the specimens were tested. These 50 specimens had no net change in weight distribution because of postcure.

Based upon these results the postcure condition of 245°C for as much as 24 hours is not detrimental and does not cause a weight

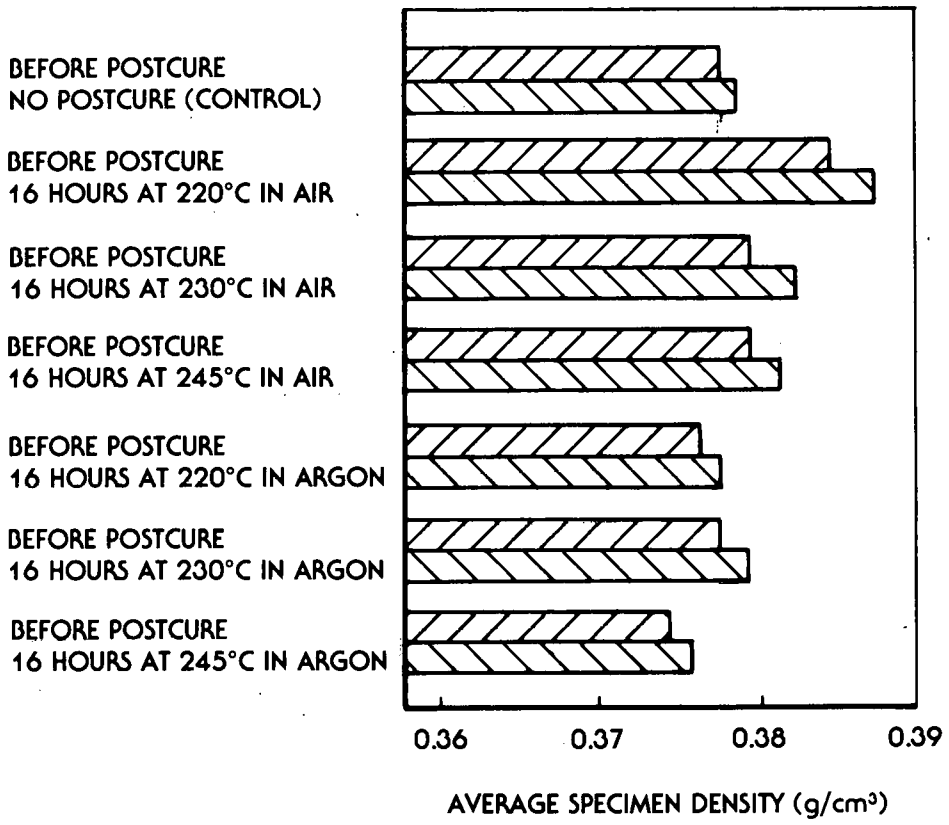

Figure 1. Effect of Postcure on Foam Density

loss in the material. In air, the foam definitely oxidizes and turns dark, but the oxidation does not result in a measurable weight loss.

Compressive Properties

The compressive properties of the foam as a function of postcure conditions were measured at room temperature for all the sets or test levels in Table 1. The intent was to test a constant density foam of 0.38 g/cm^3 and to determine the effects of postcure atmosphere, cure temperature, cure time, postcure temperature, and postcure time on the compressive properties of the foam. However, the density variation in the molded blocks was as much as 5 percent and the density from top to bottom of the billet was 8 to 10 percent.

Figure 2 shows that the compressive strength of the foam is a power function of the density. The data for Figure 2 are crush strength values of 45/55 and 50/50 weight blends of Resin/GMB molded to densities from 0.32 to 0.43 g/cm^3. Each data point is an average of five individual test values. The equation for the curve that fits the data is:

Table 1. Cure and Postcure Levels

Mold Cure at 190°C (Hours)	Postcure			Temperature Test (°C)
	Temperature (°C)	Time (Hours)	Atmosphere	
1.5				25 and 50
1.5	220	16	Air	25
1.5	230	16	Air	25
1.5	245	16	Air	25 and 50
1.5	220	16	Argon	25
1.5	230	16	Argon	25
1.5	245	16	Argon	25 and 50
1.5				25
1.5	245	4		25
1.5	245	8	Air	25 and 50
1.5	245	16	Air	25
1.5	245	24	Air	25
1.5	245	4	Air	25 and 50
1.5	245	8	Argon	25 and 50
1.5	245	16	Argon	25
1.5	245	24	Argon	25 and 50
1.5				25 and 50
4				25 and 50
8				25 and 50
4*				25 and 50
4**				25 and 50
1				25 and 50
1	190	5	Argon	25 and 50
1	220	5	Argon	25 and 50
1	245	5	Argon	25 and 50
1	245	16	Argon	25 and 50

*220°C Mold Cure
**245°C Mold Cure

$y = 3.93 \times 10^4 \ DEN^{2.86}$

where

y = compressive strength in psi and

DEN = foam density in g/cm^3.

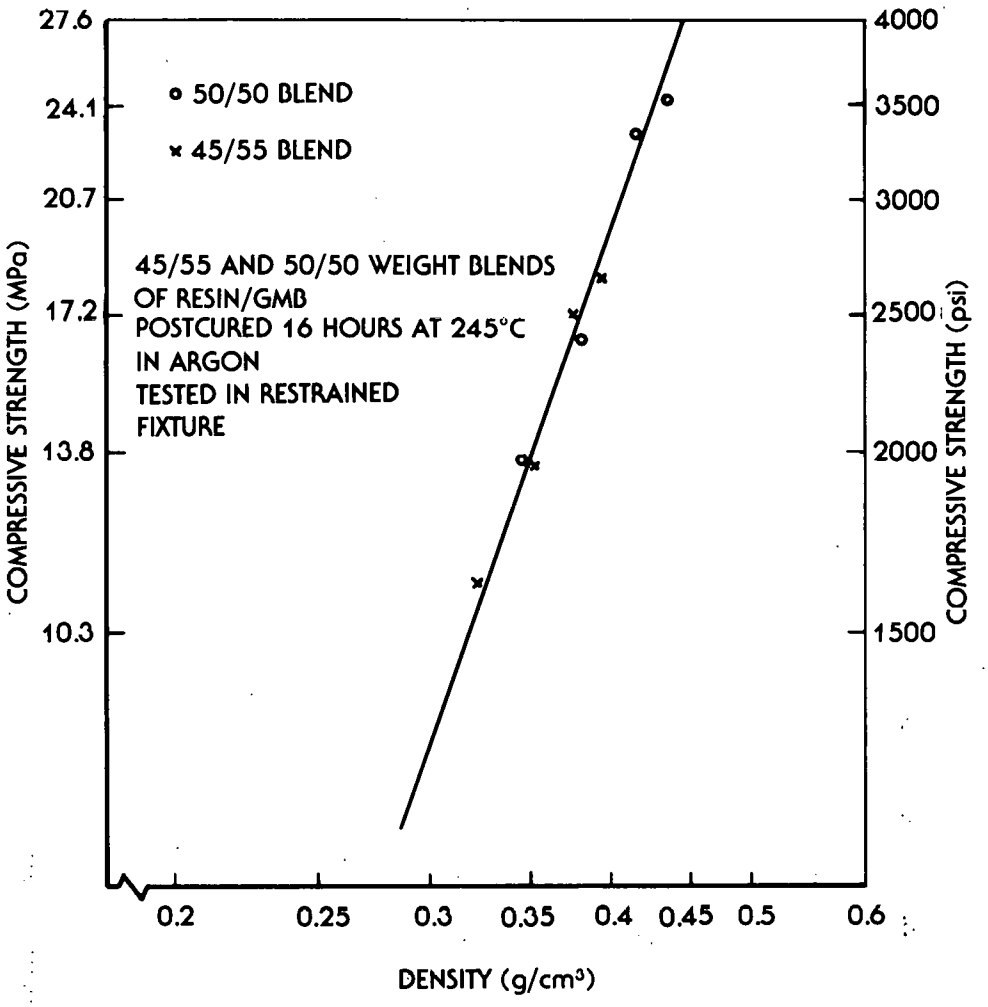

Figure 2. Effect of Foam Density on Compressive Strength

To obtain the compressive strength values in MPa the equation is:

