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PLASTEC REPORT 30

EFFECT OF LOW TEMPERATURE (0 TO -65°F) ON THE PROPERTIES OF PLASTICS

DEPARTMENT OF DEFENSE
UNITED STATES OF AMERICA

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EFFECT OF LOW TEMPERATURE (0 to -65°F) ON THE PROPERTIES OF PLASTICS

By

JOAN B. TITUS

July 1967

PLASTICS TECHNICAL EVALUATION CENTER
Picatinny Arsenal, Dover, New Jersey

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Harry E Pebly Jr

Harry E. Pebly, Jr., Director

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ABSTRACT

The effects of low temperature on the mechanical, electrical and thermal properties of plastics is discussed. Data are given where available at three temperatures; namely, low temperature, about -65°F, room temperature and around 160°F to permit complete evaluation. The material is presented by plastic family (in alphabetical order) and is divided into three parts: thermoplastics, thermosets and foams.

TABLE OF CONTENTS

PART I. THERMOPLASTICS

PART II. THERMOSETS

PART III. FOAMS

ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of Mrs. Barbara Rafer in the preparation of this report and the below companies who so generously cooperated in submitting data for inclusion in this report.

B. F. Goodrich Chemical Co.
Celanese Plastics Co.
Dow Chemical Co.
Eastman Chemical Products, Inc.
E. I. du Pont de Nemours & Co., Inc.
Fiberite Corp.
Furane Plastics, Inc.
General Electric Co.
Goodyear Tire & Rubber Co.
Hooker Chemical Corp.
Minnesota Mining & Manufacturing Co.
Mobay Chemical Co.
Phillips Petroleum Co.
Rohm & Haas Co.
Shell Chemical Co.
Westinghouse Electric Corp.

TRADE NAMES

For identification purposes the following is a list of trade names appearing in this report. The mention of a particular commercial product neither constitutes an endorsement by PLASTEC, nor a voucher for the accuracy of a manufacturer's claim unless specifically noted.

Absafil	Fiberfil, Inc.
Acrylafil	Fiberfil, Inc.
Alathon	E. I. duPont de Nemours & Co., Inc.
APCO 1414	Hexcel Products, Inc.
Bisoflex	The Distillers Co., Ltd.
Blue C Nylon	Monsanto Chemical Co.
Celcon	Celanese Plastics Co.
CTL-91-LD	Cincinnati Testing Research Labs
Crush-N-Foam	Hudson Foam Plastic Corp.
Cycolac	Marbon Chemical Co.
Cycolon	Marbon Chemical Co.
DC 2106	Dow Corning
Delrin	E. I. duPont de Nemours & Co., Inc.
DEN 438	The Dow Chemical Co.
DER-332	Dow Chemical Co.
DMP-30	Rohm & Haas Co.
DP-437, DP-125	Ciba Co., Ltd.
Durez	Hooker Chemical Corp.
Eccospheres	Emerson & Cuming, Inc.
Empol 3162-D, 1014	Emery Industries, Inc.
Epi-Cure	Jones Dabney Co.
Epi-Rez	Jones Dabney Co.
Epon	Shell Chemical Co.
Epotuf	Reichhold Chemicals, Inc.
ERL 2256	Bakelite, Union Carbide
Ethafoam	Dow Chemical Co.
Ethocel	Dow Chemical Co.
Fiberite	Fiberite Corp.
Geon	B. F. Goodrich Chemical Co.
Grace	W. R. Grace & Co.
H Film	E. I. duPont de Nemours & Co., Inc.
Harcure A	Wallace & Tiernan, Inc.
Hewitex	Hewitt-Robins, Inc.
High Grade	American Collo Corp.
HB-40	Monsanto Chemical Co.
K-1	Tennessee Eastman Co.
Kel-F	Minnesota Mining & Mfg. Co.
Kralastic	U.S. Rubber Co.
Kydex	Rohm & Haas Co.
LD-314	The Dayton Rubber Co.
Leecure A	Leepoxy Plastics
Lexan	General Electric Co.
Lockfoam BH 610-R	Nopco Chemical Co.
Lucite	E.I. duPont de Nemours & Co., Inc.
Merlon	Mobay Chemical Co.
Micarta	Westinghouse Electric Corp.
Montothene G50	Monsanto Chemical Co.
Mylar	E.I. duPont de Nemours & Co., Inc.
Nopcofoam	Nopco Chemical Co.
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Nylafil	Fiberfil, Inc.
Oleform	Avisun Corp.
Oxiron 2000	FMC Corp.
PAPI-1008	Carwin Corp.
P-2300	Union Carbide
Plastikon	The B. F. Goodrich Industrial Products Co.
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Polycarbafil	Fiberite Corp.
Polycel 420	Polytron Corp.
Polyfoam	The General Tire & Rubber Co.
Polymer-SP	E. I. duPont de Nemours & Co., Inc.
Resilo-Pak	Armstrong Cork Co.
Saran	Dow Chemical Co.
SC 1008	Monsanto Chemical Co.
Scotchcast	Minnesota Mining & Mfg. Co.
Scotchply	Minnesota Mining & Mfg. Co.
Selectron 5003	Pittsburgh Plate Glass Co.
Shell D	Shell Chemical Co.
Silastic 250	Dow Corning Corp.
Solithane 113	Thiokol Chemical Corp.
SP	E. I. duPont de Nemours & Co., Inc.
Spiralloy	Hercules Powder Co.
SR-100	Sheller Manufacturing Co.
SRP-C-114, 1104P	B. F. Goodrich
Stafoam	American Latex Products Corp.
Stycast	Emerson & Cuming, Inc.
Stypol 25	Freeman Chemical Corp.
Styrafil	Fiberfil, Inc.
Styrofoam	Dow Chemical Co.
Suprether Uniform	W. [illegible]. Burnett & Co., Inc.
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Tipersul	E. I. duPont de Nemours & Co., Inc.
Tyril	Dow Chemical Co.
Unox 201	Union Carbide Chemical Co.
Versamid 140	General Mills, Inc.
Vespel	E. I. duPont de Nemours & Co., Inc.
Vibrafoam	U. S. Rubber Co.
X226-32A	American Latex Corp.
X-2673.2	The Dow Chemical Co.
X-2674	The Dow Chemical Co.
XT500-XT250, XT150	American Cyanamid Co.
Zerlon	Dow Chemical Co.
Zytel	E. I. duPont de Nemours & Co., Inc.

INTRODUCTION

Plastics have a prominent place in the spectrum of materials used in the design and development of military materiel. This is due to their many attributes, such as cost, light weight, insulation capabilities, corrosion resistance, fabrication versatility, etc. Therefore a tremendous wealth of information is required to fulfill the engineering demands for today's advanced weaponry designs and breadth of materials operation.

Military materiel, from the simplest guerrilla infantry weapons to the most sophisticated space vehicles, must be capable of storage and operation in a wide temperature range. Since plastics are visco-elastic and therefore temperature sensitive, much research has been done and recorded on the effects of cryogenic and high temperatures on their properties. However, little work has been recorded in the lower limit temperature portion 0° to -80°F of the U. S. Army's "Research and Development of Materiel, Regulation 705-15," "Operation of Materiel Under Extreme Conditions of Environment."

Therefore, at the request of the military, PLASTEC has prepared this report to cover the effects of low temperatures, specifically 0° to -80°F, on the various properties of plastics. Since most materiel is not designed to function at one temperature, it would be impossible to evaluate a material at the low temperature alone. The author has attempted to include data at ambient and approximately 155°F (the Army's high temperature design requirement) for comparative purposes.

After completing a rather comprehensive literature search which included the PLASTEC Document Index, Engineering Index, Environmental Effects on Materials and Equipment Abstracts Index, Applied Science and Technology Index, Defense Documentation Center, International Aerospace Abstracts, OTS Selective Bibliography - "Low Temperature Research on Materials," and trade literature, the author found very little data in the low temperature range. This is attributed to two factors: (1) All searches are based on key words, and there was no specific key word to use in this case as "low temperature" has unfortunately become synonymous with "cryogenics." (2) Non-military applications do not require data to such low temperatures, therefore little interest is displayed in this area.

The author, in an attempt to obtain data, contacted plastics suppliers for information. This also resulted in little data, due to hesitancy to release data and lack of testing in this temperature range. Therefore, the following sections of this report contain all the information that could be found, even though incomplete, on the mechanical, electrical and thermal properties of plastics. Where no data exists on a specific property for a designated plastic, this should not be construed as an evaluation of this material at that temperature range. The omission of data is merely an indication that the author could find no data to include.

Much data could not be found in some areas because that property was not critical in many applications, and therefore little or no testing was accomplished. Again, the reader is reminded that the material presented is only that which could be found and that the data are typical values and are not to be used for specific design purposes.

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PART I - THERMOPLASTICS

SECTION I. ACETAL

Mechanical Properties

Table 1 and Figures 1 and 2 present average tensile elongation and impact values for various acetal compositions. Table 1 also compares regular molding materials with a Teflon-fiber filled material. As can be seen, the filled material is inferior in all respects to the regular molding material. Tensile strength increases moderately while percent elongation and impact strength decrease with lowering temperatures.

TABLE 1. MECHANICAL PROPERTIES OF ACETAL COMPOSITIONS

			Delrin								Celcon [c]	
Property	Unit	ASTM No.	Temp.	100	150	500	550	507	900	AF	M25	M90
Tensile Strength	psi	D 638	-68°F	[a] 14,700						[b] 9,000		
			73°F	10,000						6,900		
			158°F	7,500						4,700		
Elongation	%	D 638	-68°F	38		13		11		6		
			73°F	75		15		13		12		
			158°F	460		330		280		38		
Impact Strength, Izod	ft lb/in. of notch	D 256	-40°F	1.8		1.2		1.0		.6	1.2	1.0
			73°F	2.3		1.4		1.3		.7	1.4	1.2

Note: a. Ref. 1
b. Ref. 2
c. Ref. 7
d.

COMPOSITIONS	CHARACTERISTICS
Delrin 500	General purpose molding material. Surface lubricated.
Delrin 550	Comparable to Delrin 500 but not surface lubricated.
Delrin 507	Comparable to Delrin 500 but contains a light stabilizer. Surface lubricated.
Delrin 100	Special purpose molding material: high viscosity hence suitable for easy-to-fill molds. Surface lubricated.
Delrin 150	High viscosity resin for extrusion. Comparable to Delrin 100 but not surface lubricated.
Delrin 900	Low viscosity hence better flow than Delrin 500; tensile strength, elongation and toughness are comparable to Delrin 500 except 900 is not as resistant to repeated impact. Surface lubricated.
Delrin AF	Oriented Teflon TFE fibers dispersed in matrix injection molding.
Celcon M25 and M90	Series of copolymers for extrusion and injection molding.

Impact strength of acetal composition changes little with decreasing temperature, but they are notch sensitives as are most thermoplastics. Izod unnotched impact strength is about 20 to 30 times the notched impact strength at room temperature.

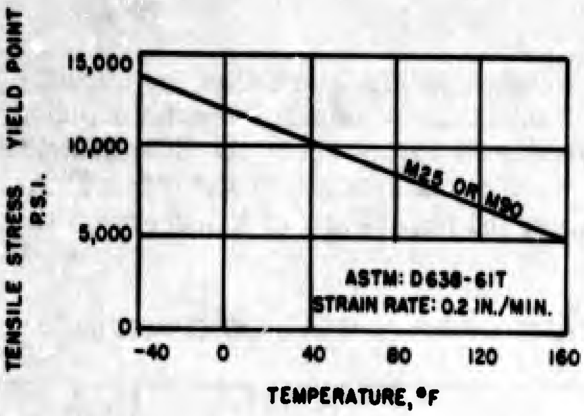


Figure 1. Effect of ambient temperature on tensile stress at yield point of M-grade Celcon(1)

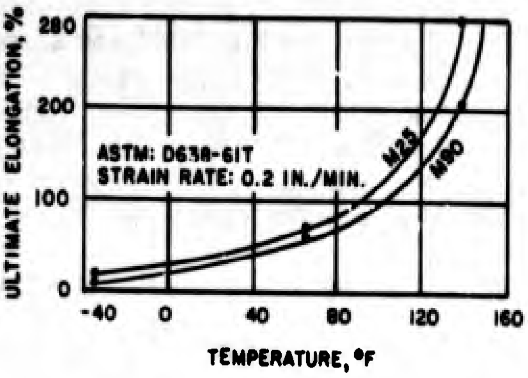


Figure 2. Effect of ambient temperature on ultimate elongation of M-grade Celcon(1)

Tensile tests were conducted on four experimental Celcon copolymers, SV-149, 150, 151 and 290 and two commercial Delrin polymers, 150 and 500.(3) Standard 8-1/2" tensile bars were injected molded on the 1 oz. Moslo machine and run at four crosshead speeds of .2 to 20 in./min. on the Instron Tester. For temperature other than 73°F, the tests were conducted in an environmental chamber. Test specimens were allowed to equilibrate in the chamber 30 minutes before testing. No extensometers were used because of the tendency for failure to occur prematurely at the knife-edge of the extensometer. Instead, an "effective" gage length for the ASTM bar was determined. Because the average effective gage length was determined to be within 1 percent to the jaw separation of 4.50 in., this number was used in all subsequent calculations of elongations. For tests at crosshead speeds greater than 0.2 in./min., the standard recorder used with the Instron was replaced with a Sanborn recorder whose pen response speed was faster. The results are shown in Table 2 and Figures 3 and 4.

Figure 3 plots the maximum stress versus strain rate at various temperatures for Celcon M90-01-SV-290. As would be expected, the stress increases with increasing strain rate and decreasing temperature. In fact, the data form almost parallel lines. It should be noted that the maximum and ultimate strength (see Table 2) do not vary too much over the range studied.

Figure 4 indicates that ultimate elongation at low temperatures is almost independent of strain rate. At higher temperatures the elongation varies markedly with both strain rate and temperature.

TABLE 2. TENSILE DATA OF INJECTED MOLDED ACETAL SPECIMENS (3)

Material[a]	Cross-head speed:	Maximum Stress, K psi, at -					Break Stress K psi, at -					Elongation at Break, %, at -				
		-30F	0F	30F	73F	140F	-30F	0F	30F	73F	140F	-30F	0F	30F	73F	140F
Celcon SV-149	0.2 in./min.	12.8	12.4	11.0	9.2	6.6	12.6	12.1	10.6	8.2	5.3	9	11	13	26	84
	2 in./min.	15.4	14.0	11.9	10.1	7.4	14.5	13.5	11.5	9.4	4.8	10	11	12	18	40
	20 in./min.	15.7	15.0	13.2	10.7	8.5	15.0	14.2	13.1	10.2	7.9	10	12	9	16	22
Celcon SV-150	0.2 in./min.	14.1	12.4	11.1	9.4	6.7	13.6	12.1	10.6	8.1	5.3	14	14	14	32	116
	2 in./min.	15.7	14.0	11.9	10.0	7.5	15.5	13.9	11.3	9.0	5.1	8	8	14	20	38
	20 in./min.	16.2	15.2	13.6	10.9	8.7	16.2	15.1	13.3	10.2	7.7	6	8	10	19	25
Celcon SV-151	0.2 in./min.	13.8	12.0	10.7	8.9	6.4	13.5	11.5	10.3	8.0	5.1	16	26	24	37	115
	2 in./min.	15.4	13.7	11.8	10.2	7.3	14.3	12.8	10.6	8.8	5.1	15	18	20	30	57
	20 in./min.	15.8	14.8	13.4	10.5	8.2	14.3	13.9	12.3	9.0	6.4	17	18	22	33	44
Delrin 150	0.2 in./min.	13.9	13.1	12.2	10.6	7.7	13.9	13.1	12.2	10.6	7.0	16	17	25	33	81
	2 in./min.	15.6	14.6	12.9	12.0	9.0	15.3	14.5	12.7	11.7	8.3	23	27	29	34	44
	20 in./min.	15.7	15.2	14.2	12.0	10.1	15.4	15.1	14.2	11.8	9.9	23	24	27	31	38
Delrin 550	0.2 in./min.	13.6	12.6	11.5	10.3	7.7	13.6	12.6	11.5	10.3	7.2	9	9	8	11	38
	2 in./min.	15.0	14.1	12.2	11.7	9.0	15.0	14.1	12.2	11.7	9.0	6	8	7	9	16
	20 in./min.	15.3	14.5	13.4	11.4	9.9	15.3	14.5	13.4	11.4	9.9	7	6	6	6	11
Celcon M90-01 SV-290	0.2 in./min.	14.7	-	-	9.4	-	13.4	-	-	8.1	-	11.9	-	-	25.9	-
	2 in./min.	15.1	-	-	10.0	-	13.6	-	-	8.2	-	14.2	-	-	22.8	-
	20 in./min.	16.5	-	-	10.7	-	14.6	-	-	9.6	-	14.3	-	-	22.3	-
Celcon M90-01 SV-290[b]	Temp.		-10F	-	-	130F	-	-10F	-	-	130F	-	-10F	-	-	130F
	0.2 in./min.	-	13.7	-	-	6.9	-	12.2	-	-	5.8	-	13.0	-	-	136
	0.5 in./min.	15.1	-	-	9.7	7.4	13.6	-	-	8.0	5.9	12.3	-	-	25.9	72.2
	2 in./min.	-	14.4	-	-	7.6	-	13.0	-	-	5.3	-	13.0	-	-	34.2
	20 in./min.	-	15.2	-	-	8.7	-	13.8	-	-	6.7	-	14.0	-	-	31.8

a. Average of three determinations

b. Average of ten determinations

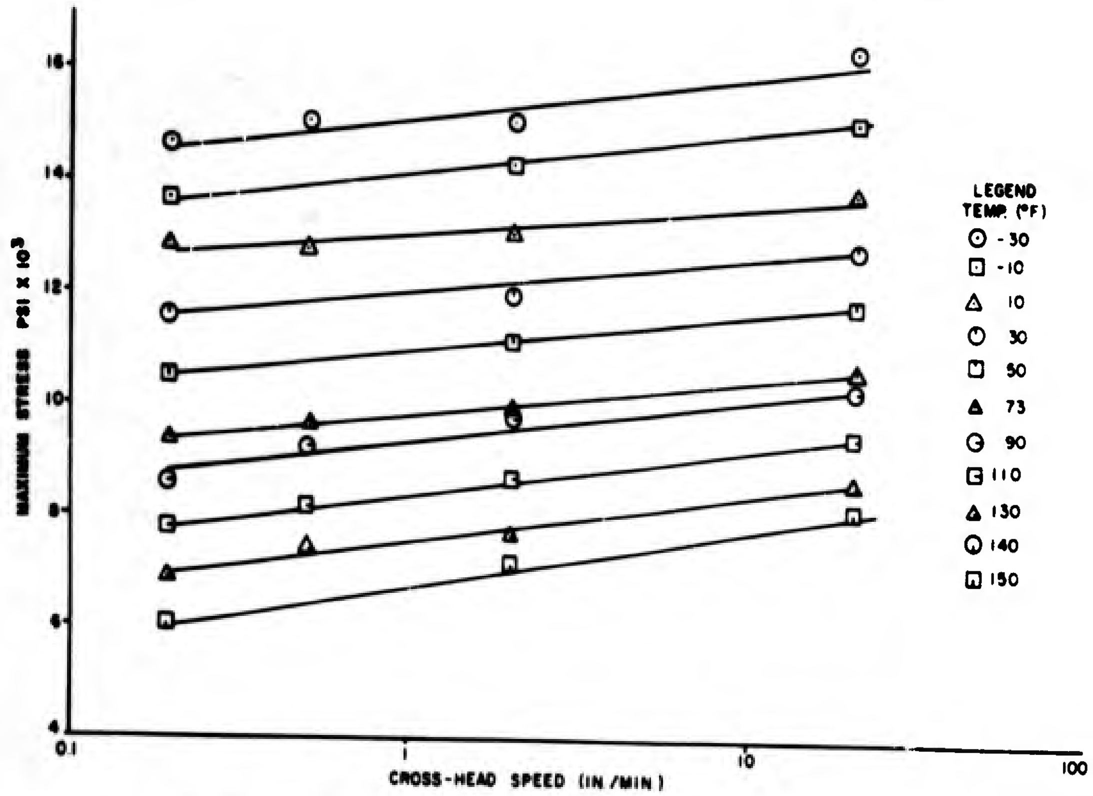


Figure 3. Maximum stress versus strain rate SV-290(3)

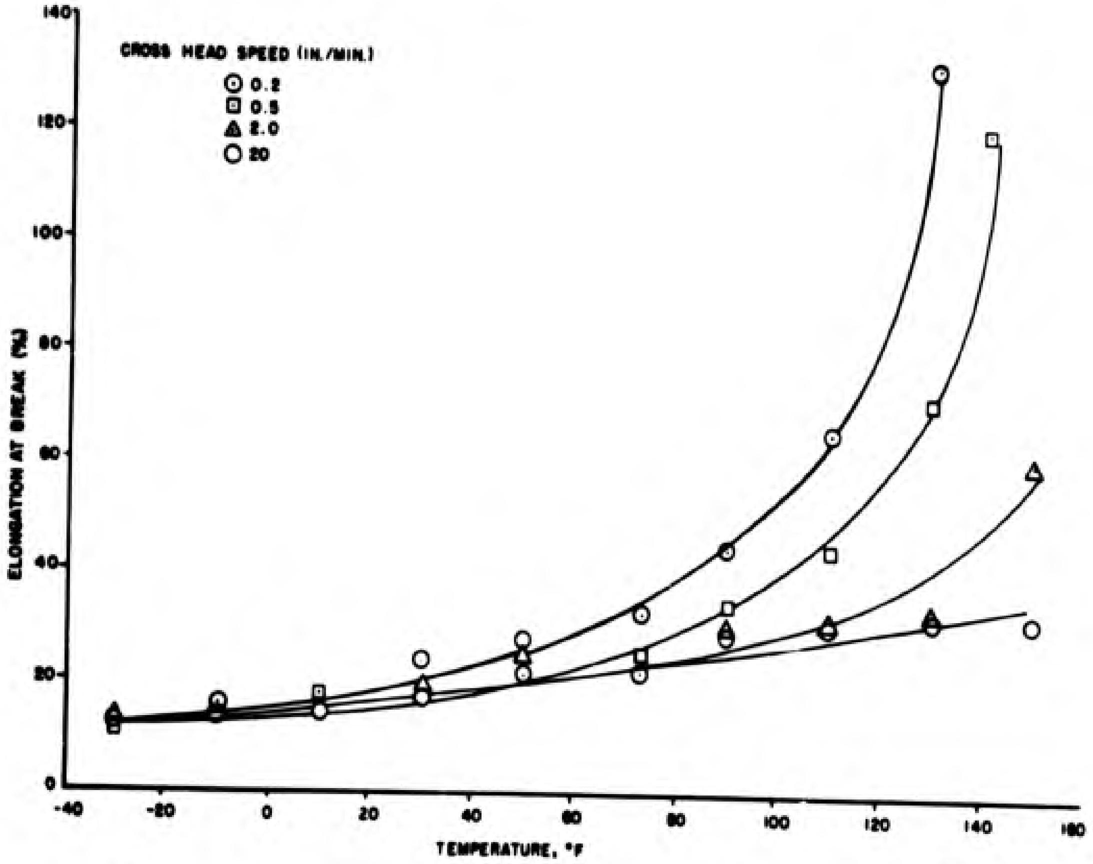


Figure 4. Break elongation versus temperature SV-290(3)

The modulus of rigidity of acetals was determined over the temperature range -60°C to +140°C by ASTM D1043.(4) Results are shown in Table 3 at five temperatures. It will be noted that all the materials have about the same behavior.

TABLE 3. MODULUS OF RIGIDITY OF ACETALS(4)

Torsional Rigidity, psi	A	B	C
-40 C	123,000	150,000	150,000
0 C	94,000	115,000	120,000
+40 C	72,000	91,000	85,000
+80 C	43,000	59,000	50,000
+120 C	23,000	30,000	27,000

A = General purpose, extrusion grade
B = General purpose, injection molded grade
C = Weather-resistant, injection molded grade

The curve for general purpose, extrusion grade polyacetal is shown in Figure 5. Sharp transitions were not evident; however, there is an indication that transitions occur at about -50°C to +30°C but these changes are not drastic.

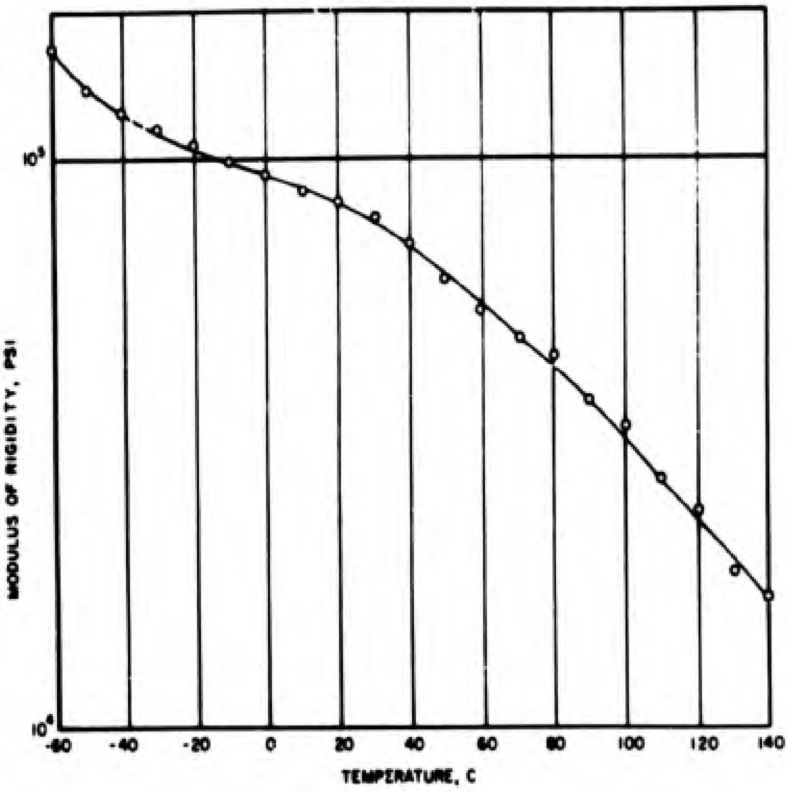


Figure 5. Effect of temperature on stiffness of polyacetal, general purpose, extrusion grade (compression molded; ASTM D 1043)(4)

Figure 6 illustrates the effect of moisture and temperature on the flexural modulus. As can be seen, moisture has a slight effect, reducing the property by about 20 percent at minus 40°F under wet conditions. Decreasing the temperature from 80°F to -40°F increases the modulus by about 20 percent at 50 percent RH.

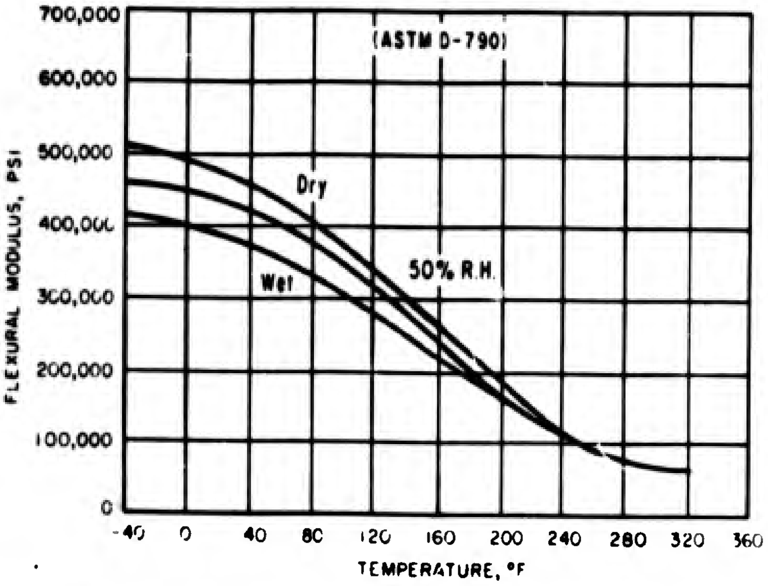


Figure 6. Flexural modulus of acetal resin versus temperature(5)

The degree of crystallinity of the material in the temperature range under study can influence mechanical properties such as elongation, yield point and modulus. The molecular structure of acetals permits a high degree of crystallinity. This crystallinity can be altered by annealing or by varying the quench temperature. Therefore, the stiffness as determined by tensile and flexural modulus can be affected to a limited extent (10 to 20 percent) by the procedure used in fabrication. At higher mold temperatures it becomes stiffer and modulus increases with decrease in elongation. This is most pronounced in thin sections. (5)

Electrical Properties

In most applications the mechanical properties of acetals will be more important than the electrical properties. Nevertheless, there may be some specific areas where electrical properties are critical.

Table 4 gives the dissipation factor and dielectric constant of Delrin 500, a general-purpose molding compound.

TABLE 4. ELECTRICAL PROPERTIES OF DELRIN 500, NC-10(1)

Temp °F	Dissipation Factor 1/ 10^3 cps	Dissipation Factor 1/ 10^5 cps	Dielectric Constant 1/ 10^3 cps	Dielectric Constant 1/ 10^5 cps
-94	0.0160	0.0130	3.00	2.84
-76	0.0225	0.0200	3.15	2.91
-58	0.0260	0.0300	3.30	3.00
-40	0.0150	0.0360	3.36	3.14
-22	0.0030	0.0300	3.38	3.28
-4.0	0.0011	0.0170	3.40	3.36
14	0.0007	0.0080	3 [illegible]	3.39
32	0.0007	0.0036	3 [illegible]	4.41
50	0.0007	0.0018	3.41	4.43
68	0.0007	0.0012	3.43	4.43
77	0.0007	0.0010	3.44	4.43
104	0.0009	0.0018	3.52	3.50
140	0.0035	0.0029	3.58	3.55
176	0.0038	0.0060	3.68	3.62

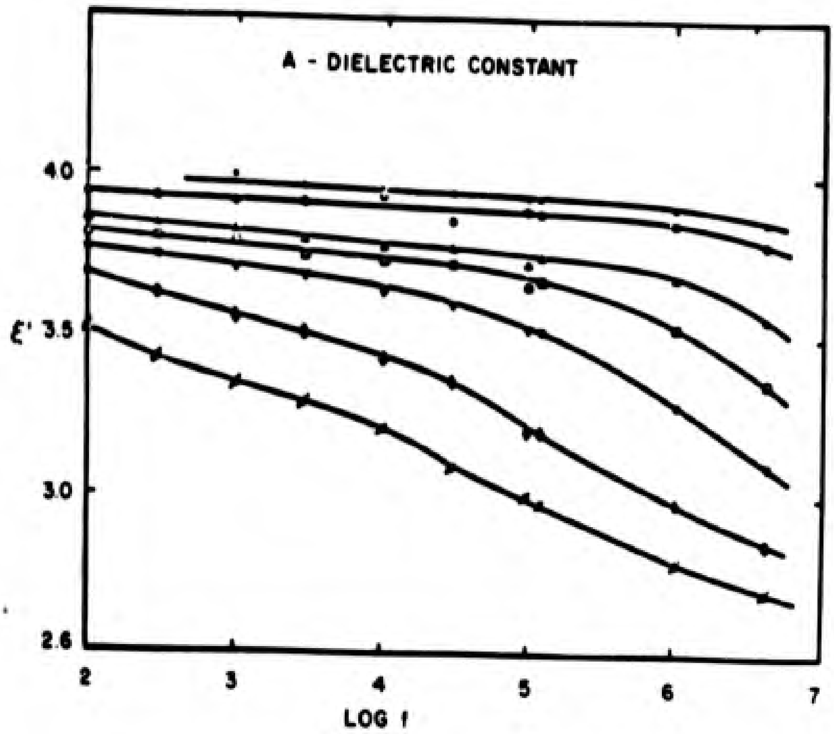
1/ ASTM D150-54T
Bone Dry (run in dry nitrogen)

Figure 7 shows the dielectric constant (ϵ') and loss factor (ϵ'') as a function of frequency for given temperatures of compression molded acetal. (6) Measurements were made on a WTW modified Schering bridge (10^2 - 10^5 c/sec.) and a Hartshorn-Word resonance circuit (10^5 - 10^6 c/sec.).

A dielectric dispersion and absorption region is seen where the height of the absorption decreases with decreasing temperature. Also, the loss curve broadens and becomes more complex at lower temperatures. (6)

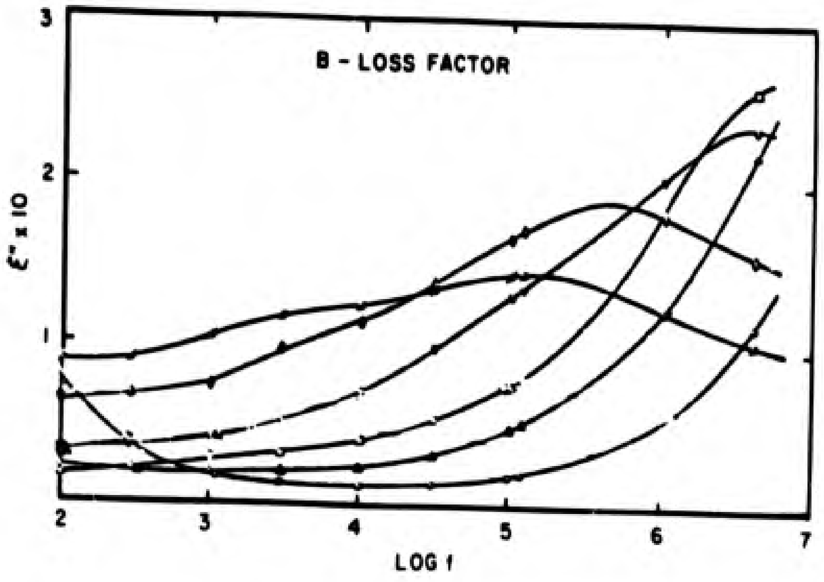
The dielectric constant and loss tangent were measured on a Schering bridge in the frequency range 30 cps to 30,000 cps and temperature of -112 to 250°F on Celcon samples in air. (7) The data appear in Figures 8 and 9. Figure 9 indicates that there are two main regions of high loss: a low frequency, high temperature region due to a condition process and a loss hump at low temperature attributed to a di-polar relaxation mechanism.

This data indicates that there is a useful temperature range of -40 to 120°F where losses are relatively low and dielectric constant varies little with temperature and frequency.



∅-67.2°C, ◊-58.6°C, ▽-47.9°C, ◻-37.1°C,
▲-27.2°C, ○-9.8°C, ●+20.9°C

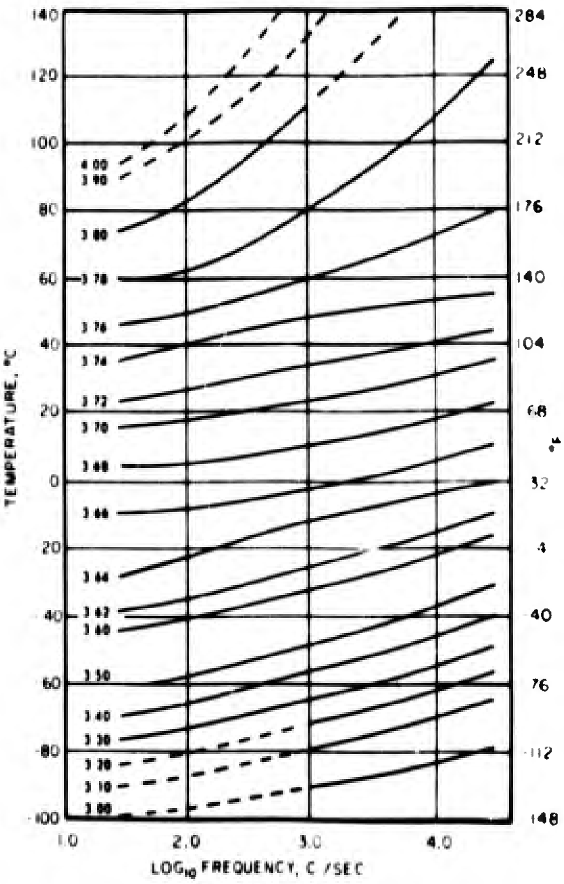
(a)



∅-67.2°C, ◊-58.6°C, ▽-47.9°C, ◻-37.1°C,
▲-27.2°C, ⊙-9.8°C

(b)

Figure 7. The dielectric constant (ε') and loss factor (ε'') as a function of frequency for given temperatures (6)



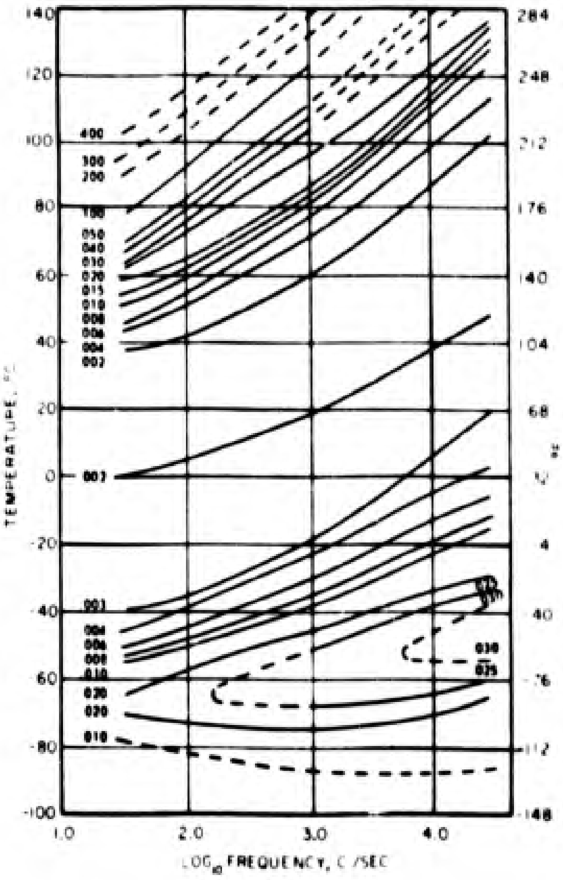


Figure 8. Variation of acetal dielectric constant with temperature and frequency(7)

Figure 9. Variation of acetal loss tangent with temperature and frequency(7)

Thermal Properties

One method for indicating low-temperature is by means of the brittleness temperature as outlined in ASTM D 746. In this test, unnotched molded specimens are subjected to an impact blow. That temperature at which 50 percent of the specimens fail from one impact blow is the brittleness temperature.