$y = 2.71 \times 10^{8}\ DEN^{2.86}$.

Small changes in density can have a major effect on the foam properties, particularly the compressive strength. Test results for the compressive strengths are given in Table 2 for room temperature tests and in Table 3 for elevated temperature (150°C) tests. The values in Table 2 are normalized to a 0.38 g/cm^3 using the 2.86 exponent.

The data in Tables 2 and 3 show no significant difference in compressive strengths of the foam as a result of the postcure time or temperature. Postcuring in air seems to reduce the crush strength although the tested values are still within an expected range of values for the sets postcured in argon.

Table 2. Compressive Strengths of Postcured Foams Tested at 25°C

Postcure			Specimen Density (g/cm^3)	Compressive Strength			
				Measured		Normalized to 0.38 g/cm^3	
Time (Hours)	Temperature (°C)	Atmosphere		(MPa)	(psi)	(MPa)	(psi)
0			0.377	18.7	(2720)	19.2	(2780)
0			0.372	11.9	(1730)	12.7	(1840)
0			0.379	17.5	(2540)	17.6	(2560)
0*			0.393	17.8	(2590)	16.2	(2350)
0*			0.391	17.9	(2600)	16.5	(2400)
4	245	Air	0.371	15.9	(2290)	16.9	(2450)
8	245	Air	0.372	15.4	(2240)	16.4	(2380)
16	245	Air	0.371	14.8	(2150)	15.8	(2300)
24	245	Air	0.366	14.8	(2150)	16.5	(2390)
4	245	Argon	0.366	15.4	(2240)	17.2	(2490)
8	245	Argon	0.366	15.9	(2260)	17.4	(2520)
16	245	Argon	0.368	15.6	(2270)	17.2	(2490)
24	245	Argon	0.369	16.2	(2350)	17.6	(2560)
16	220	Air	0.388	17.9	(2600)	17.0	(2450)
16	230	Air	0.383	17.7	(2570)	17.3	(2510)
16	245	Air	0.382	17.6	(2550)	17.3	(2510)
16	220	Argon	0.378	17.7	(2570)	18.0	(2610)
16	230	Argon	0.380	18	(2610)	18.0	(2610)
16	245	Argon	0.376	16.5	(2400)	17.0	(2470)

Table 2 continued. Compressive Strengths of Postcured Foams Tested at 25°C

Postcure			Specimen	Compressive Strength			
				Measured		Normalized to 0.38 g/cm^3	
Time (Hours)	Temperature (°C)	Atmosphere	Density (g/cm^3)	(MPa)	(psi)	(MPa)	(psi)
5*	190	Argon	0.394	19.5	(2830)	17.6	(2550)
5*	220	Argon	0.390	19.4	(2820)	18.1	(2620)
5*	245	Argon	0.390	18.7	(2720)	17.4	(2520)
16*	245	Argon	0.393	19.3	(2800)	17.5	(2540)

*Specimens (cylinder) cured for 1 hour at 190°C in mold. All other specimens (cubic) cured for 1.5 hours at 190°C in mold.

All values are an average of five individual tests.

Table 3. Compressive Strengths of Postcured Foams Tested at 150°C

Postcure			Average Specimen Density	Average Compressive Strength	
Time (Hours	Temperature (°C)	Atmosphere	(g/cm^3)	(MPa)	(psi)
			0.377	17.5	(2540)
			0.374	18.5	(2690)
*			0.391	17.9	(2600)
*			0.393	17.9	(2590)
4	245	Air	0.370	15.8	(2300)
76	245	Air	0.377	16.5	(2400)
24	245	Air	0.366	14.8	(2140)
4	245	Argon	0.367	15.7	(2280)
16	245	Argon	0.383	18.3	(2650)
24	245	Argon	0.390	16.3	(2370)
5*	190	Argon	0.392	19.6	(2850)
5*	220	Argon	0.393	20.7	(3000)
5*	245	Argon	0.392	20.0	(2900)
16*	245	Argon	0.392	19.6	(2850)

*Specimens (cylinder) cured for 1 hour at 190°C in mold. All other specimens (cubic) cured for 1.5 hours at 190°C in mold.

All values are an average of five individual tests.

As mentioned previously, the data for the density change and compressive properties were taken from both cylinders and cubes tested at different times. The set of data marked with an asterisk in Tables 2 and 3 were taken from the molded billet with controlled relative humidity during the processing and storage prior to testing. These data of average values show a possible increase in strength with any postcure and a possible maximum crush strength with a postcure at 220°C.

All of the individual values for both the room temperature and elevated temperature tests of the cylindrical specimens are given in Figures 3 and 4. The test values for the room temperature test (Figure 3) show no significant difference in the effect of postcure on crush strength. The foam with the "green state" cure (1 hour at 190°C) is as strong as the postcure samples of foam. The effect of the minimum cure (green state) is more evident in

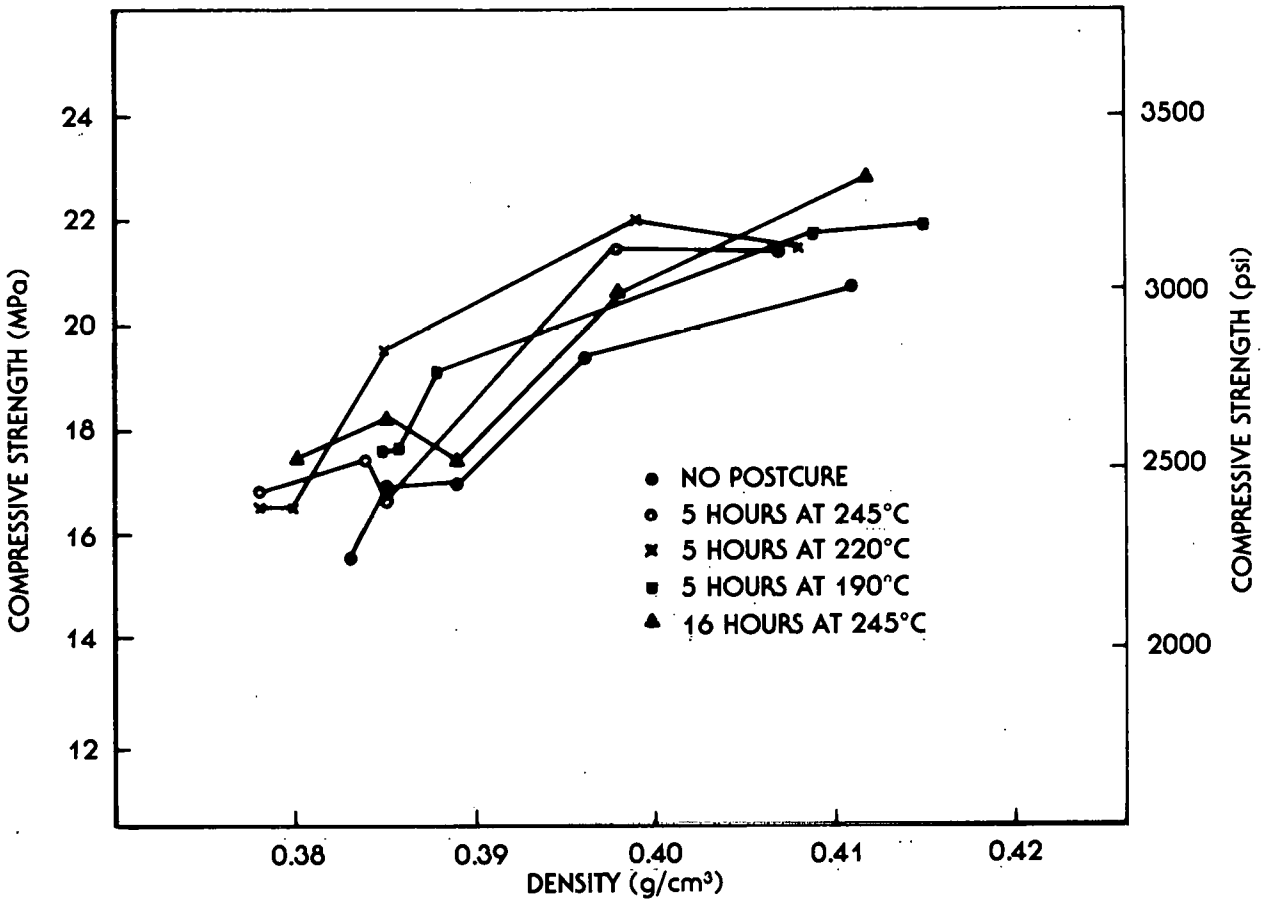

Figure 3. Compressive Strengths of Individual Specimens Tested at 25°C

the elevated temperature cure (Figure 4). The strength of the undercured foam is significantly less than the crush strength of the postcured foams, but the postcured foams all have equal crush strengths.

Tensile Properties

The data for the tensile modulus and strength at failure are listed in Table 4. The density of the actual test specimen bonded to the aluminum tensile plugs was not measured. The densities listed in Table 4 are the ranges of values from the 10 compressive test specimens. This data is arranged so that for each postcure condition, the first value is for a specimen taken from the top of the molded billet and the last value is for a specimen taken from the bottom of the billet. Data is not available to determine the tensile strength as a function of density but based upon the data in Table 4, the tensile strength is extremely sensitive to density changes. The minimum cure of

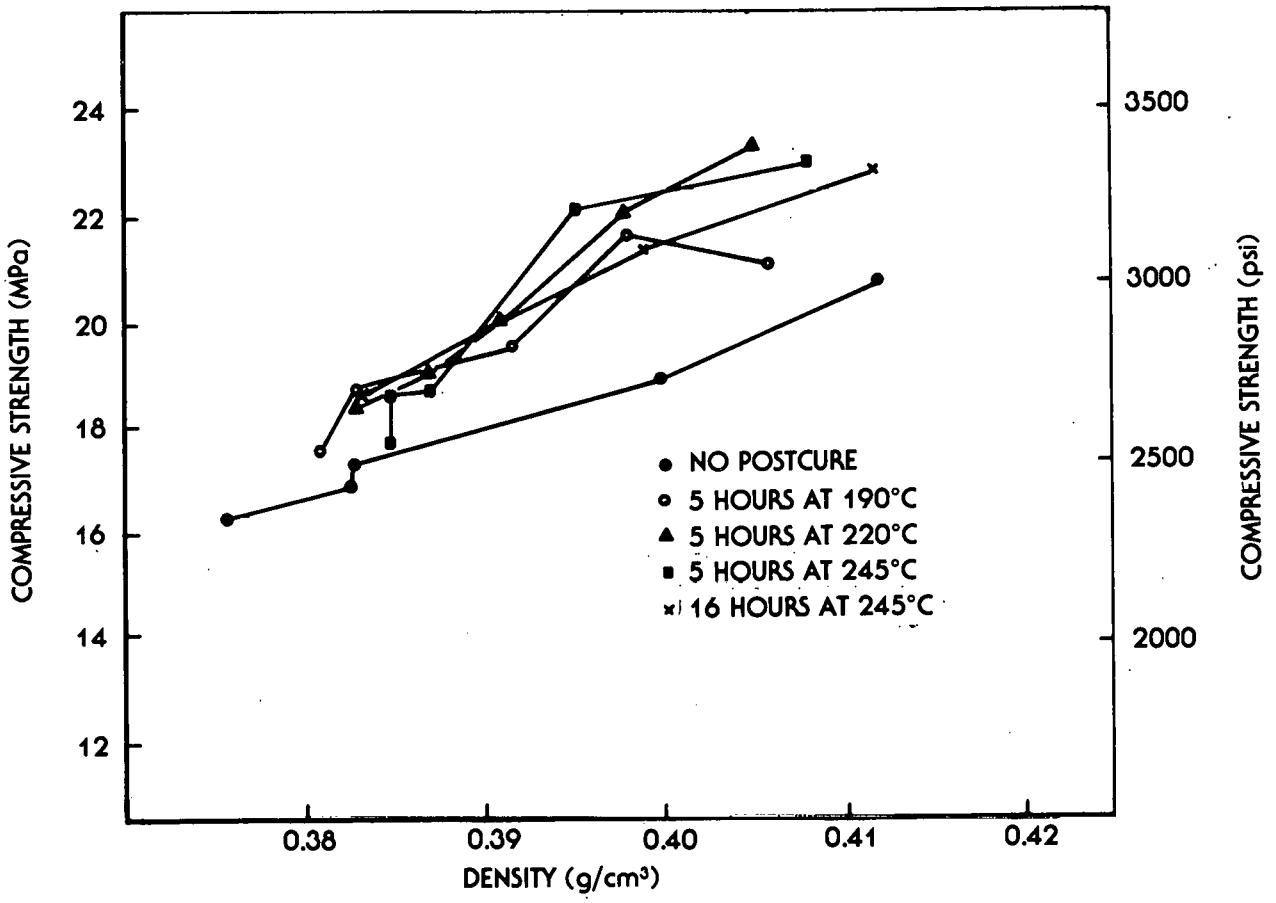

Figure 4. Compressive Strengths of Individual Specimens Tested at 150°C

1 hour at 190°C (no postcure) is not sufficient to develop the tensile strength of the foam. The postcured samples have similar tensile strength regardless of actual postcure.