Specimens from compression molded disks from four acetal molding grades were cooled to -55°C and subjected to impact load; all specimens failed.(4) Tests were then conducted at 0°C; 100 percent of two molding grades failed. About 17 percent of one sample and 8 percent of a general purpose composition failed at 0°C. The brittleness temperature was not established but it did point out that acetals have poor resistance to shock loads at low temperature.

Little information on thermal properties could be obtained. However, the average coefficient of linear thermal expansion (ASTM D 696-44) for Celcon acetal copolymers M90 and M25 is 4.7 x 10^{-5} in./in./°F over the temperature range of -30°C to +30°C.(7) These were as-molded, unannealed specimens.

The following data shows the environmental shrinkage of acetal moldings due to stress relaxation.(1)

Temperature	Molding	Shrinkage mils/inch	Shrinkage cm/cm
-40 to 170°F	Molded with mold temperature of 250°F (121°C)	< 0.5	< 0.0005
	Routine molding - cold mold	≤ 1	≤ 0.001
	Annealed Samples - 320°F (160°C) for 15 minutes	< 0.5	< 0.0005

SECTION II. ACRYLICS

Acrylics are known for their outstanding optical properties. Because of this, most effort has been directed towards applications utilizing this attribute. Hence, there is a great deal of information in the literature on optical properties, weathering, aging, etc. Little data is available on low temperature effects on mechanical, electrical and thermal properties, especially on molded materials.

Acrylonitrile styrene and methyl methacrylate styrene are normally considered more in the styrene than in the acrylic family, and the properties are discussed in the section on styrene. Acrylonitrile-butadiene-styrene is covered separately.

Mechanical Properties

Table 5 presents data on the tensile properties of five molded acrylic resins.(8) The modulus of elasticity of three of the resins is shown in Figure 10.

TABLE 5. PROPERTIES OF MOLDED ACRYLIC RESIN (8)

Property(2)	Acrylic (Lucite)(1) (2)				
	#30 and 130	#29 and 129	#40 and 140	#147	#148
Tensile strength, K psi(3)					
-70 F	14.5	14.5	14.5	15.0	-
73 F	9.5	10.0	10.5	10.5	11.0
158 F	3.5	4.0	5.0	6.0	6.7
Tensile elongation, %					
-70 F	2	2	2	2	-
73 F	3-5	3-5	3-5	4-7	5
158 F	100	90	80	29	30

(1) Lucite 140 - heat-resistant composition for injection molding, extrusion
129 - general purpose composition
130 - general purpose composition
147 - high molecular wt. polymer for extrusion
148 - extrusion for matte surface
29, 30 and 40 - molding and extrusion compositions

(2) ASTM D 638-52T. All specimens were annealed under following conditions

Annealing Temp		Annealing Time	
30 and 130	140 F	1/8" thick	2 hours
29 and 129	160 F	1/4" thick	4 hours
40 and 140	180 F		
147 and 148	180 F		

(3) Thickness, 1/8 in.

The tensile strength of acrylics, which is among the highest of the thermoplastics, increases with decreasing temperature. Its moderately low modulus increases and elongation decreases with decreasing temperature.

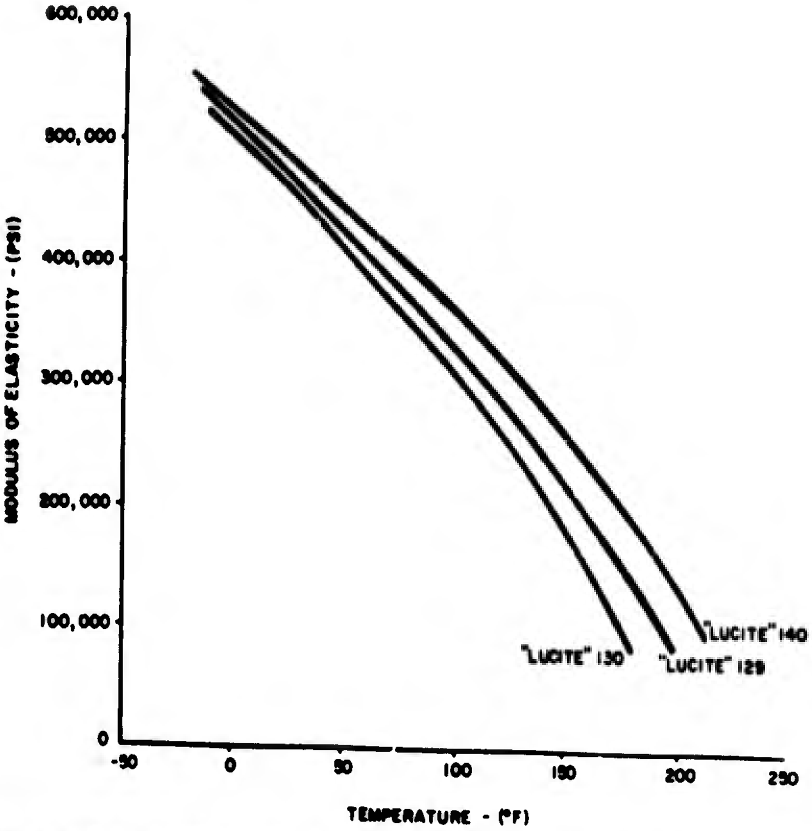


Figure 10. Modulus of elasticity of acrylic (Lucite) versus temperature (ASTM D 638-52T) (8)

Mechanical tests were conducted on 100 percent general purpose molded polymethylmethacrylate and a heat resistant cast sheets; results are contained in Table 6.(9) Table 7 gives the tensile strength and modulus of two acrylic cast sheets. (10)

TABLE 6. PROPERTIES OF POLYMETHYL METHACRYLATE(9)

Property(1)	GP Molding Material				Heat Resistant Cast Sheet			
	-65 F	-40 F	+10 F	+77 F	-65 F	-40 F	+10 F	-77 F
Tensile, K psi	16.0	15.8	14.2	10.1	15.0	15.0	13.9	11.2
Elongation, %	2.5	2.8	3.6	5.9	2.1	2.5	3.3	5.8
Modulus of Elasticity, K psi	800	660	557	420	848	726	597	447
Work to Produce Failure (Ft/lb/in.^{3})	18.4	21.2	25.8	36.8	14.5	17.7	24.3	39.2
Proportional Limit K psi	7.2	6.8	8.5	6.1	7.0	4.4	4.0	4.1
Izod Impact Strength (Ft/lb/in. of notch)	.45	.45	.45	.44	.40	.49	.50	.45

(1)Median Values

TABLE 7. TENSILE PROPERTIES OF ACRYLIC (PLEXIGLAS) CAST SHEETS(10)

Property(1)	Value(2) at Degrees F						
	Type	-76	-40	32	77	140	160
Tensile strength, K psi, max.							
	1-A	12.5	-	-	8.0	4.0	2.4
	11 UVA	14.0	-	-	9.5	5.7	4.4
Modulus of elasticity in tension, x 10^5	1-A	8	7	5	4	2	-
	11 UVA	8	7	5	4	2	-

(1) ASTM D 638-46T (0.2"/minute)

(2) Values not to be used in calculating allowable stresses in Aircraft parts.

Figure 11 shows the variation in flexural strength with decreasing temperatures for a cast Lucite sheet. Flexural strength of the Lucite sheet is not greatly affected by moisture. After 96 hours of water soaking the flexural strength did not change appreciably. At temperatures of -40°F and -112°F, samples were reported to show slight increase in strength.(11)

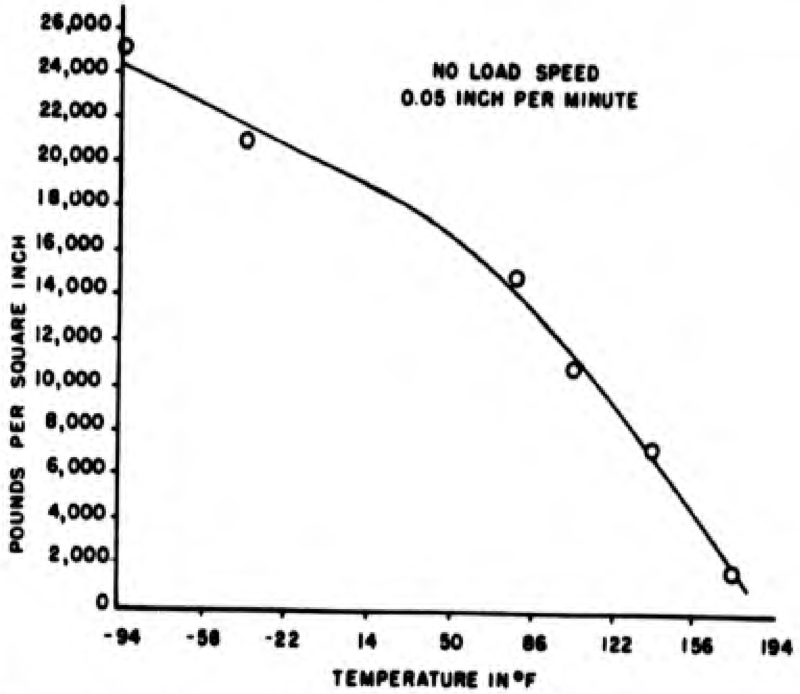


Figure 11. Flexural strength variation with temperature of cast acrylic sheet(11)

An indication of the toughness of an acrylic-PVC alloy is shown in Table 8. (12) Useful ductility is retained down to -22°F and impact data exceeds ASTM specification for type II PVC.

TABLE 8. PROPERTIES OF ACRYLIC-PVC ALLOY (KYDEX) AT VARYING TEMPERATURES(12)

Property	Value							
	Temp:	-58 F	-22 F	14 F	50 F	73 F	122 F	158 F
Elongation, %		16.3	44.9	>60(1)	>80(1)	167.2	>80(1)	>80(1)
	Temp:	-65 F	-40 F	-20 F	0 F	32 F	73 F	
Impact strength, charpy,(2) unnotched, ft/lb.		14	23	26	35	80	118	
	Temp:	-65 F	-40 F	-20 F	0 F	25 F	48 F	73 F
Impact strength, falling dart,(3) ft/lb		3.2	4.0	4.4	12.0	>30(4)	>36(4)	>36(4)

(1) Not carried to rupture due to limits of test equipment.

(2) ASTM D 256-56, 1/2" x 1" section, 2" span.

(3) R & HP -24 F, 2 lb dart with 1/8" radius.

(4) Dent - no break.

Figure 12 plots the flexural modulus of the acrylic-PVC alloy over a range of temperature. (12) This modulus is among the highest offered by the unreinforced thermoplastic materials and it varies very little in the temperature range of interest. Thus, this alloy successfully combines toughness with rigidity.

The effect of low temperature on impact strength of Acrylic multipolymers is shown by the following. (13)

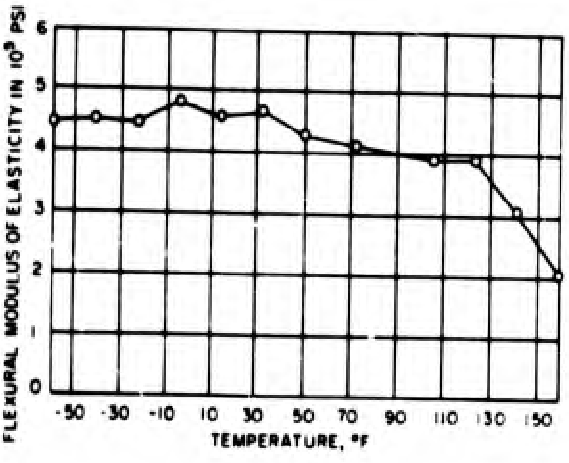


Figure 12. Flexural modulus of elasticity versus temperature of acrylic-PVC alloy (Kydex) (12)

Property	XT-500	XT-250	XT-150
Izod Impact Strength, D 256-56 Notched ft. lbs/inch of notch (1/4 inch bar)			
73°F	3.0	1.0	0.7
32°F	2.3	0.9	0.7
-40°F	1.6	0.9	0.7

As can be seen, the impact strength of the alloy is superior to the unmodified acrylics.

The effect of low temperature on acrylic transparency is shown in Figure 13. It is interesting to note the rather significant decrease in percent light transmission with decreasing temperature. The author is offered no explanation for this phenomena.

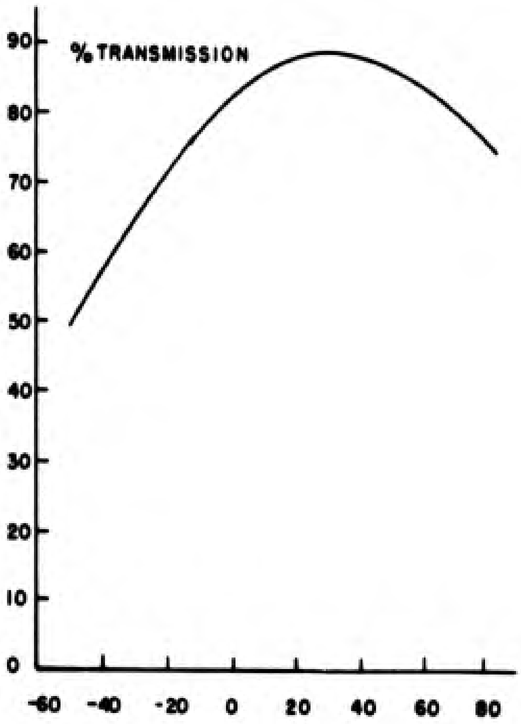
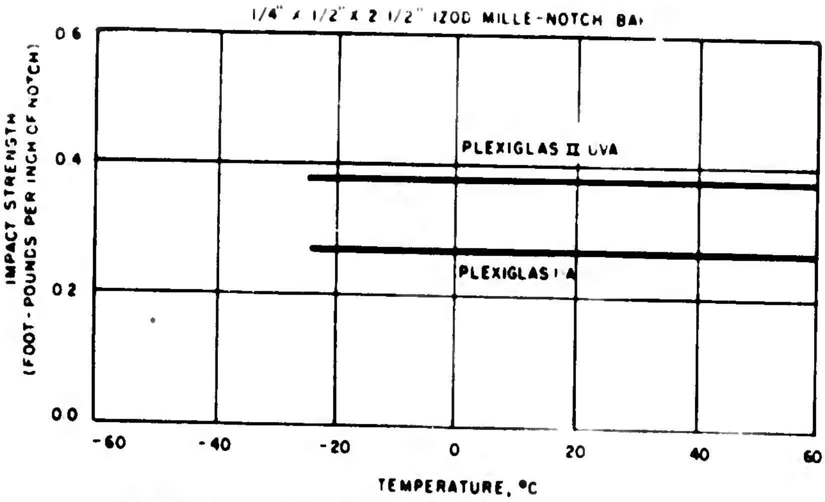


Figure 13. Percent light transmission versus temperature for XT-500(13)

The impact strength of unmodified acrylates (as opposed to the alloys and copolymers, etc.) are low and change very little with decreasing temperature.(10) (See Table 6 and Figures 14, 15 and 16.) However, they are highly notch sensitive. This is demonstrated in Figure 15.



TEST METHODS -ASTM D256-47, PROCEDURE A, ASTM D758-48
PRE-CONDITIONING - ASTM D618-47T, PROCEDURE A
CONDITIONING MORE THAN ONE, BUT LESS THAN FIVE HOURS IN TEST ATMOSPHERE PRIOR TO TESTING

Figure 14. Effect of temperature on the impact strength of Plexiglas I-A and II UVA(10)

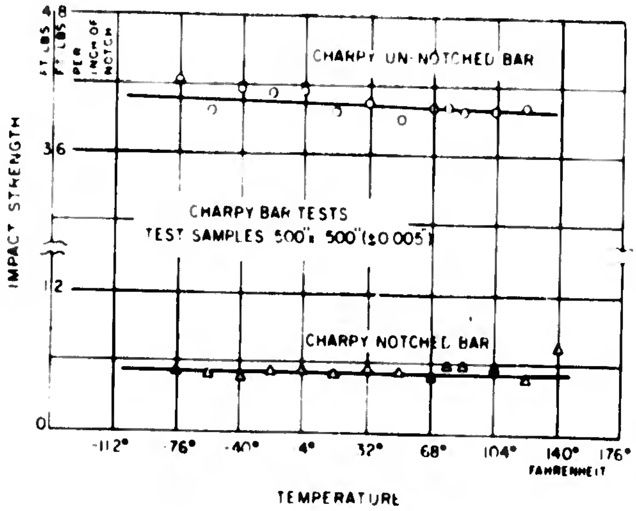


Figure 15. Impact strength versus temperature of cast acrylic sheet (Plexiglas) (10)

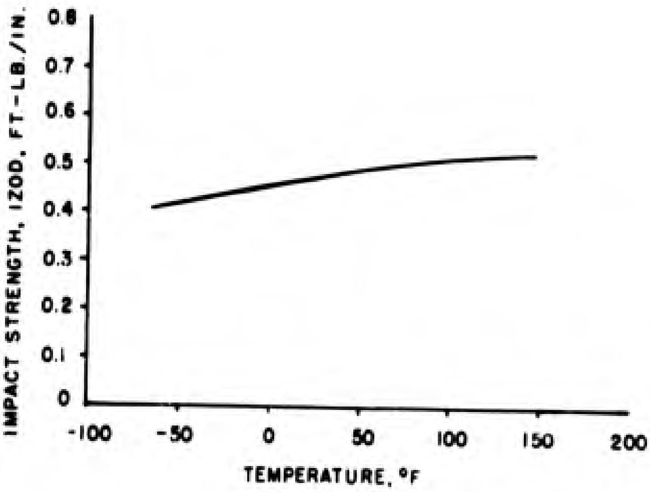
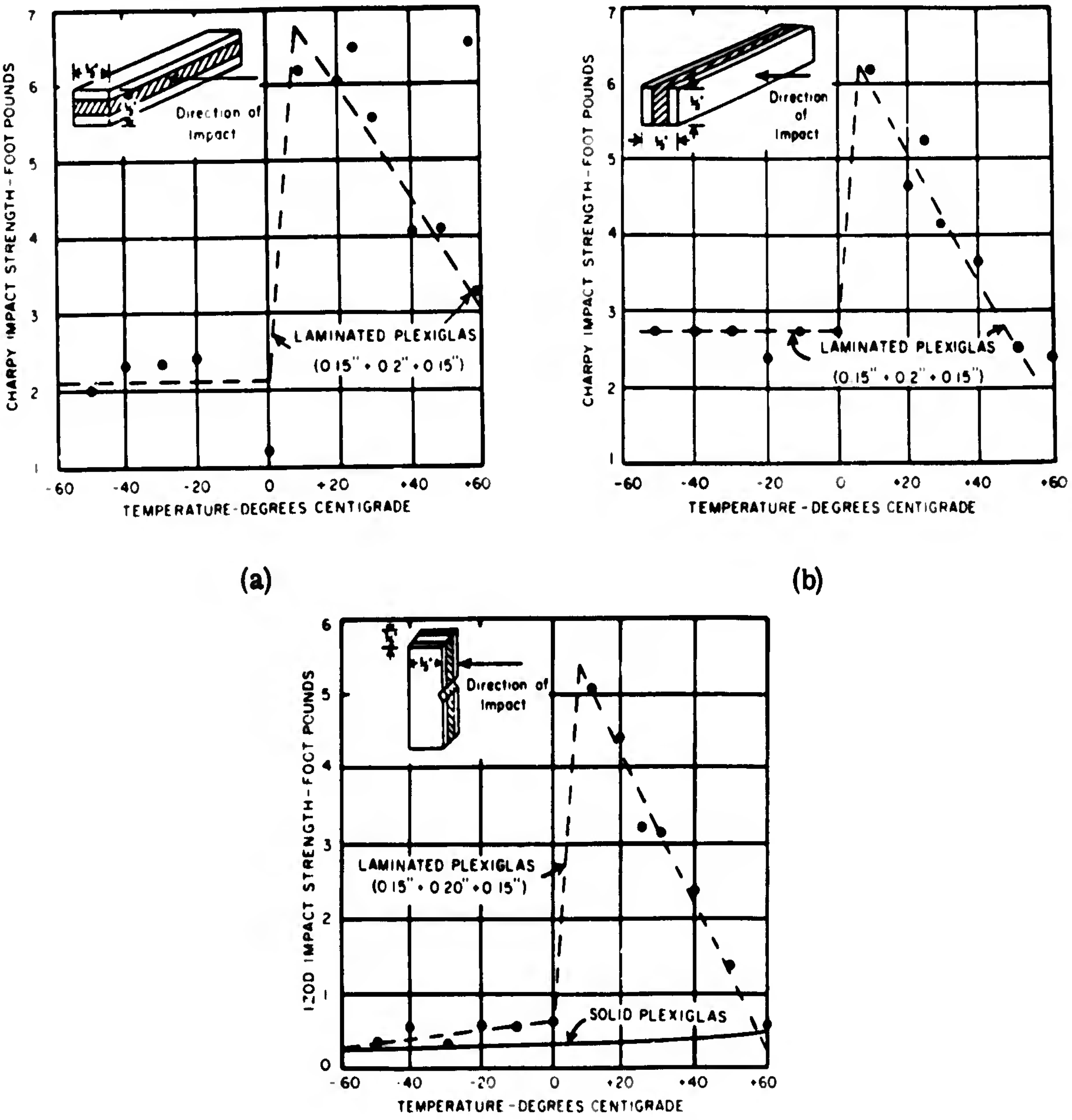


Figure 16. Effect of temperature on impact strength of acrylic (Lucite) HC-201), (general purpose cast sheet) (14)

Figure 17 shows the effects of temperature on the impact strength of an acrylic laminate (Plexiglas). Unlike the solid sheets, the impact strength of the laminate changes radically at about 32°F. This is attributed to a transition occurring in the polyvinyl butyral interlayer. Also, the laminated acrylic is less notch sensitive than the solid acrylic. This again is the result of the interlayer material.

The effect of direction on impact strength is well illustrated in Figure 17a and 17b.



(a)

(b)

(c)

Figure 17. Impact strength of laminated acrylic (Plexiglas)(10)

Electrical Properties

Dielectric measurements at 60 cycles per second on .2 g samples of acrylic polymers over a temperature range of -40° to +200°C are shown in Figure 18. (15) A General Radio Co. (Type 740 BG or Type 1611-B) 60 cycle bridge and an electrically heated oven which can be controlled at temperatures from 25°C up to 300°C were used. Temperatures down to -40°C were obtained in another chamber using a dry-ice cooled, water-ethylene glycol mixture for control. Also a plastic enclosure and P_2O_5 for avoiding difficulties from condensation at low temperatures were used. The tests and calculations were made following the ASTM method D 150-59T.

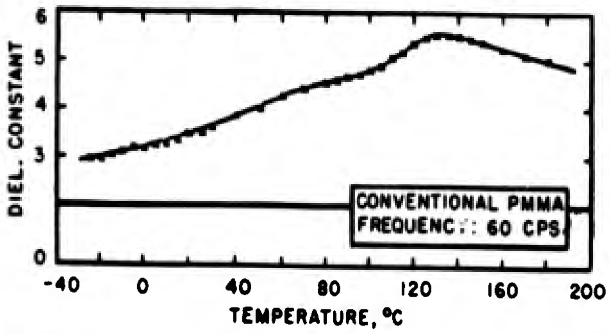


Figure 18. Dielectric constant of polymethyl methacrylate versus temperature

Figures 19, 20, and 21 compare the dissipation factors at 60 c/s of various acrylic compositions.(15) These figures illustrate very well the susceptibility of dielectric properties to changes in formulations and temperature. The various absorption peaks point out the need for testing material over the planned temperature range in applications where dielectric properties are critical.

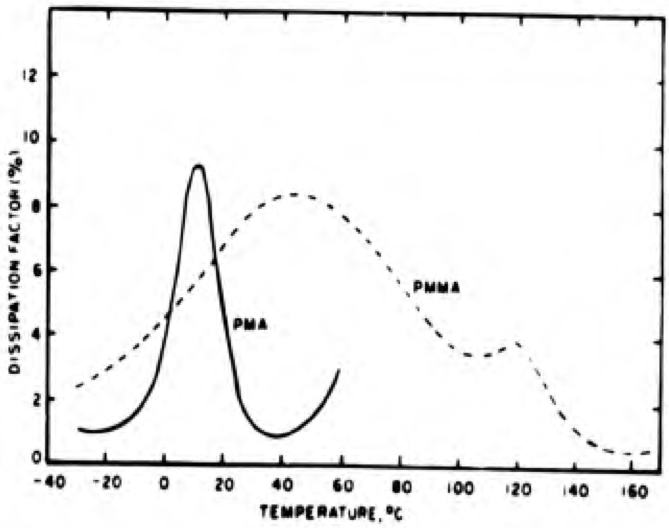


Figure 19. Comparison of polymethyl methacrylate and polymethyl acrylate at 60 c/s(15)

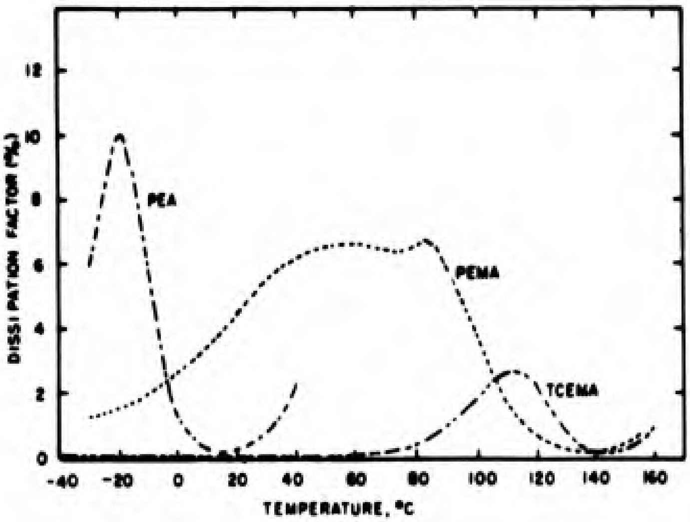


Figure 20. Comparison of polyethyl methacrylate polyethyl acrylate and trichloro ethyl methacrylate at 60 c/s(15)

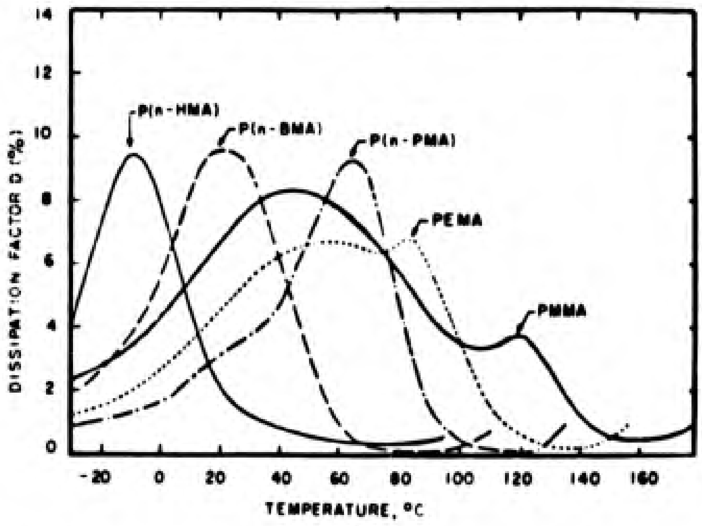


Figure 21. Comparison of methacrylate homologues showing the merging of α - and β -peaks (15)

Note: P(n-HMA) - Hexenyl methacrylate
P(n-BMA) - Butyl methacrylate
P(n-PMA) - Propyl methacrylate
PEMA - Polyethyl methacrylate
PMMA - Polymethyl methacrylate

Figures 22 and 23 show the variation of acrylic dielectric properties with temperature and frequency.(16) No radical changes, i.e. peaks, are evident in the low temperature range.

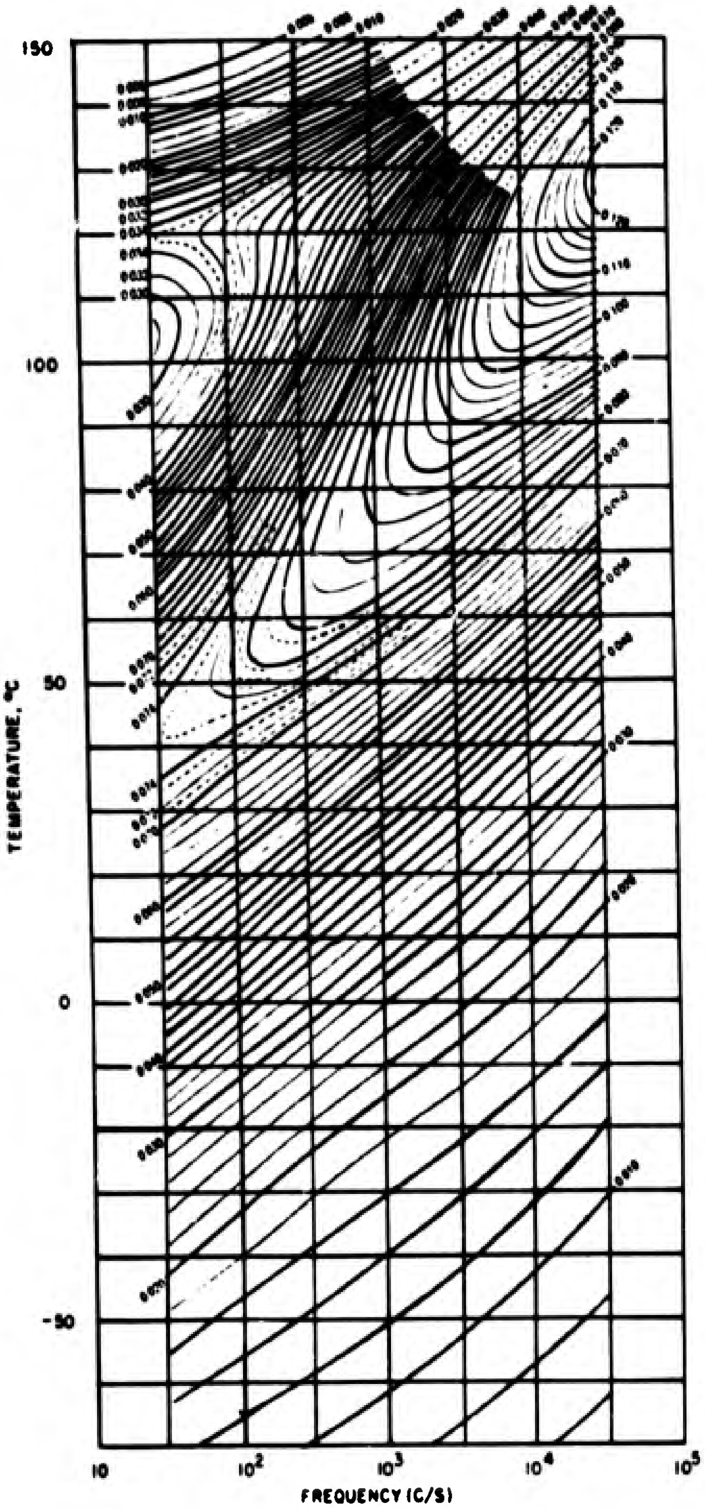


Figure 22. Variation of acrylic power factor with temperature and frequency. (16)

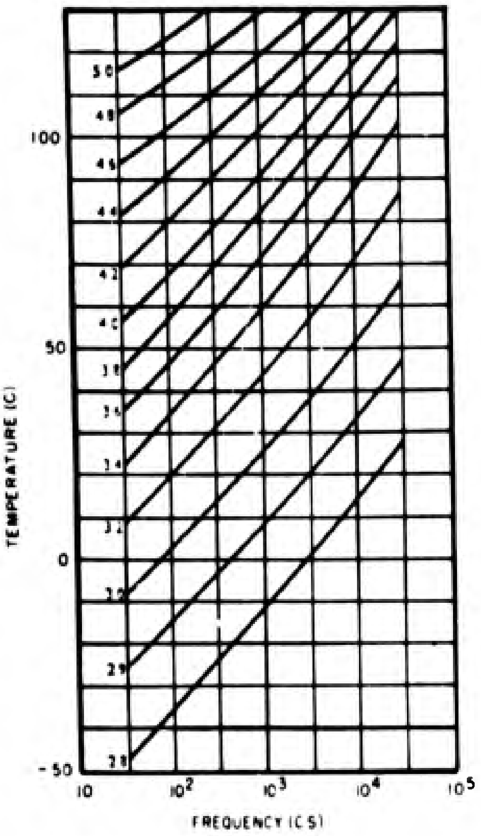
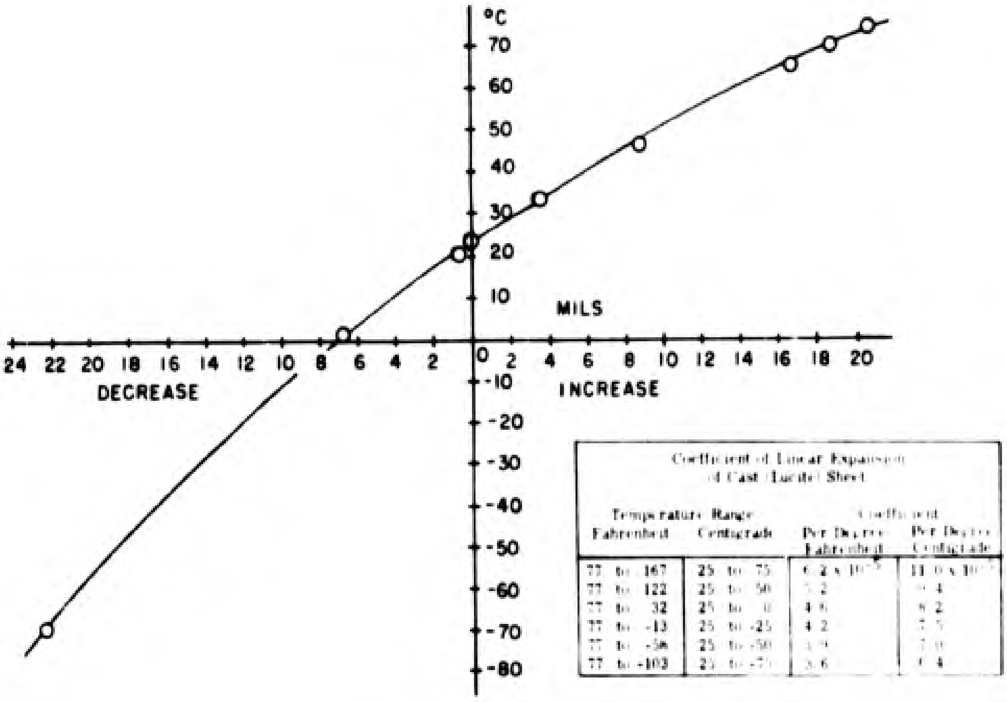


Figure 23. Variation of acrylic dielectric constant with temperature and frequency. (16)

Thermal Properties

The coefficient of linear thermal expansion of cast acrylic sheets at different temperatures is given in Figure 24 and Table 9. Cast Lucite is stated to have a coefficient of linear thermal expansion about three times that of aluminum, eight times that of steel and ten times that of glass.(11)



Coefficient of Linear Expansion of Cast (Lucite) Sheet

Temperature Range Fahrenheit	Temperature Range Centigrade	Coefficient Per Degree Fahrenheit	Coefficient Per Degree Centigrade
77 to 167	25 to 75	6.2×10^{-5}	11.0×10^{-5}
77 to 122	25 to 50	5.2	9.4
77 to 32	25 to 0	4.6	8.2
77 to -13	25 to -25	4.2	7.5
77 to -58	25 to -50	3.9	7.0
77 to -103	25 to -75	3.6	6.4

Figure 24. Increase and decrease in length of cast acrylic (Lucite) bar (measured with quartz dilatometer - initial length 3.74") (11)

TABLE 9. THERMAL EXPANSION OF PLEXIGLAS CAST SHEET

Coefficient of Thermal Expansion(1) (in./in./°F) x 10^{-5}	Plexiglas Type						
	G & II UVA	IA UVA	55	V	V (920)	VM	5009 White TI
Thickness, In.	1/4	1/4	1/4	-	-	-	1/8
At: -40 F	2.8	3.1	2.7	2.7	2.7	2.8	3.7
-20 F	2.9	3.2	2.8	2.9	2.9	3.0	-
0 F	3.1	3.4	3.0	3.1	3.1	3.2	4.2
20 F	3.3	3.6	3.2	3.2	3.2	3.3	-
40 F	3.6	3.9	3.5	3.4	3.4	3.5	4.9
60 F	3.9	4.2	3.8	3.6	3.6	3.7	-
80 F	4.2	4.6	4.2	3.9	3.9	4.0	5.5
100 F	4.6	5.0	4.7	4.2	4.3	4.5	-

(1) R & H P4A

SECTION III. ACRYLONITRILE-BUTADIENE-STYRENE

Mechanical Properties

Tables 10 and 11 give some typical values for mechanical properties of ABS molding and extrusion materials.(5) These tables also illustrate the differences in properties between the general purpose, heat resistant, medium and high impact grades. Some of these are also shown in figures 25 and 26.

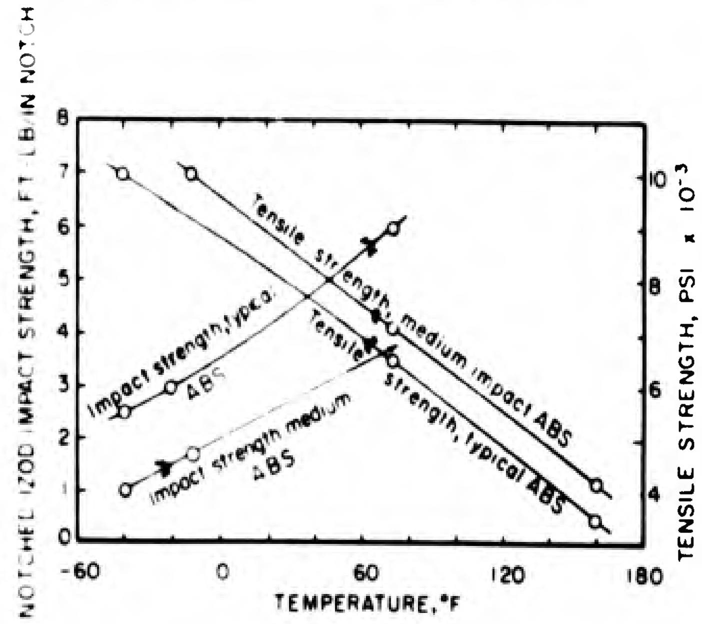


Figure 25. ABS tensile and impact properties versus temperature(5)

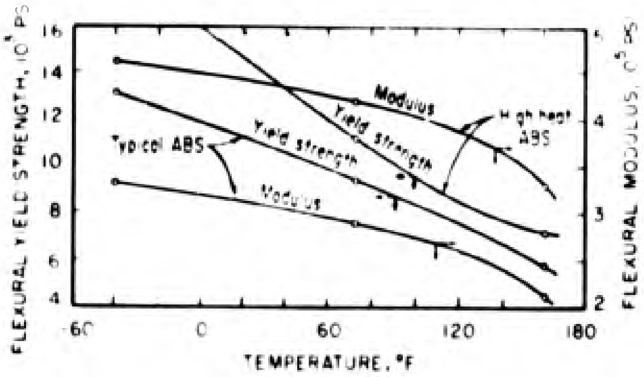


Figure 26. Flexural properties of ABS versus temperature(5)

As would be expected, strength and rigidity are reduced with the higher impact strength grades. ABS's, in general, have very good impact strength down to -40°F. However, they are also notch sensitive. Failure is ductile rather than brittle, indicating toughness is not drastically affected by orientation of internal stresses.