Dielectric Spectroscopy

Dielectric spectra were made on each of the five syntactic foams with the different postcures. These spectra did show some minor differences between the samples, but overall there were no significant differences because of the postcure conditions. Although the dielectric properties of the glass microbubbles were considered constant, the spectra of the foams show unexpected and unexplained events. The masking of the changes in the resin is attributed to the high volume of GMB (65 percent) and low volume of resin (10 percent).

To circumvent the problem of not being able to separate the effect of the GMB and the resin in the spectrum, a series of

Table 4. Tensile Strengths of Foams Tested at 25°C

Postcure	Probable Density Range (g/cm^3)	Tensile Properties			
		Strength (MPa)	(psi)	Modulus (MPa)	(ksi)
No postcure	0.376 to 0.412	4.48	(650)	1910	(277)
		1.90	(275)	1875	(272)
		2.86	(415)	1379	(200)
		1.93	(280)	1062	(154)
		1.62	(235)	1365	(198)
		2.56*	(371)	1517*	(220)
5 Hours at 190°C	0.381 to 0.415	7.24	(1050)	2027	(294)
		4.76	(690)	1641	(238)
		6.76	(980)	1503	(218)
		3.38	(490)	1400	(203)
		2.27	(330)	1551	(225)
		4.88*	(708)	1627*	(236)
5 Hours at 220°C	0.380 to 0.408	8.62	(1250)	1875	(272)
		8.34	(1210)	1813	(263)
		6.34	(920)	1668	(242)
		6.69	(970)	1634	(237)
		7.49*	(1087)	1751*	(254)
5 Hours at 245°C	0.378 to 0.408	7.93	(1150)	1613	(234)
		7.52	(1090)	1875	(272)
		6.27	(910)	1489	(216)
		5.45	(790)	1551	(225)
		2.90	(420)	1351	(196)
		6.01*	(872)	1579*	(229)
16 Hours at 245°C	0.383 to 0.412	8.96	(1300)	2000	(290)
		8.69	(1260)	1600	(232)
		6.90	(1000)	1462	(212)
		4.34	(630)	1724	(250)
		2.72	(395)	1186	(172)
		6.32*	(917)	1593*	(231)

*Average

tests with resin only were made. For these test runs a heating rate of 5°C/min. and a hold of 30 minutes at 120°C was a constant to simulate the heating and melting phase of manufacturing the foam. After the 30 minutes at 120°C, the resin was cured by heating at 5°C/min. to either 250, 235, 220, 190, or 175°C and holding at the constant temperature

until the resin cured. In this case, cure is defined as that point in time when no change in dielectric properties are evident with an increase in temperature to 250°C. Spectra for the cures at 250 and 175°C are given in Figures 5 and 6.

The dissipation factor curves are the same for each run through the hold at 120°C. As the temperature is increased from 120°C, the dissipation factor increases rapidly to a maximum at 160 to 170°C. Figure 6 for the 250°C hold and cure temperature shows that after 2 hours an increase in temperature does not cause an increase in dissipation factor. However, for the cure at 175°C an increase in temperature to 250°C results in a significant increase in dissipation factor. At 175°C the resin does not cure for 7 to 8 hours. The cure times by this method are:

2 hours at 250°C,
3 hours at 235°C,
5 hours at 220°C,
6 hours at 190°C, and
7 hours at 175°C.

The cure times are minimum since the resin cures at a faster rate when the temperature is increased to check the degree of cure.

Weight Loss

The weight loss by thermogravimetric analysis (TGA) was measured as a function of postcure. For a heating rate of 10°C/min., the postcure conditions did not affect the thermal stability of the foam. Both the green cured foam and the foam cured for 16 hours at 245°C have an initial weight loss at about 355°C. A typical TGA curve for this Resin/GMB foam is given as Figure 7.

Degree of Cure

The degree of cure can be obtained from DSC data. Small differences in temperature between the reference and sample are a measure of heat liberated or absorbed in the sample. The area bounded by an exotherm or endotherm curve is related to the energy of the cure reaction and changes in area are a measure of degree of cure. The intent was to obtain numbers for the degree of cure, but the Resin cures rapidly and the difference in areas for the five postcures could not be measured accurately.

A typical DSC trace for Resin is shown in Figure 8 for curing resin from 15 to 340°C and the same sample after cure. The endotherm at about 80°C is not a melt point of the Resin powder but is possibly the glass transition of the partially reacted powder or vaporization of adsorbed water. The actual melting range is 90 to 110°C when measured by a capillary tube method or hotplate method. The melting is evidently very low energy and does not dominate the DSC trace.