TABLE 10. MECHANICAL PROPERTIES OF ABS POLYMERS (CYCOLAC)(5)

Property	F	ASTM Test	Types(3)														
			GS	T	X-27	X-17	X-7	H	L	CG	E	A	D	AH	DH	LL	EP 3510
Izod Impact Strength, 1/8" bar,(2) unnotched, ft-lb in. notched, ft-lb, in. 1/4" bar, notched, ft-lb in. " " "	73 73 -20 -40	D256 Method A	29.3 6.2 2.6 2.1	26.0 5.3 2.1 1.8	33.0 3.1 1.3 1.0	- 2.5 - 0.7	32.0 4.2 1.4 1.2	No(4) 6.3 2.8 2.6	No 6.7 4.3 2.8	No 8.0 - 2.0	No 8.5 - 3.0	23.0 4.0 1.4 1.0	16.9 3.5 1.2 0.8	18.3 4.0 1.4 1.0	15.0 3.5 1.2 0.8	No 8.5 4.5 2.5	26.0 4.5 1.4 1.0
Charpy Impact Strength, 1/4" bar, unnotched, ft-lb/in. 1/4" bar,(1) notched, ft-lb/in.	73 73 -20 -40	D256 Method B	35.0 4.7 2.6 2.2	26.0 3.7 2.1 1.7	19.3 1.9 1.1 0.9	- - - -	33.0 3.1 1.7 1.4	No 5.3 2.9 2.6	No 6.0 3.7 3.1	- - - -	- - - -	19.0 2.6 1.5 1.2	15.6 1.6 1.3 1.0	- - - -	- - - -	No 7.5 4.0 3.2	26.0 3.7 1.6 1.5
Tensile Strength, Type 1, 1/8" thickness,(2) K psi	160 73 -40	D-638 0.2" min.	3.4 5.9 9.2	3.2 6.0 9.5	4.6 7.3 11.5	- 7.2 -	4.0 6.9 10.4	2.4 4.7 7.2	2.6 5.0 7.4	- 5.0 6.5	- 4.8 -	3.5 6.3 10.0	3.9 6.9 11.0	- 6.8 -	- 7.8 -	2.9 5.1 7.8	3.2 6.0 9.5
Tensile Modulus, Type 1, 1/8" thickness,(2) K psi	73	D-638 0.2" min.	310	300	390	380	330	230	230	260	230	330	350	350	380	240	310
Flexural Strength, 1/8" x 1" x 4" bar, K psi	160 73 -40	D-790 0.05" min.	No(4) No No	No No No	No No No.	No No No	No No No	No No No	No No No	No No No	No No No	No No No	No No No	No No No	No No No	No No No	No No No
Flexural Yield Strength, 1/8" x 1" x 4" bar,(1) K psi	160 73 -40	D-790 0.05" min.	5.7 9.7 15.2	5.5 9.6 15.1	7.5 11.8 18.8	6.8 10.6 18.1	6.2 10.2 16.1	4.1 7.6 11.8	4.3 7.8 12.1	3.5 7.2 10.5	- 7.3 -	5.4 9.9 17.3	5.6 10.2 17.1	6.8 11.0 17.5	7.7 11.8 18.1	5.1 7.7 11.0	5.5 9.2 15.1
Flexural Modulus, 1/8" x 1" x 4" bar,(1) Kpsi	160 73 -40	D-790 0.05" min.	220 320 360	210 320 360	310 400 460	270 380 420	260 360 390	160 240 280	160 250 180	140 260 300	- 230 -	240 360 420	260 390 430	250 370 410	300 400 440	190 240 260	210 330 360

(1) Compression molded.
(2) Injection molded.
(3) The types of ABS polymers are described as

GS	- Maximum toughness with high modulus (Inj, blow, extru)
T -	- Toughness with high flow (Inj)
X-27	- Maximum high heat with high modulus (Inj, extru)
X-17	- Maximum high heat with good moldability (inj, extru)
X-7	- High heat with toughness (Inj, extru)
H	- Good toughness (Inj, extru)
L	- Good toughness at low temp (extru)
CG	- Toughness with low gloss (extru)
E	- Very high impact at low temp (extru)
A	- Medium impact (Inj, extru)
D	- Medium impact (Inj, extru)
AH	- Medium impact, high heat (Inj)
DH	- Medium impact, high heat (Inj)
LL	- Black only, for pipe (extru)
EP-3510	- Grey only, for chrome plated parts (Inj, extru)

(4) No - No break, No failure

TABLE 11. MECHANICAL PROPERTIES OF ABS POLYMERS (KRALASTIC) (5)

Property	F	ASTM Test	Pipe			Moldings, Profiles			Sheet			
			W	400	450	K-2938	MH	MV	SR	SRA	SRB	K-2540
Izod Impact Strength, 1/8" bar,[1] notched, ft-lb/in.	73	D256 (A)	8.5	3.5	10.0	3.5	4.5	6.5	7.5	4.0	2.0	8.0
	32		7.5	2.5	-	2.5	3.5	5.5	6.0	2.5	1.5	7.0
	-20		4.5	1.5	-	1.5	2.0	4.5	4.5	1.5	1.0	5.5
	-40		3.0	1.3	-	1.5	1.5	4.0	3.0	1.0	0.8	4.5
Izod Impact Strength, 1/4" bar, Notched, ft-lb/in.	73	D256	6.0	3.0	8.0	2.5	4.0	5.0	5.5	2.5	1.5	7.5
	32		5.0	2.0	6.0	1.5	3.0	4.5	4.5	1.5	1.3	6.0
	-20		3.5	1.0	1.0	1.5	1.8	4.0	3.5	1.2	1.0	4.0
	-40		2.5	1.0	-	1.0	1.5	3.5	2.5	1.0	0.7	3.0
Charpy, 1/4" bar, unnotched, ft-lb/in.	73	D256	50	55	60	35	45	55	65	35	30	65
	32		45	45	25	30	40	50	55	35	30	55
	-20		40	40	-	25	40	50	50	30	30	50
	-40		40	35	-	25	40	50	45	30	30	45
Flexural Strength (yield), K psi	160	D790	3.9	7.0	-	7.8	5.0	4.0	5.0	6.3	6.3	3.7
	73		7.8	11.2	6.7	11.0	9.0	7.5	9.0	11.5	12.5	6.4
	-40		11.2	16.7	-	15.5	14.0	11.0	13.5	17.5	19.0	9.5
Flexural Modulus, K psi	160		170	290	-	290	220	180	230	280	300	190
	73		240	340	220	350	300	240	300	380	420	220
	-40		280	420	-	400	360	270	340	430	480	240

(1) Injection molded.

Table 12 presents data on 20 percent and 40 percent fiberglass reinforced ABS compositions. The glass reinforcement greatly increases the tensile and flexural properties. Also, the glass reinforcement reduces the effect of temperature on the properties. This is especially significant in the lower temperature range, 73°F to -40°F as evidenced by the small change in values.

Impact strength at -40°F is comparable to many grades of unreinforced ABS. However the reinforced ABS's are definitely inferior at room temperature.

TABLE 12. PROPERTIES OF FIBERGLASS REINFORCED ABS (ABSAFIL)(17)

Property	Temp °F	Type G 1200/20	Type G 1200/40
Tensile Strength, K psi(1)	170	8.5	9.5
	73	15.0	18.0
	-40	16.2	19.0
Elongation, %(1)	170	2.5	2.2
	73	3.0	2.5
	-40	3.0	2.8
Modulus of Elasticity, psi x 10^5(2)	170	7.5	9.0
	73	8.0	10.3
	-40	8.0	10.8
Flexural Modulus, psi x 10^5(2)	170	8.3	11.5
	73	9.2	13.0
	-40	10.0	14.5
Flexural Strength, K psi(2)	170	16.0	20.0
	73	23.0	27.0
	-40	24.5	28.0
Impact Strength, Izod, ft lb/in(3) (1/2" x 1/4" bars)	73	2.0	2.4
	-40	1.5	2.5

(1) ASTM D 638-60T

(2) ASTM D 790-59T

(3) ASTM D 256-56

SECTION IV. CELLULOSICS

Mechanical Properties

Tensile and impact tests were conducted on cellulosic materials at +77, +10, -40 and -65°F.(18) Tensile values were obtained on a Tinius-Olsen Plastiversal testing machine. Determinations in tension were made using a constant rate of load increase of 2500 psi per minute. Load-elongation graphs were automatically recorded to the point of fracture using Baldwin Models PS-6, PS-7 and PS-8 Microformer-type plastics extensometers, coupled to a Baldwin Microformer-type recorder.

Tests at 77°F were conducted in a controlled room at 50 ±2 percent relative humidity. Tests at below 77°F were conducted in an insulated housing where low-temperature air, precooled with carbon dioxide, was circulated. Temperature was within ±2°F; relative humidity was not controlled. Specimens were conditioned for two hours before testing.

Determinations were made based on Method 1011, Federal Specification L-P-406a modified for the rate of loading. The data and range are shown in Figures 27, 28, and 31.

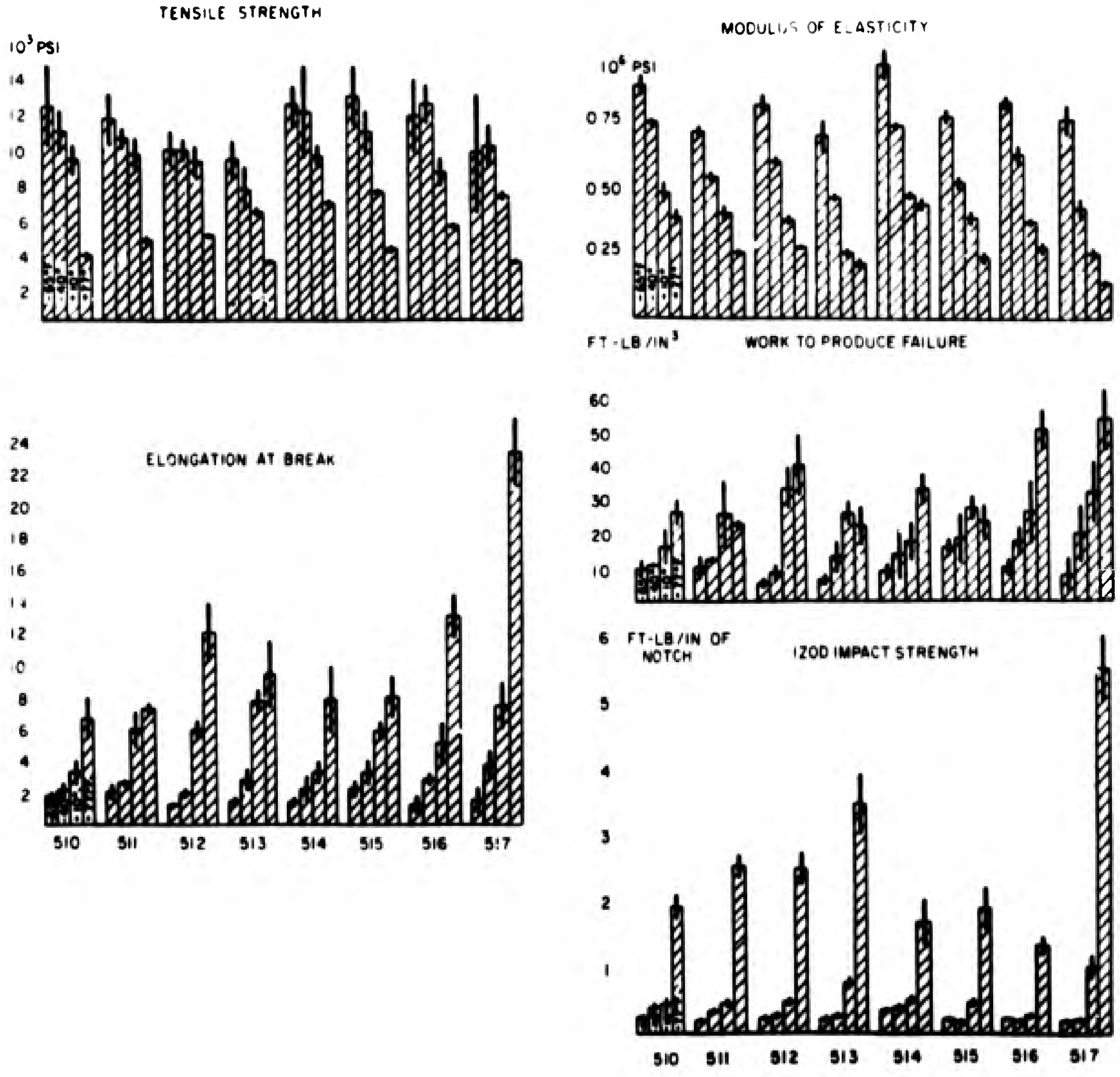
Impact tests were conducted on a Baldwin-Southwark pendulum-type impact machine conforming for accuracy to Federal Specification L-P-406. ASTM D 256-41T was used for obtaining the Izod impact data. Depending on the strength of the individual materials, either a 1- or a 2-ft. -lb. hammer was used.

For tests at lower temperatures the entire apparatus was placed in a heavily insulated cabinet. Air precooled by solid carbon dioxide was circulated in the cabinet, and the temperature was maintained to within ± 2°F. Specimens were conditioned in this cabinet at test temperature for 2 hours before the test. The results are shown in Figures 27, 28, and 31.

Cellulose Acetate

All compositions in Figure 27 showed an increase in modulus with decreasing temperatures with the largest increase occurring between -40 and -65°F. This illustrates the progressive embrittlement in this temperature range. Also indicative of embrittlement is the decrease in elongation to only 2 to 4 percent in the -40 and -65°F range. Work-to-produce-failure varied with temperature similar to elongation. Tensile strength increased with decreasing temperature.

Impact values dropped rapidly between 77° and 10°F, but changed little with further decreasing temperature.



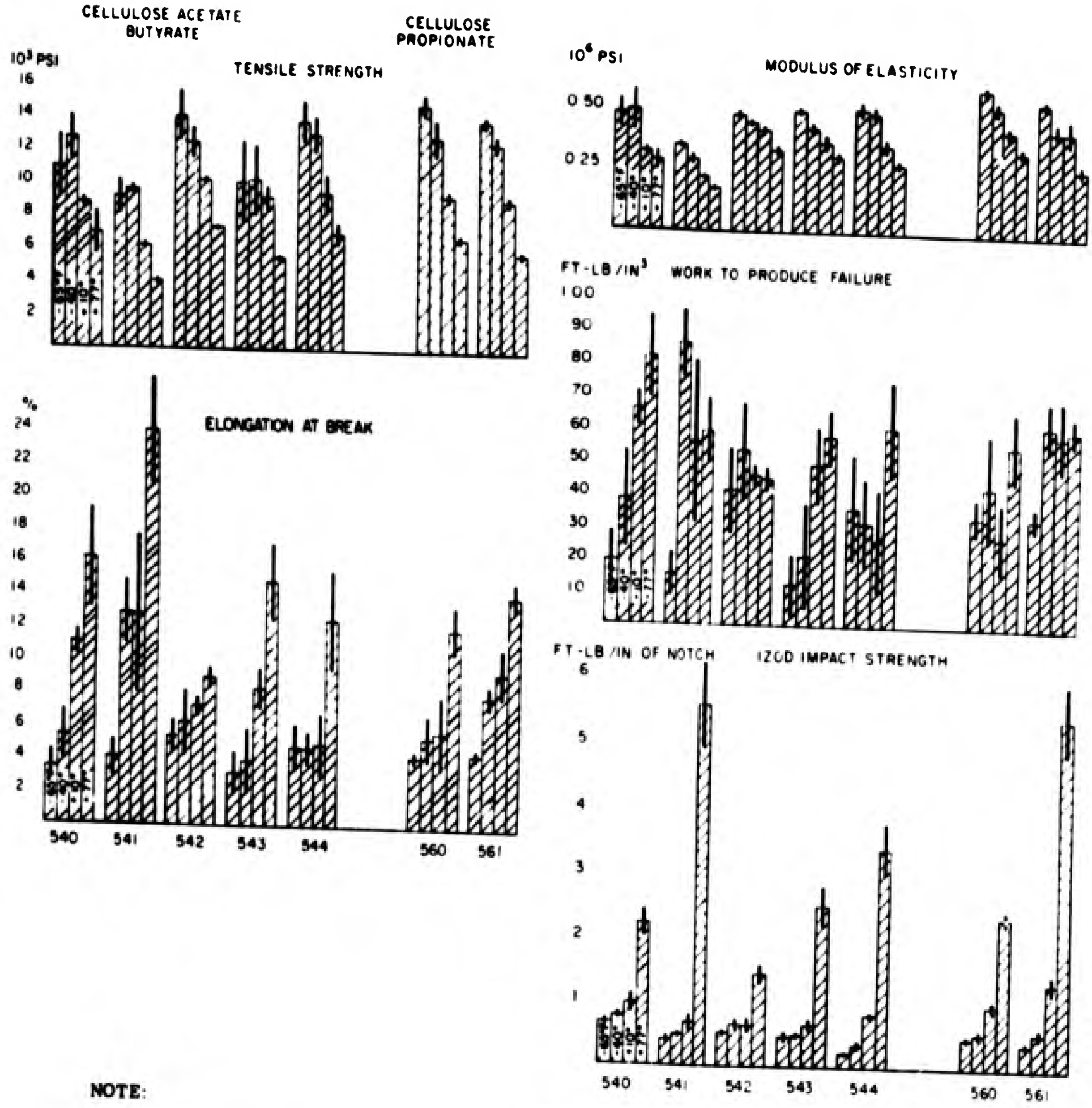
NOTE:

Materials

510	General purpose, hard	74% normal acetate, 26% mixed phthalate plasticizer[a]
511	General purpose, medium soft	67.5% normal acetate, 32.5% mixed phthalate plasticizer[a].
512	Water resistant, hard	68.75% normal acetate, 20.75% mixed phthalate plasticizer;[a] 10.5% triphenyl phosphate plasticizer
513	Water resistant, medium soft	62.5% normal acetate, 25% mixed phthalate plasticizer[a], 12.5% triphenyl phosphate plasticizer
514	Improved water resistance, hard	74% high acetyl acetate, 26% mixed phthalate plasticizer[a]
515	Improved water resistance, medium soft	67.5% high acetyl acetate, 32.5% mixed phthalate plasticizer[a]
516	Maximum water resistance, hard	68.75% high acetyl acetate, 20.75% mixed phthalate plasticizer[a], 10.5% triphenyl phosphate
517	Maximum water resistance, soft	62.5% high acetyl acetate, 25% mixed phthalate plasticizer[a], 12.5% triphenyl phosphate

[a] Mixed phthalate plasticizer is made up of dimethyl phthalate and diethyl

Figure 27. Tensile properties of cellulose acetate



NOTE:

		Material
540	General purpose, hard	Sebacic acid ester plasticizer, low
541	General purpose, medium soft	Sebacic acid ester plasticizer, medium
542	Maximum water resistance, minimum plasticizer volatility hard	Phosphoric acid ester plasticizer, low
543	Maximum water resistance, medium soft	Phosphoric acid ester plasticizer, medium
544	Low Temperature resistant, medium soft	Adipic acid ester plasticizer, medium
545	Low temperature resistant, hard	Adipic acid ester plasticizer, low
560	Only composition available, hard	Low plasticizer content
561	Only composition available, medium soft	Medium plasticizer content

Figure 28. Tensile and impact properties of cellulose acetate butyrate and cellulose propionate

Cellulose Acetate Butyrate

The cellulose acetate butyrate compositions plotted in Figure 28 varied only in type and amount of plasticizer. Of the different plasticizers selected, the sebacic acid ester gave the best elongation and impact values at room temperature, whereas the phosphoric acid ester showed a slight advantage at lower temperatures. As in the case of cellulose acetate, the most pronounced drop in elongation and impact strength occurred between 77 and 10°F. Tensile strength and modulus showed a more gradual change with temperature. Work-to-produce-failure varied somewhat erratically and no definite trend could be established.

Figure 29 shows the increase in shear modulus with decreasing temperature for typical grades of cellulose acetate butyrates.

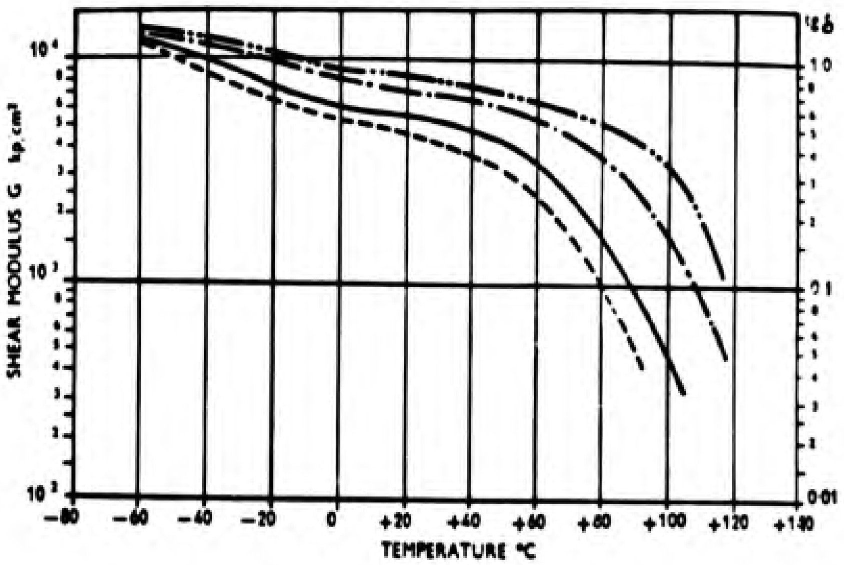


Figure 29. Shear modulus (G) versus temperature for four typical grades of cellulose acetate butyrate(16)

Cellulose Propionate

The tensile and impact properties of a low and a medium plasticized cellulose propionate composition are shown in Figure 28. As would be expected, the results reflect a slightly higher tensile strength for the harder composition with decrease in elongation and rigidity. All differences at -65° F were negligible.

Figure 30 presents the shear modulus of four cellulose propionate grades at various temperatures.

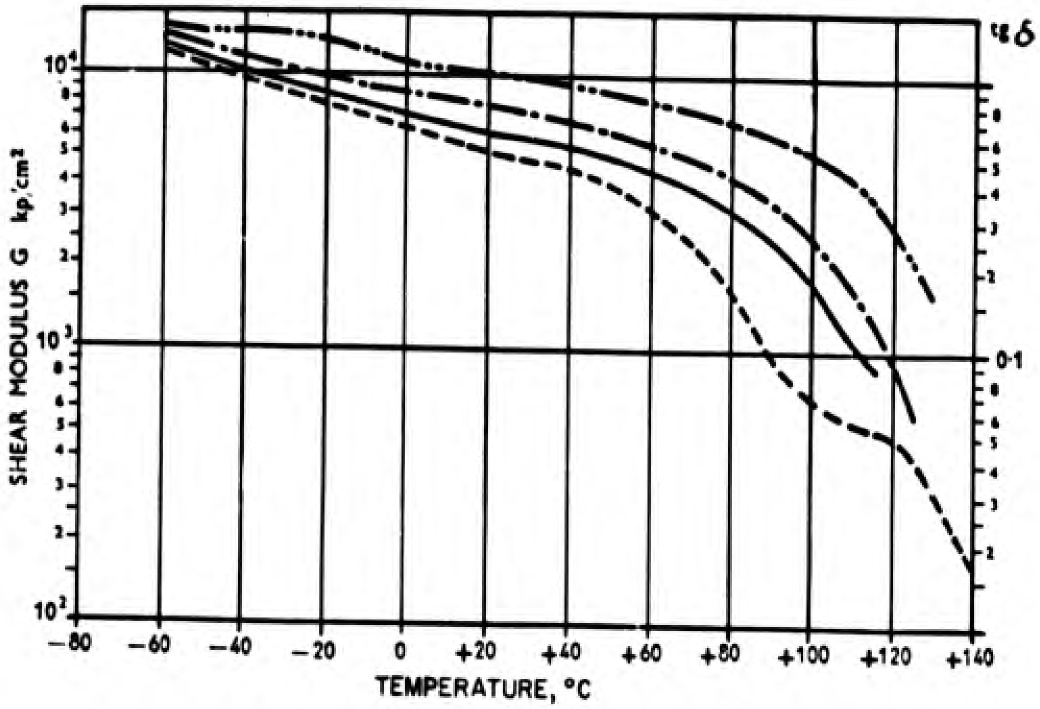


Figure 30. Variation in shear modulus (G) versus temperature for four grades of cellulose propionate(16)

Impact tests were conducted down to -50°F on a 4 inch square sheet and a 1-11/16" diameter vacuum formed blister.(19) The sheet was looped into a "c" and the ends stapled to cardboard. The fold was parallel to the length (machine) direction and was not creased.

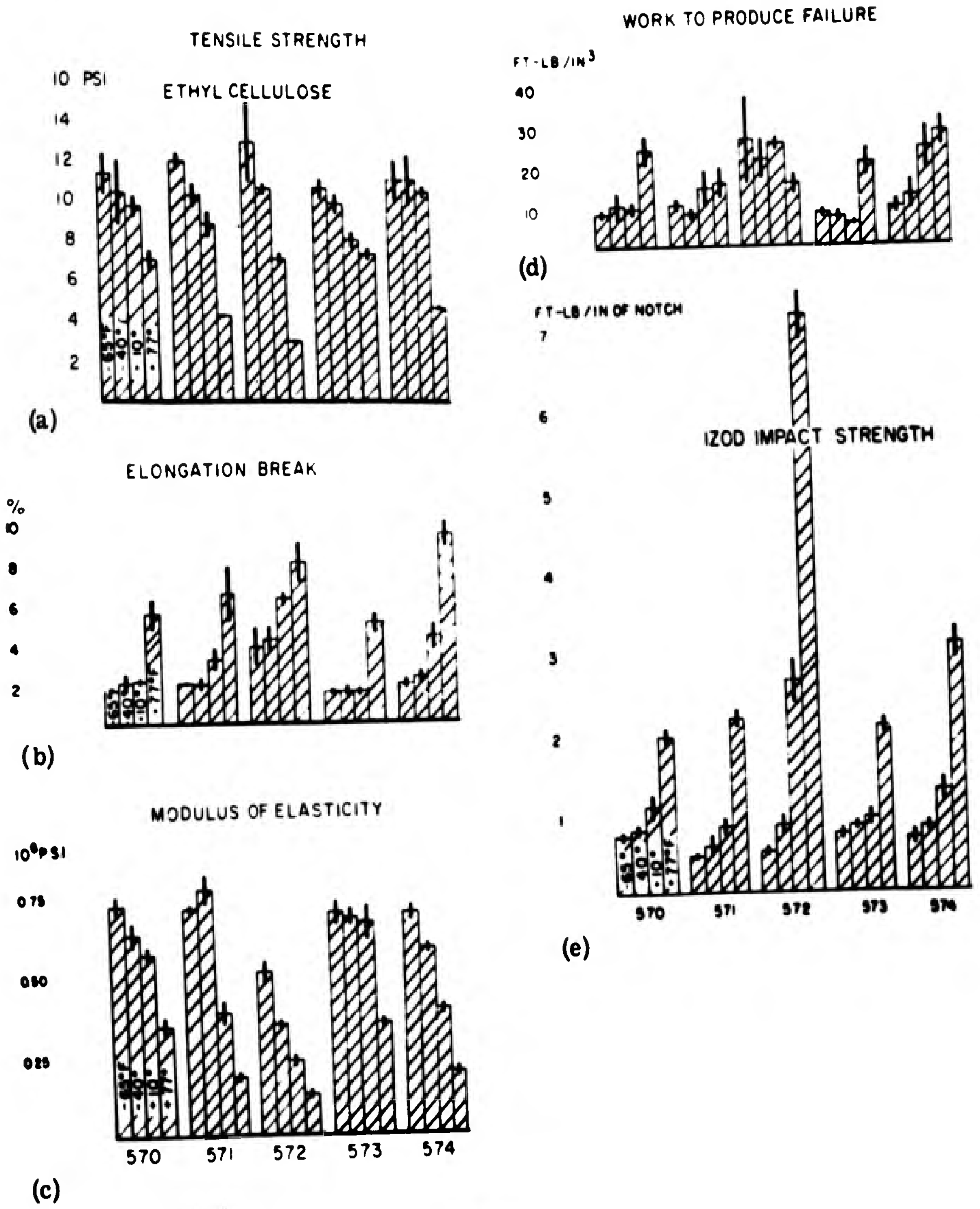
The impact tester was a 1 pound weight, 1-1/2" wide and 3/16" thick with a rounded impacting edge. The weight was dropped one foot on the center of the blister and on the center and at right angles to the fold of the sheet.

The results were as follows: sheet specimen - no failures to -50°F; blister - 0, 15 and 25 percent failures at -30°, -40° and -50°F respectively.

Ethyl Cellulose

Figure 31 shows the tensile and impact properties of five ethyl cellulose specimens.(18) The properties of three different compression molded grades of ethyl cellulose are charted in Figure 32 a-d, from -40°F to 130°F. As in the case of the other cellulosic plastics, the general trend of the data with decreasing temperature was normal.

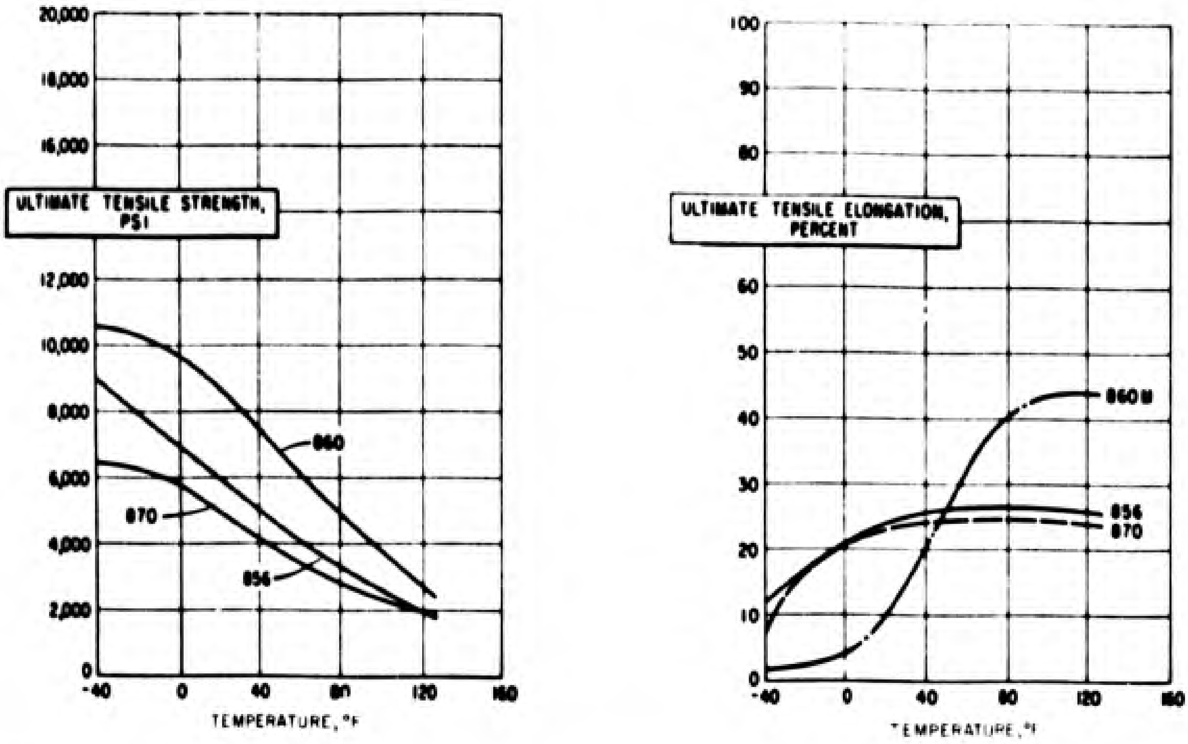
Again, it is interesting to note that the plasticizers in specimen 572 (Figure 31) were of little benefit at -65°F.



NOTE:

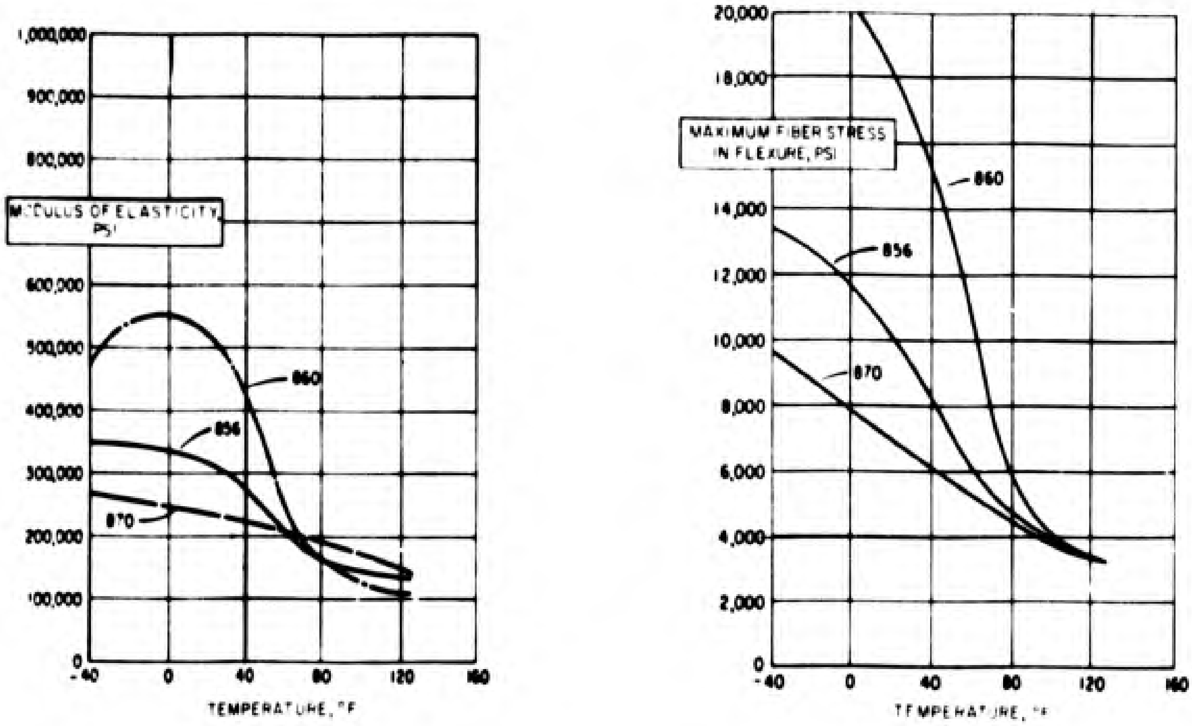
		Material
570	General purpose, hard	90% K100 ethyl cellulose, 7% Dow V2 resin, 3% triphenyl phosphate
571	General purpose, medium soft	80% K100 ethyl cellulose, 14% Dow V2 resin, 6% triphenyl phosphate
572	Nitroglycerine resistant, high impact	77% N100 ethyl cellulose, 23% Cerisen
573	General purpose, hard	90% K100 ethyl cellulose, 7% Dow V2 resin, 3% tricresyl phosphate
574	General purpose, soft, improved water resistant	80% K100 ethyl cellulose, 14% Dow V2 resin, 6% tricresyl phosphate

Figure 31. Tensile and impact properties of ethyl cellulose



a. ASTM D 638-58T

b. ASTM D 638-58T



c. ASTM D 638-58T

d. ASTM D 790

NOTE: Compression Molding Material

856	Flexible grade, general purpose	860	General purpose
		870	High impact grade

Figure 32. Mechanical properties of ethyl cellulose (ethyocel)(19)

SECTION V. FLUOROCARBONS

Mechanical Properties

Mechanical properties of fluorocarbon plastics are highly dependent on degree of crystallinity, also the size, number and arrangement of crystals, and, for this reason, a wide range has been given in some of the properties. TFE, which is cooled rapidly from the melt stage or quenched, will have relatively low crystallinity and a relatively high tensile strength and elongation, coupled with low rigidity. Slower cooling, on the other hand, will lead to a higher crystallinity with relatively low tensile strength and elongation but higher rigidity. Orientation also affects tensile properties. In general, tensile strength is greater in the direction of orientation but ultimate elongation is lower.

Tensile stress-strain curves for polytetrafluoroethylene (TFE) and fluorinated ethylene propylene (FEP) in Figure 33 show that yield occurs at high deformations. Also, elastic response deviates from linearity at strains of only a few percent. Figure 34 gives the tensile properties of FEP film over a wide temperature range.

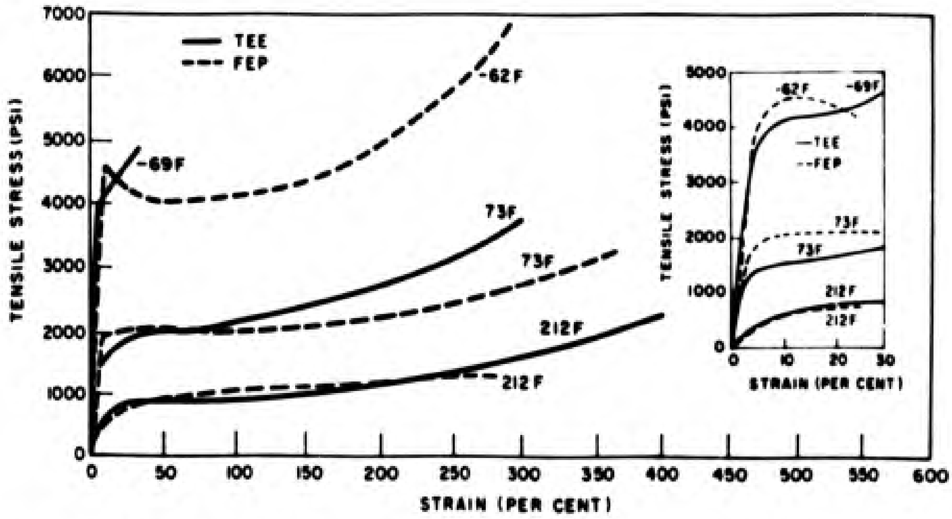


Figure 33. Tensile stress versus strain (to fracture) of TFE and FEP(21)

The effects of crystallinity on the tensile yield strength, elongation and flexural modulus of TFE are shown in Table 13. This data supports the previous statement that tensile strength and elongation decrease with increased crystallinity and rigidity increases.