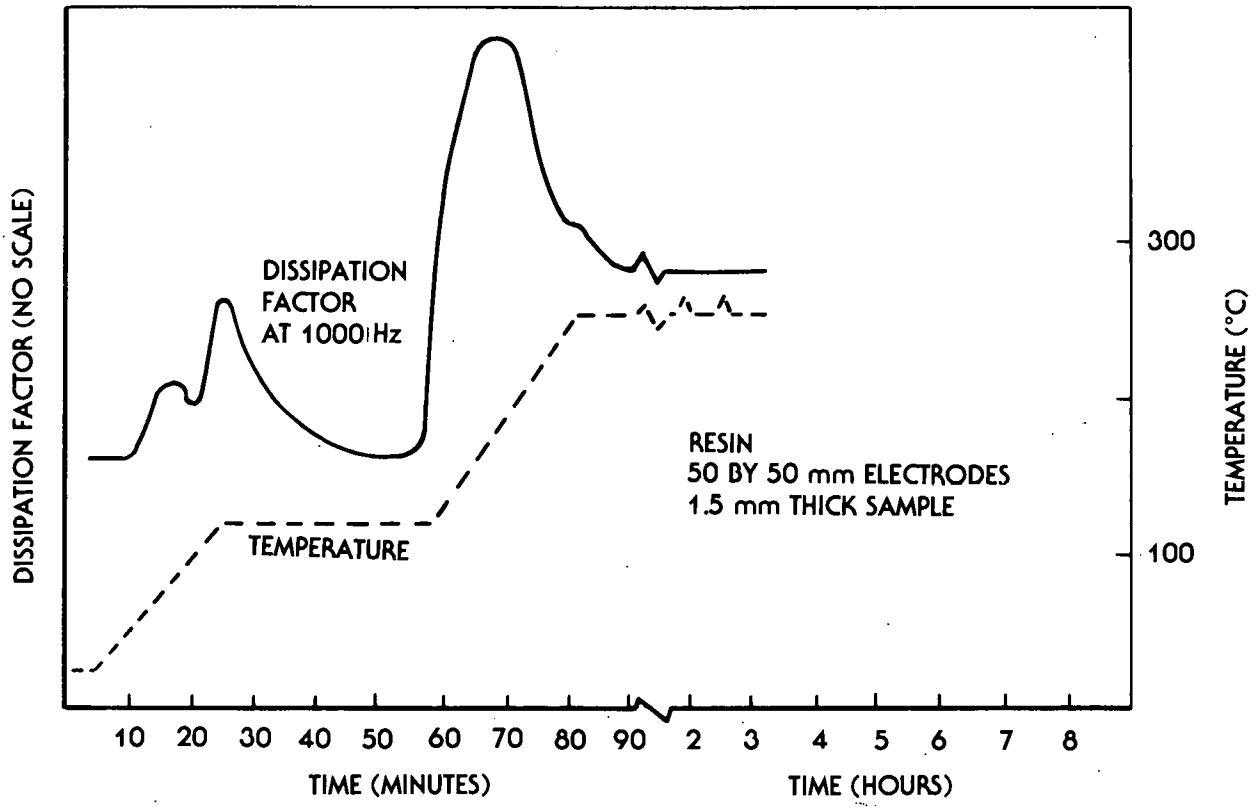

Figure 5. Dissipation Factor of Specimen Cured at 250°C

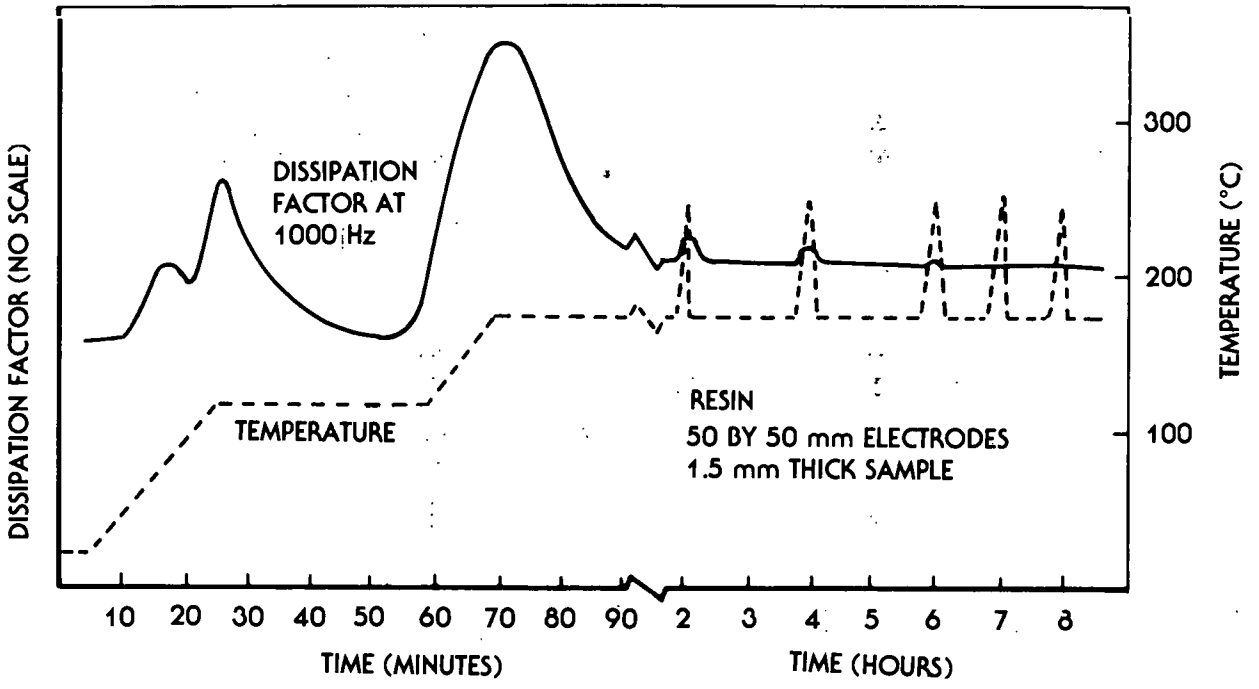

Figure 6. Dissipation Factor of Specimen Cured at 175°C

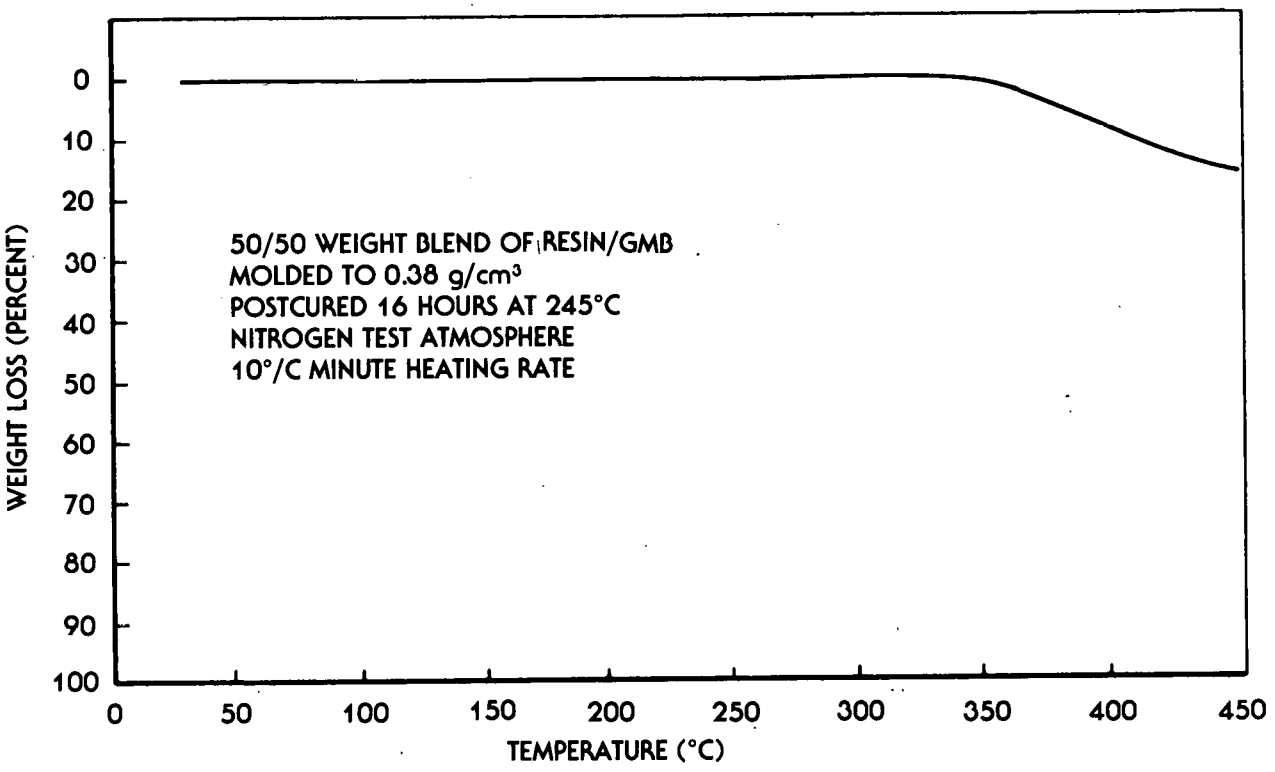

Figure 7. Weight Loss of Resin/GMB Syntactic Foam

DSC traces for the syntactic foam are given in Figure 9. A typical trace of the Resin is also given for comparison. A slight amount of exotherm is occurring for the foams with no postcure, 5 hours at 190°C, and 5 hours at 220°C postcure. No exotherm could be measured for foams cured at 245°C. As mentioned previously, the dielectric spectrum had no change (indicating a complete cure) at about 5 hours at 220°C. The information from the two methods is not inconsistant since the dielectric spectrum was made to 250°C and the DSC does not show an exotherm until about 265°C.