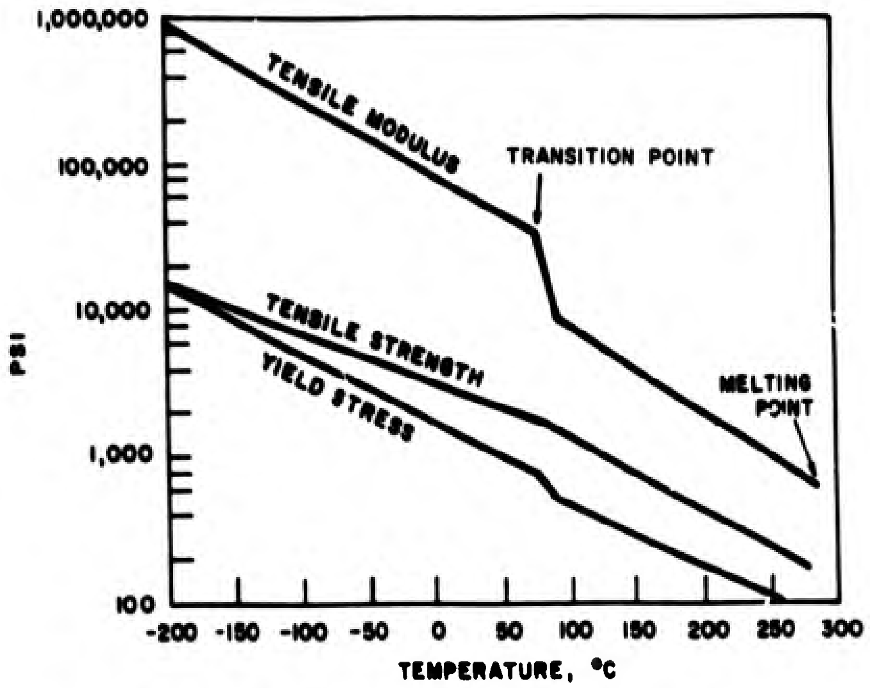


Figure 34. Tensile properties versus temperature of Teflon FEP film(26)

TABLE 13. EFFECT OF CRYSTALLINITY ON TEFLON TFE FLUOROCARBON RESINS(22)

Property	°F	Crystallinity, %						
		50	55	60	65	70	75	80
Tensile Yield Strength, Kpsi(1)	212	1.20	1.14	1.08	1.04	0.98	0.92	0.83
	73	1.90	1.80	1.60	1.40	1.30	1.20	1.00
	-40	3.80	3.20	2.80	2.40	2.30	2.10	1.60
Elongation, %(1)	212	—	44	39	33	25	—	—
	72	58	50	40	32	25	14	6
	-40	40	35	30	25	20	10	8
Flexural Modulus(2) Elasticity, Kpsi	212	26	33	41	49	57	64	72
	73	45	67	88	110	132	154	176
	-88	210	240	270	310	340	370	400

(1) ASTM D638-52T modified for test specimens 3/16" x 1/16" x 7/8" for low-and high-temperature tests. Crosshead speeds of 0.02 in./min. (Baldwin) for low temperatures and 0.05 in./min. (Instron) for high temperatures.

(2) ASTM D790

The impact strength of TFE and FEP resins is very high and is retained over a wide temperature range. Average values of Teflon specimens subjected to tensile-impact and Izod-impact tests are given in Table 14. Figure 35 compares the tensile impact strength of Teflon FEP and TFE films with polyethylene, acrylic and nylon films.

TABLE 14. IMPACT STRENGTH OF TFE AND FEP RESINS(23)

Property	Temp. (°F)	TFE (Teflon 1)	FEP (Teflon 100X)
Tensile Impact (ft-lb/cu in.)	73	320	1020
	-65	105	365
Izod Impact (1) (ft-lb/in.)	73	2.9	--(2)
	-65	2.3	2.9

(1)ASTM D256-56
(2)Sample bends

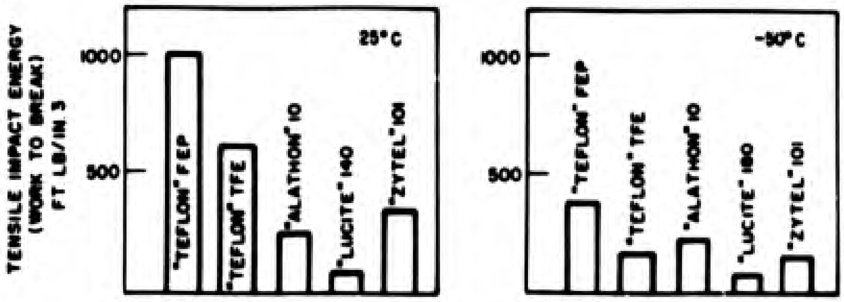


Figure 35. Comparison of tensile impact energy of FEP and TFE with other films(24)

Electrical Properties

TFE and FEP fluorocarbon resins differ from other organic insulations in that their electrical properties are extremely stable over a wide range of temperature and frequency.

Figure 36 shows that the dissipation factor and dielectric constant of TFE molding material remain constant over a temperature range of -40 to +464° F.

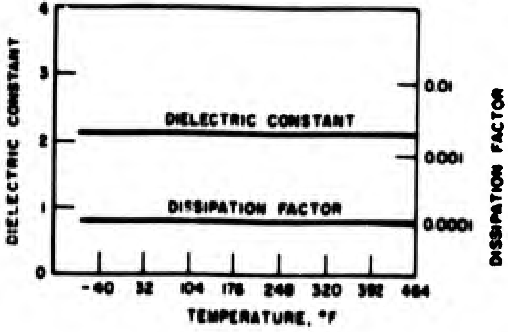


Figure 36. Dielectric constant, dissipation factor versus temperature of Teflon TFE (25)

The dielectric constant of Teflon FEP film is independent of thickness and increases slightly with lower temperatures, as shown in Figure 37.

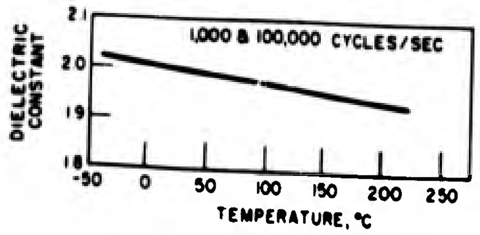


Figure 37. Dielectric constant versus temperature of Teflon FEP film(26)

Figure 38 shows the consistently low dissipation factor of FEP film over a broad range of temperatures.

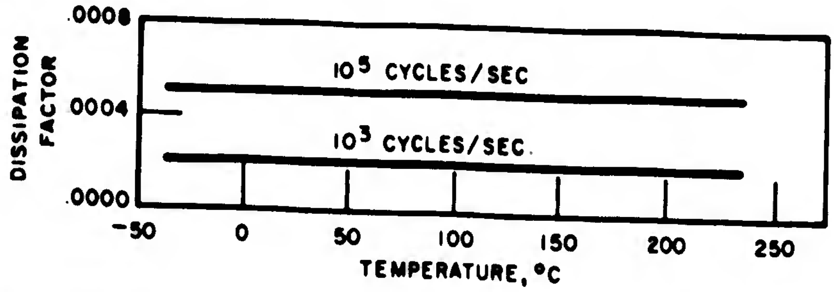


Figure 38. Dissipation factor versus temperature of (Teflon) FEP film(26)

The dielectric properties of polychlorotrifluoroethylene (Kel-F grade 300) were investigated over a wide range of temperatures, -58° to 482°F (-50° to 250°C), and frequencies (0.1 c|s to 8.6 kmc|s)(27). Tests were conducted on specimens of known thermal history and at 80, 73, 44 and 12 percent crystallinity to correlate dielectric behavior with degree of crystallinity, temperature and frequency.

Figures 39 and 40 show the frequency dependency of loss index and dielectric constant, at the indicated temperatures, for 80, 73, 44 and 12 percent crystallinity. These plots illustrate the behavior with temperature of the most prominent loss process in the polymer.

The surface resistivity and volume resistivity of both TFE and FEP were higher than the other materials tested, over the temperature range -40 to 440° F.(28) Results are shown in Table 15.

Figure 41 gives the electrical strength of TFE versus temperature and frequency.

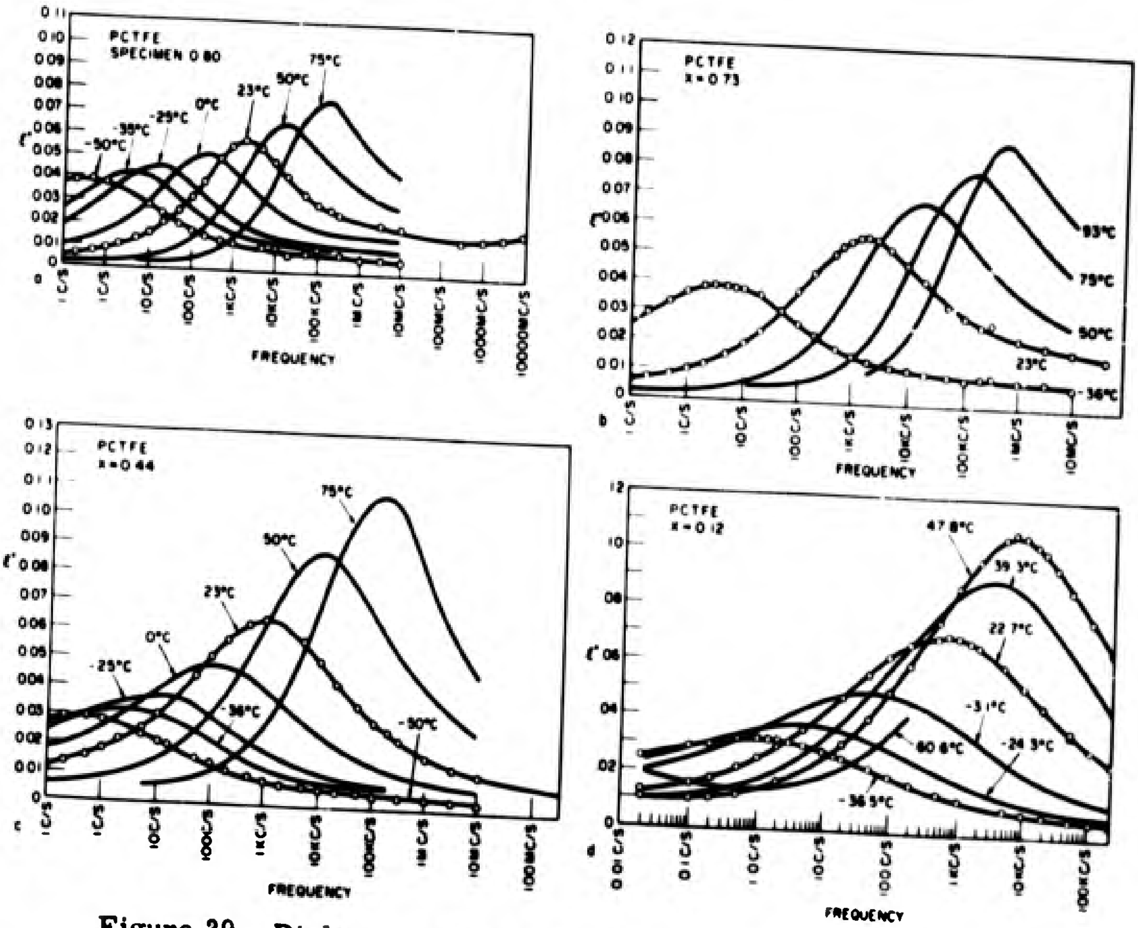


Figure 39. Dielectric loss index versus frequency at various temperatures at four crystallinities(27)

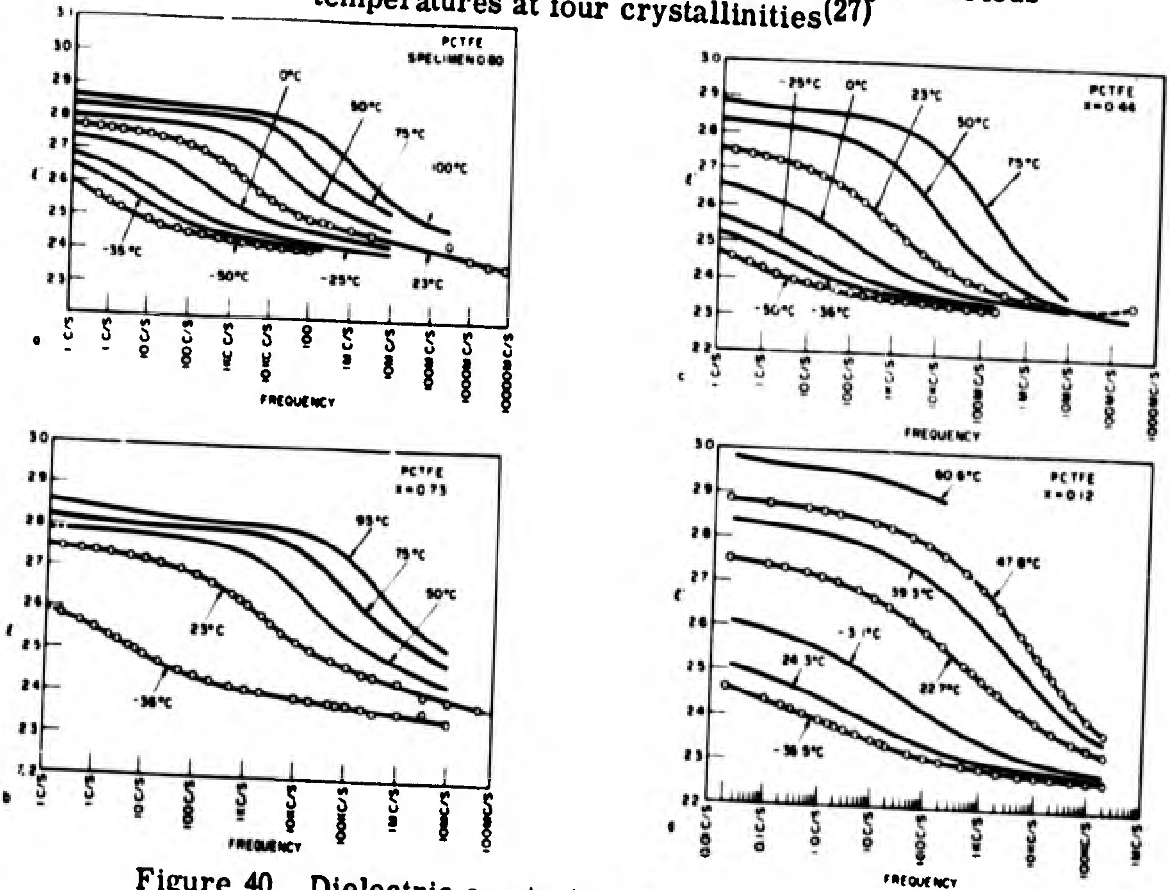


Figure 40. Dielectric constant versus frequency at various temperatures at four crystallinites(27)

TABLE 15. SURFACE RESISTIVITY AND VOLUME RESISTIVITY OF TFE AND FEP(28)

RESINS	VOLUME RESISTIVITY, OHM-CM.	SURFACE RESISTIVITY, OHMS/SQ.	MEASURED TEMP. RANGE*
TFE resins	10^{18}	10^{16}	-40°F. (-40°C.) to 440°F.
FEP resin	10^{18}	10^{16}	-40°F. (-40°C.) to 440°F.
*Limits of measuring equipment.			

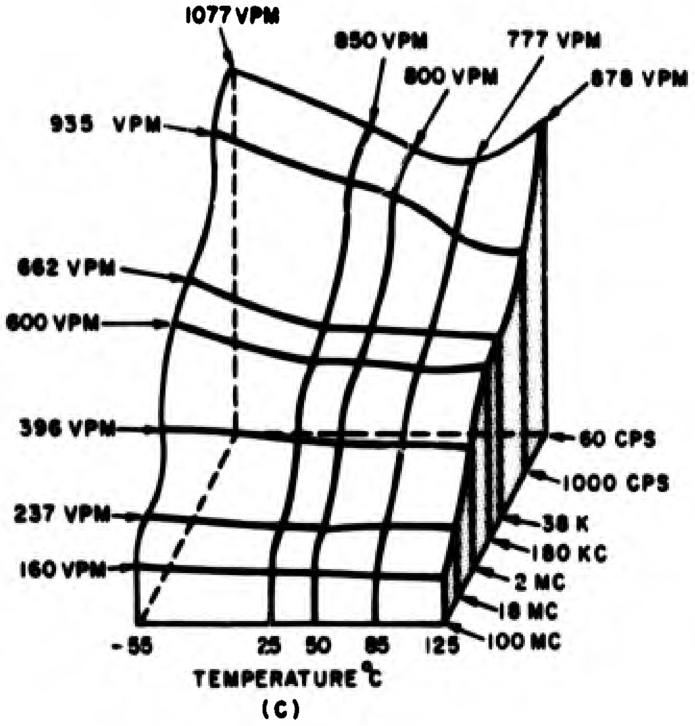


Figure 41. Electrical strength of TFE(29)

Thermal Properties

Figures 42 and 43 depict the effect of temperature on the linear expansion of three fluorocarbon molding materials. A drop in linear expansion occurs between 65 and 77° F and allowances should be made for applications crossing this transition zone.

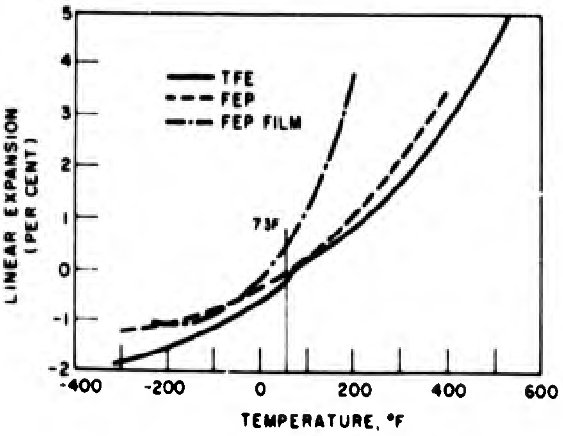


Figure 42. Effect of temperature on linear expansion of TFE and FEP molding and FEP film material (21,26)

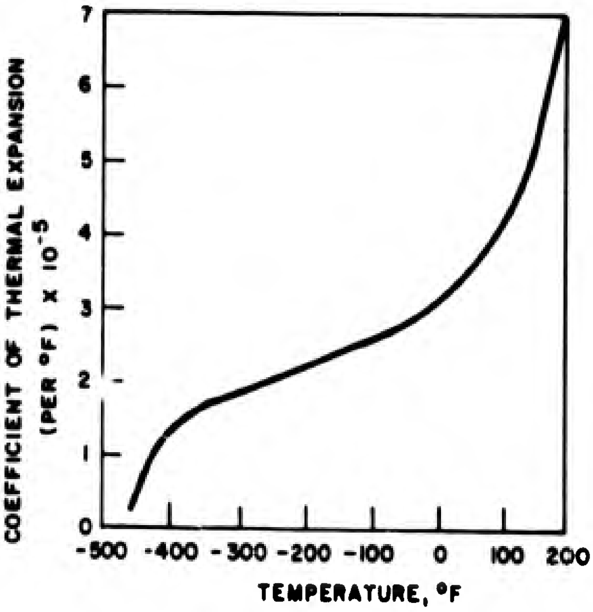


Figure 43. Linear coefficient of thermal expansion for CTFE (21)

The total linear thermal expansion was determined on four TFE materials; two laminates and two filled materials. (30) The materials and codes are given in Table 16. Measurements were made in two directions (thickness and normal to thickness) on a Leitz dilatometer from 75° to 100° F. The data is shown in Figures 44 through 47.