Transition Temperatures

Transition temperatures in the foam samples were measured by both the penetration and expansion modes of the TMA. A comparison of results from the two test methods in Table 5 shows excellent agreement between test methods. The expansion mode of the TMA was not set up to measure temperatures above 300°C and the degree of cure in the specimens postcured at 245°C was great enough to not indicate a transition. The transition temperature as measured by the penetration mode are shown in Figure 10. The transitions

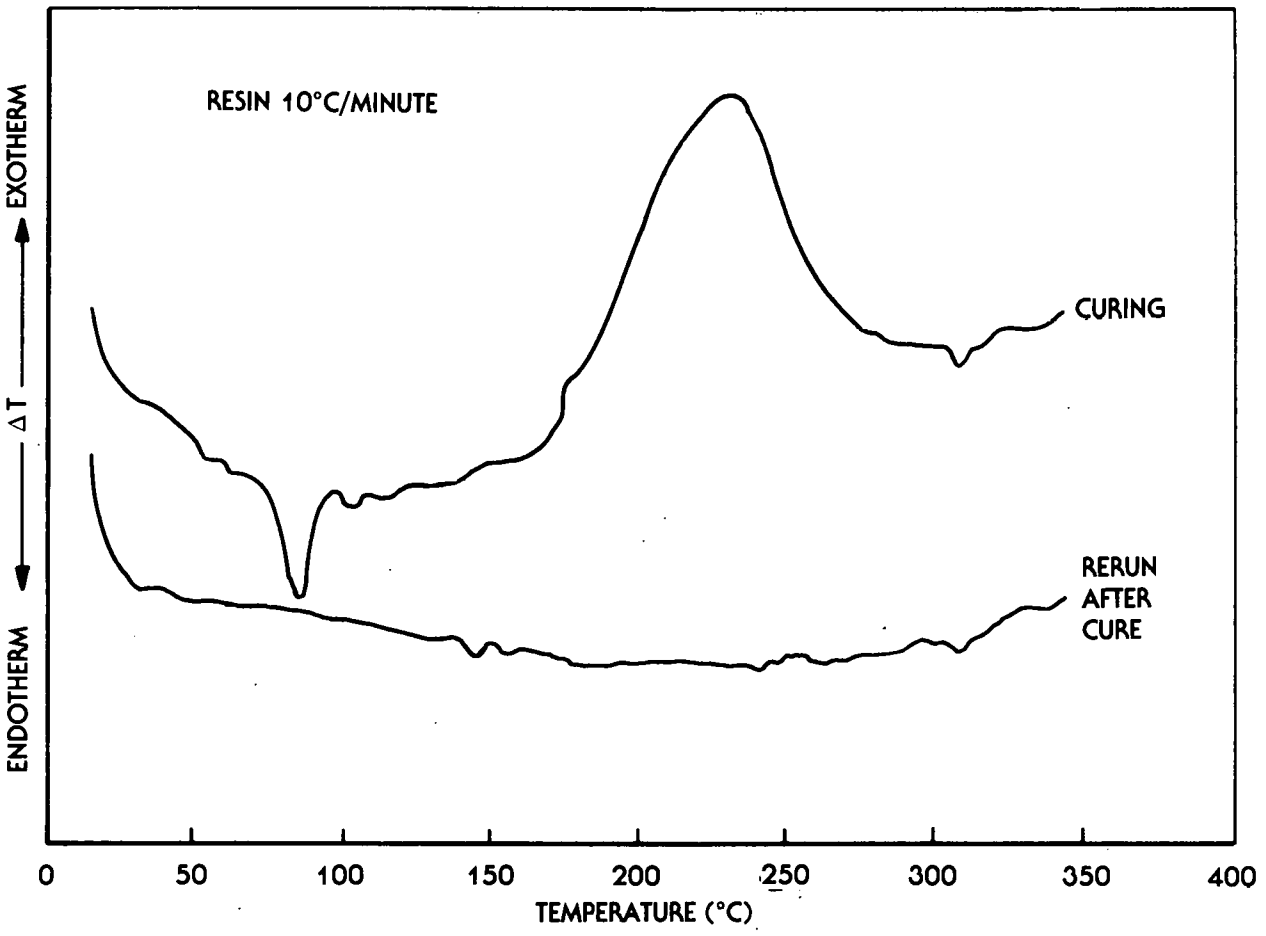

Figure 8. DSC Trace of Resin

for the foams with no postcure and those postcured at 190 and 220°C are a glass transition (Tg). The two foam samples postcured at 245°C do not have a Tg but rather start to soften because of the onset of thermal decomposition. Figure 11 shows the Tg and T softening of the foam with no postcure.

ACCOMPLISHMENTS

The cure cycle presently used to build development billets consists of a melt at 120°C, a mold cure for 1 hour at 190°C followed by a 16 hour retort postcure in an argon atmosphere. This investigation has shown that this cure cycle does give a complete cure. However, for the intended application, the postcure can be significantly reduced to save oven time and energy costs without affecting the foam properties.

Table 5. Transition Temperatures in Foams

Postcure	Transition Temperature	
	TMA Expansion* (°C)	TMA Penetration* (°C)
No postcure	172	180
5 Hours at 190°C	229	226
5 Hours at 220°C	256	250
5 Hours at 245°C		344
16 Hours at 245°C		340

*5 grams at 10°C/minute
**50 grams at 10°C/minute

Methods have been developed using the DSC, TMA, and DS techniques to follow the cure of the resin and to measure the degree of cure as a function of time and temperature.

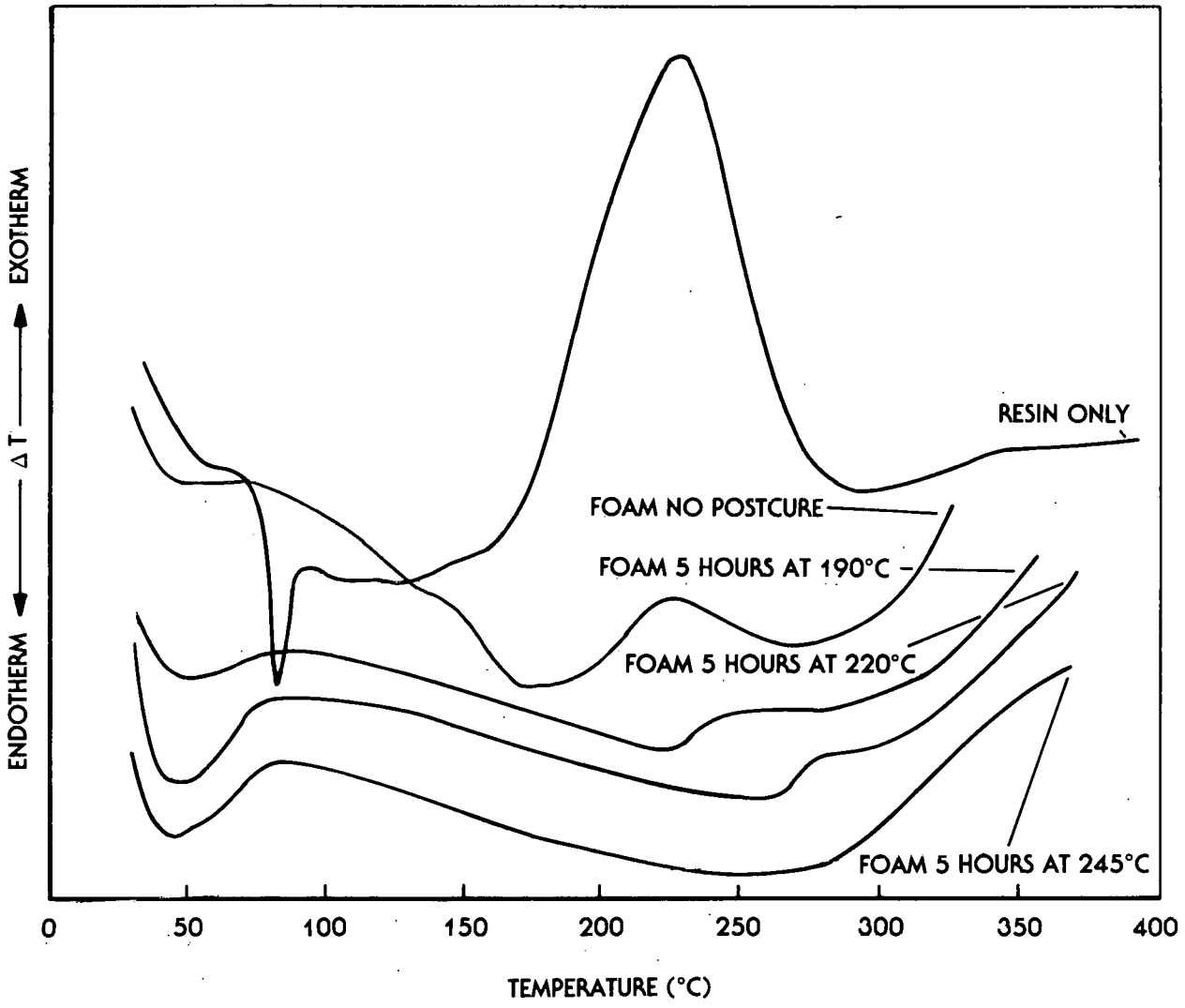

Figure 9. DSC Traces of Postcured Foams

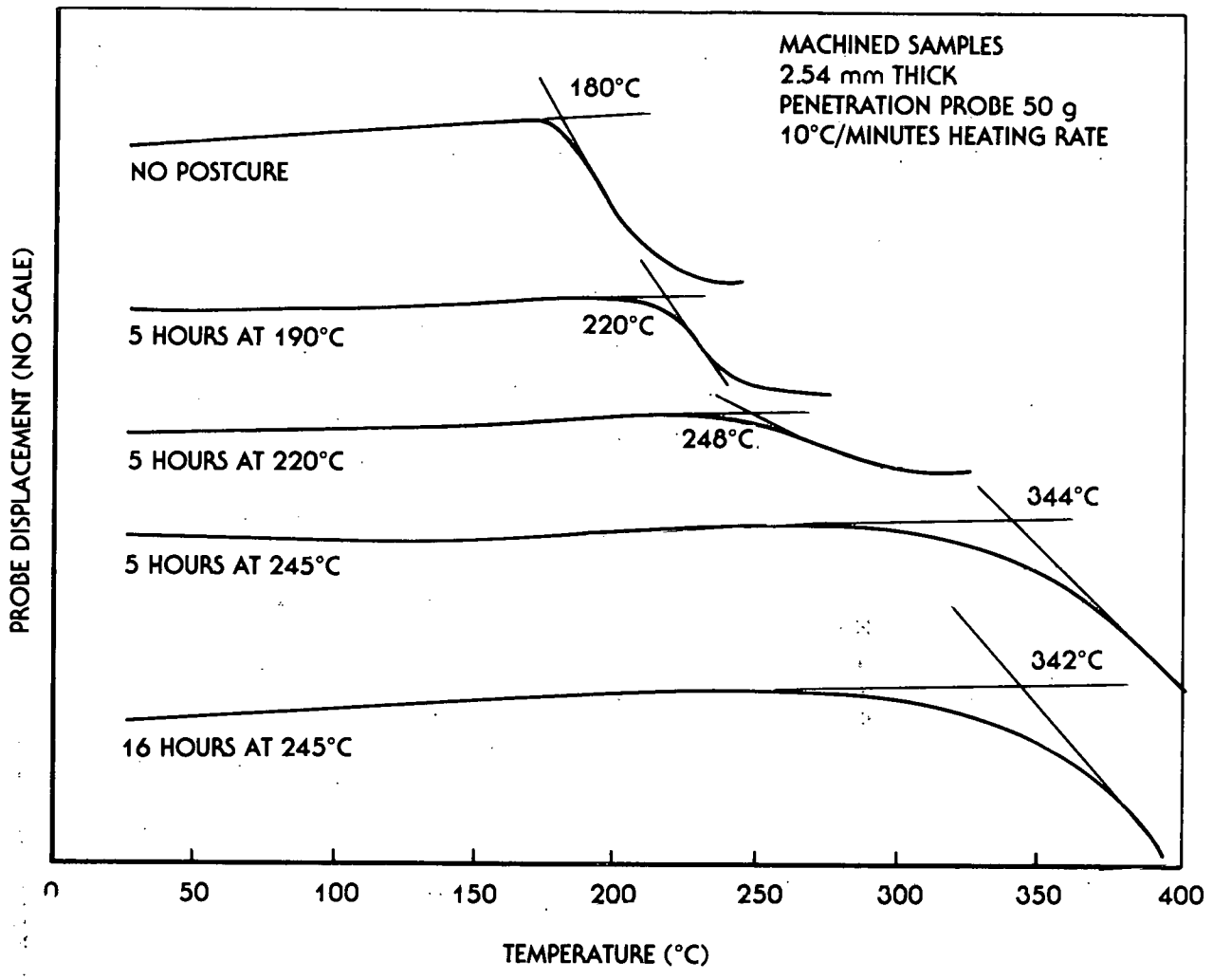

Figure 10. Transition Temperatures Using TMA

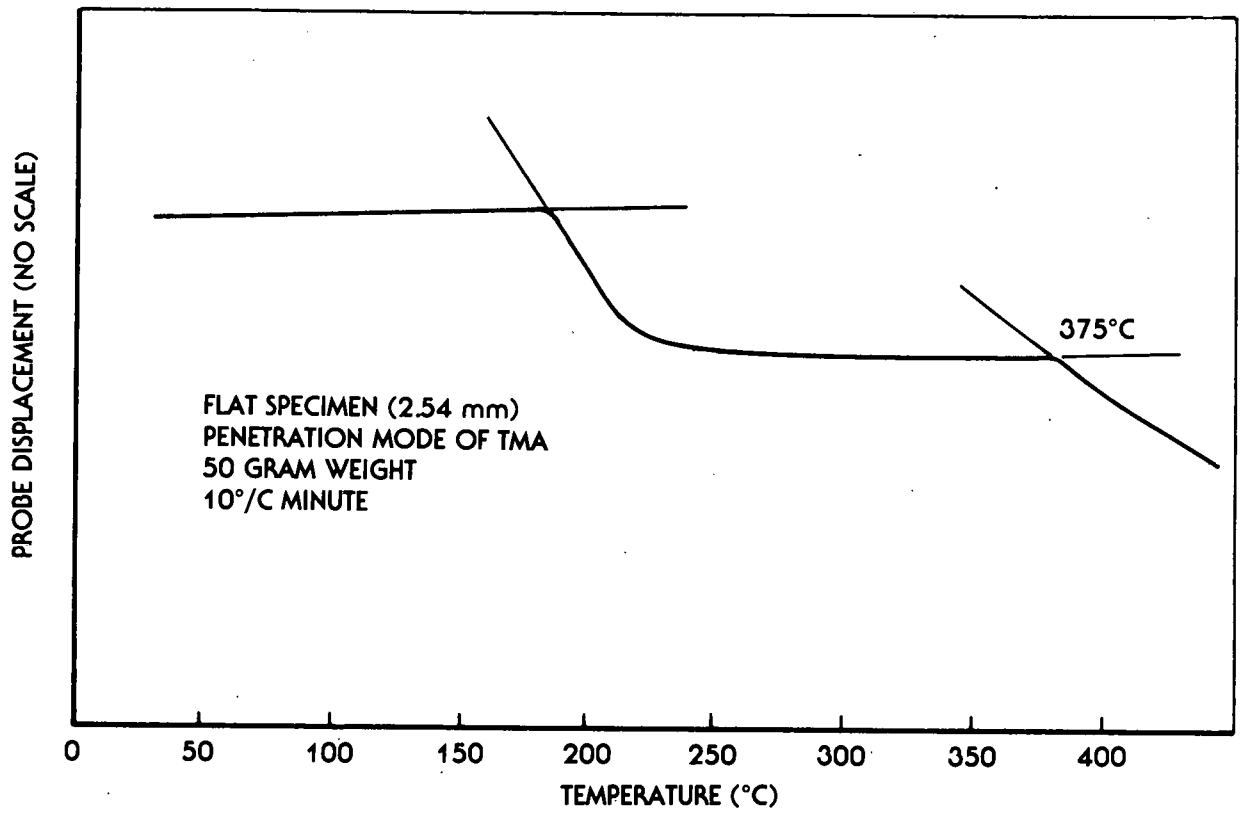

Figure 11. Transition Temperatures in Foam Without Postcure

REFERENCES

[1]M. J. Mallet, "Polyaminobismaleimide Resins," *Modern Plastics*, June, 1973, p. 78.

[2]F. P. Darmary, "Processable Polyimides," *American Chemical Society, Coatings and Plastics Preprints*, Los Angeles, 1974.

[3]H. M. McIlroy, *High Temperature Syntactic Foam* (Final Report). Bendix Kansas City: BDX-613-1534, July, 1977, (Available From NTIS).

[4]W. R. Bech and others, "Two and Three Phase Glass Bubble Composites," *SPE Journal*, Volume 25, Number 4, April 1969.

[5]H. M. McIlroy, *Glass Microbubble Composite Foams* (Topical Report). Bendix Kansas City: BDX-613-1596, February, 1978, (Available From NTIS).

[6]S. A. Yalof, "Probing Structure with Dielectric Spectroscopy," *Chemtech*, March, 1975, p. 165.

[7]J. M. Walker, *Dielectric Analysis of an Epoxy Preimpregnated Material* (Final Report). Bendix Kansas City: BDX-613-1396, January, 1976, (Available From NTIS).

[8]C. A. May, D. K. Whearty, and J. S. Fritzen, "Composite Cure Studies by Dielectric and Calorimetric Analysis," *Society for the Advancement of Material and Process Engineering*, San Diego, 1976, Vol. 21.

[9]L. T. Papalardo, "DSC Evaluation of Epoxy and Polyimide - Impregnated Laminates (Prepregs)," *Journal of Applied Polymer Science*, Volume 21, 1977, p. 809.

[10]T. E. Noll, "Evaluation of Epoxy Prepreg Using Differential Scanning Calorimetry," *IPC Technical Review*, May, 1977, p. 9.

DISTRIBUTION

	Copy
R. Bulcock, DOE-KCAO	1
K. C. Creed, G. E. Pinellas	2
F. W. Dubois, LASL	3
W. E. Cady, LLL	4
P. B. Rand, SLA	5
J. M. Brierly, SLL	6
D. W. Putz, SLL	7
J. E. Long, D/531, 1B46	8
H. T. Barnes, D/554, BD50	9-10
L. Stratton, D/554, 2C44	11-13
R. P. Frohmberg, D/800, 2A41	14
W. H. Deterding, D/814, 2C43	15
H. M. McIlroy, D/814, 2C43	16-20
F. E. Meisner, D/814, XD43	21
B. G. Parker, D/814, XD43	22
R. L. Sadler, D/814, 2C43	23
C. L. Long, D/816, SG-3	24
G. L. MacCurdy, D/816, SC-3	25
V. E. Alley, D/818, 2A31	26
J. E. Anderson, D/818, 2A31	27
G. T. Crow, D/818, 2A31	28
R. A. Daniel, D/818, 2A31	29
R. J. McWhirter, D/818, 2A31	30
R. E. Kessler, D/865, 2C40	31

BDX-613-1913 (Rev.)

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM, H. M. McIlroy, Topical, November 1978.

The effects of cure and postcure conditions on the mechanical and thermal properties of a syntactic foam made from a polyimide resin and glass microbubbles were measured. Compressive properties at 25 and 150°C and tensile properties at 25°C are given for foams cured from 1.5 to 24 hours at 175 to 250°C. Thermal properties were measured using differential scanning calorimetry, thermomechanical analysis, dielectric spectroscopy, and thermogravimetric analysis.

PLASTICS: Syntactic Foam Cure Cycles

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM, H. M. McIlroy, Topical, BDX-613-1913 (Rev.), November 1978.

The effects of cure and postcure conditions on the mechanical and thermal properties of a syntactic foam made from a polyimide resin and glass microbubbles were measured. Compressive properties at 25 and 150°C and tensile properties at 25°C are given for foams cured from 1.5 to 24 hours at 175 to 250°C. Thermal properties were measured using differential scanning calorimetry, thermomechanical analysis, dielectric spectroscopy, and thermogravimetric analysis.

EVALUATION OF CURE CYCLES FOR SYNTACTIC FOAM, H. M. McIlroy, Topical, BDX-613-1913 (Rev.), November 1978.

The effects of cure and postcure conditions on the mechanical and thermal properties of a syntactic foam made from a polyimide resin and glass microbubbles were measured. Compressive properties at 25 and 150°C and tensile properties at 25°C are given for foams cured from 1.5 to 24 hours at 175 to 250°C. Thermal properties were measured using differential scanning calorimetry, thermomechanical analysis, dielectric spectroscopy, and thermogravimetric analysis.