The large differences in the expansion in the two directions of the laminates (materials I and L) is explained as follows: A laminate is composed of alternate layers of reinforcement and resin. The expansion in the thickness direction approaches the sum of the expansion of each layer and results in a value between that of the two materials. In the normal to thickness direction, the bonded resin and reinforcement try to expand at different rates due to differences in their respective expansion coefficients. However, since they are bonded, the higher modulus material will tend to control the overall expansion. Therefore, the expansion of the composite will be closer to that of the higher modulus material. This is only applicable, of course, if the resin to reinforcement bond withstands the thermal stress.

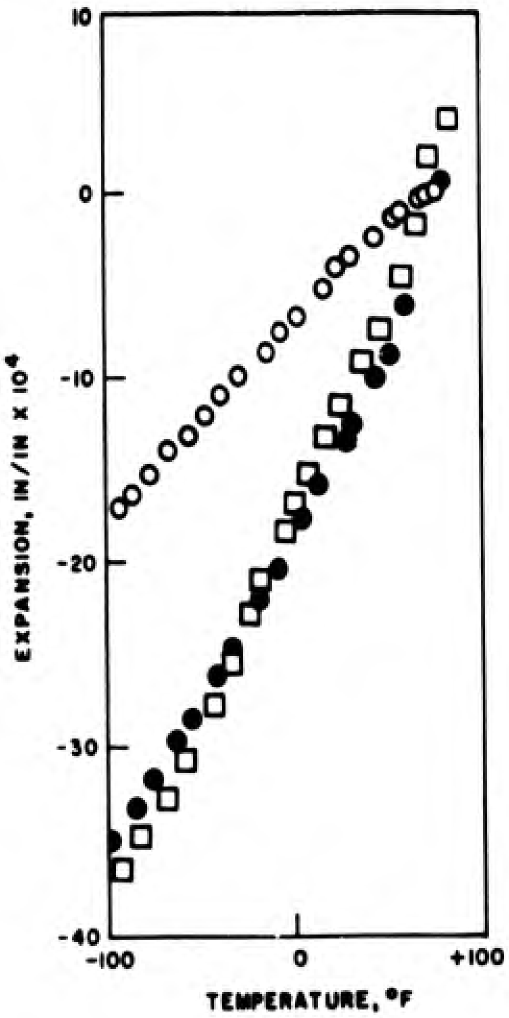
Also, many cloth reinforcements are constructed with more threads per inch in the warp direction than in the weave direction. Consequently, the expansion in the warp direction should be controlled by the reinforcement to a greater extent.

The differences in the warp and weave directions in Figure 45 were attributed by the authors to preferential fiber orientation during processing. The differences in material J, a chopped-fiber filled specimen were also due to preferentially aligned fibers, parallel to the panel's surface due to the manufacturing process. (30)

TABLE 16. MATERIAL DESCRIPTION OF TFE SPECIMENS

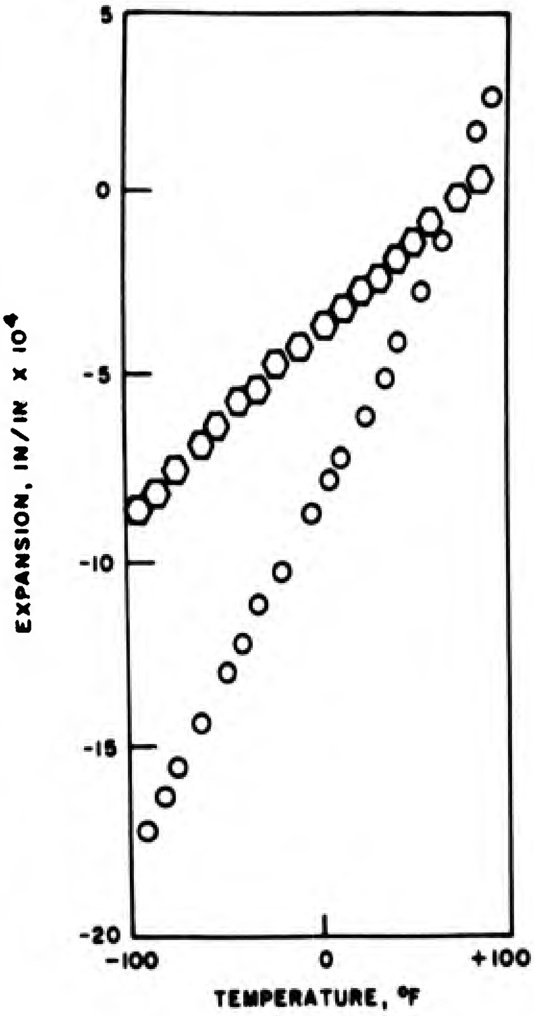
Composite System	I-1	J-1	K-1	L-1
Manufacturer	Fluorocarbon	Raybestos-Manhattan	Fluorocarbon	Raybestos-Manhattan
Resin Source	DuPont	DuPont	DuPont	DuPont
Pre-preg Source	-	-	-	-
Resin Content	75%	79%	80%	75.3%
Resin System				
Tradename	Teflon TFE	Teflon TFE	Teflon TFE	Teflon TFE
Chemical Type	Fluorocarbon	Fluorocarbon	Fluorocarbon	Fluorocarbon
Catalyst	-	-	-	-
Curing Agents, Additives, etc.	-	Copper, manganese blue ceramic pigment	-	-
Reinforcement System				
Tradename	"E" Glass	Glass	Graphite	Asbestos
Generic Name	Glass Cloth	-	Graphite cloth	-
Fabric Designation	Style 116	-	Style WCB[7]	-
Form of Reinforcement	Fabric	Chopped fibers	Chopped fabric 1/2" x 1/2" squares	Long fiber asbestos Felted sheet
Finish	-	-	-	-
Lay-up	Parallel Laminate	Molded	Molded	Parallel mat laminate
Number of Plies	-	-	-	-
Reinforcement Orientation	Cross-plied at 90°	Random	Random	-
Cure Cycle				
Pressure	1500 psi	2000 psi	1250 psi	2000 psi
Temperature	740° F	720° F	740°F	720°F
Temperature-Time History	B-stage for 15 min. at 150°F and 740°F. for 20 min. Air cool. Repeat until each layer has proper % resin. Place sheets in mold in 690°F press. Hold 1 hr. at contact pressure, raise slowly to 1500 psi. Hold for 45 min. Transfer to water cooled press and cool at 1500 psi	Sintered 2 hours at 720°F.	B-stage by repeated dipping, heating at 150°F and sintering at 740°F. Place squares in mold in 690°F press. Dwell 20 mi. at contact pressure. Raise to 1250 psi and hold for 30 min. Cool under pressure.	Sintered 2 hours at 720°F.

The data in Figures 44 - 47 show the transition, previously discussed, at about 68° F. It should be noted that the change in length at the transition temperature was less in the directions having lower expansions. This again is due to the control of the reinforcements over the expansion of Teflon.



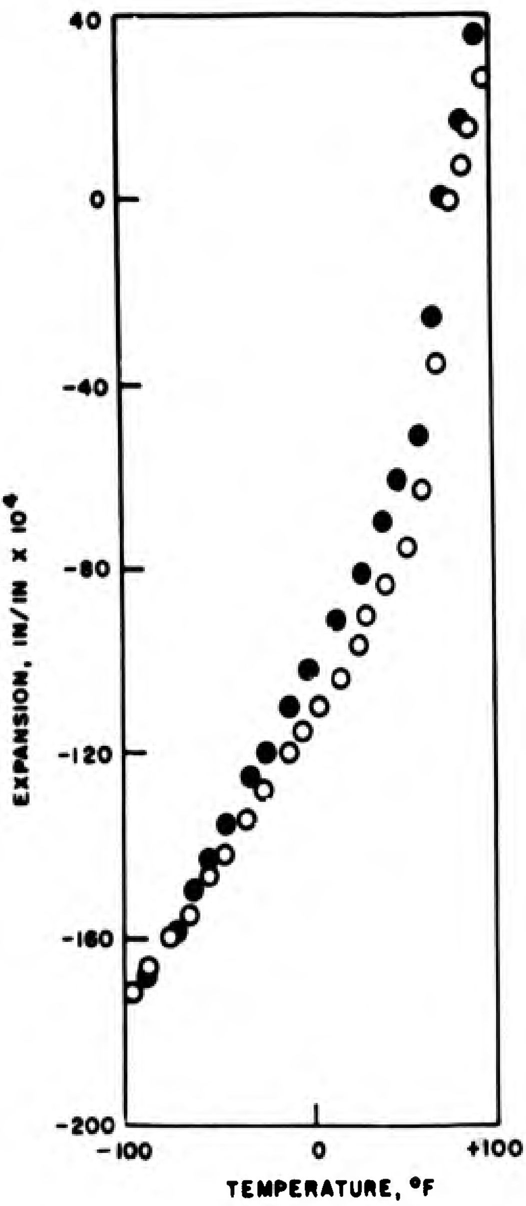
○ Normal to thickness - Material I
● Normal to thickness - Material J
☐ Normal to thickness - Material K
Points above 75 °F were taken on the same sample and were taken after those below 75 ° F.

Figure 44. Total linear thermal expansion of material I, J, and K (thickness) (normal to thickness direction) (30)



○ Weave - virgin
Points above 75°F were taken on same sample and after those below 75°F.
○ Warp - virgin
Points above 75°F were taken on same sample and after those below 75°F.

Figure 45. Total linear thermal expansion of TFE material L (normal to thickness direction) (30)

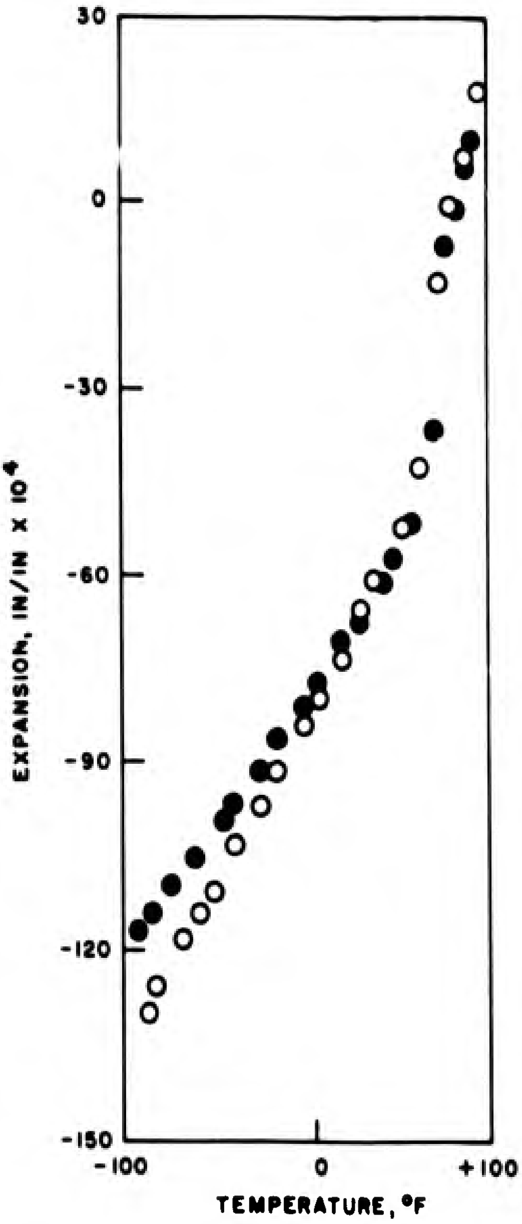


○ Thickness - Material I

● Thickness - Material L

Points above 75°F were taken on same sample and were taken after those below 75°F.

Figure 46. Total linear thermal expansion of TFE (material I and L thickness direction) (30)



○ Thickness - Material J

● Thickness - Material K

Points above 75°F were taken on same sample and after those below 75°F.

Figure 47. Total linear thermal expansion of material J and K (thickness) (30)

The thermal conductivities of the materials listed in Table 16 were determined on a modified guarded hot plate at one atmosphere in helium and one atmosphere in nitrogen. (30) The results in two directions are plotted in Figures 48 to 51.

Thermal conductivites are influenced by temperature, environment, type reinforcement, reinforcement orientation, type of resin, density, void content and fabrication history.

The influence of environment can be seen, as the presence of helium gas gives consistently higher conductivities.

The thermal conductivity of materials J and I, unexpectedly, was lower than pure TFE Teflon. This was attributed to the large number of resin-glass interfaces (high void content) and accounts for the low conductivity obtained in the nitrogen environment. The high void content was also confirmed by the large increase in conductivity in helium gas. There was a large increase in thermal conductivity in the thickness direction of material J (Figure 48). This was construed to mean that the fibers were not randomly oriented, but had their length perpendicular to the original thickness direction.

The graphite and asbestos reinforced TFE materials (K and L) had about the same conductivities as pure Teflon. (30)

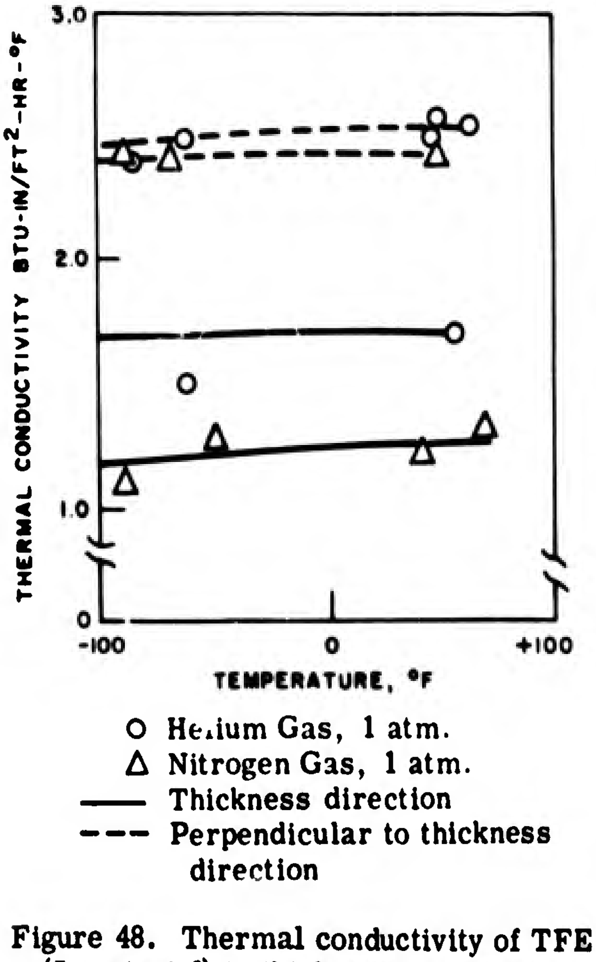


Figure 48. Thermal conductivity of TFE (J-material) in thickness and perpendicular to thickness direction (30)

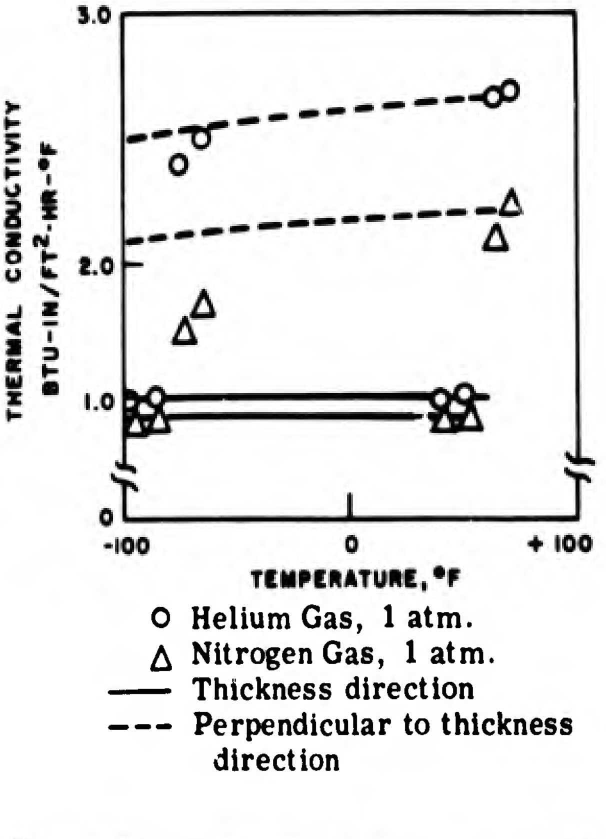


Figure 49. Thermal conductivity of TFE (I-material) in thickness and perpendicular to thickness direction (30)

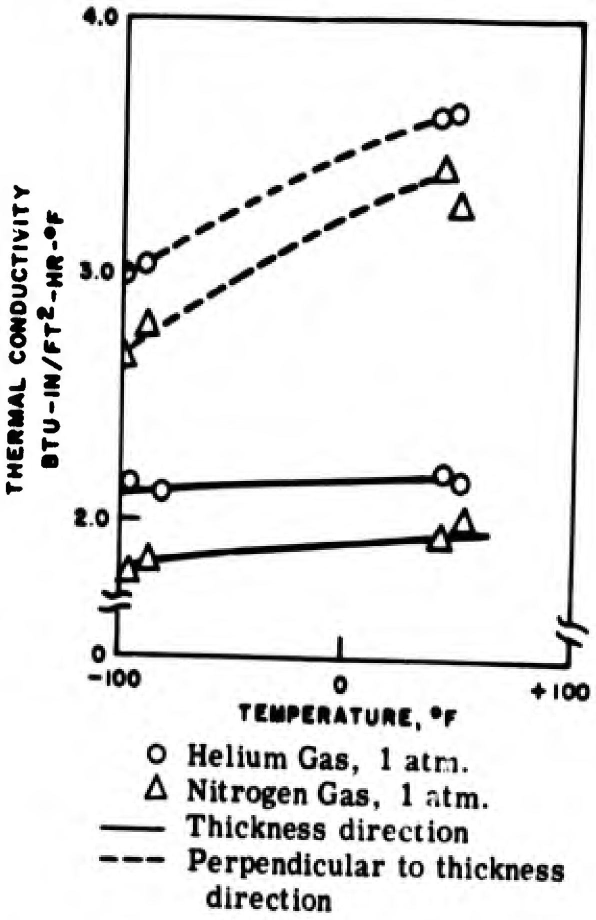


Figure 50. Thermal conductivity of TFE (K-1 material) in thickness and perpendicular to thickness direction (30)

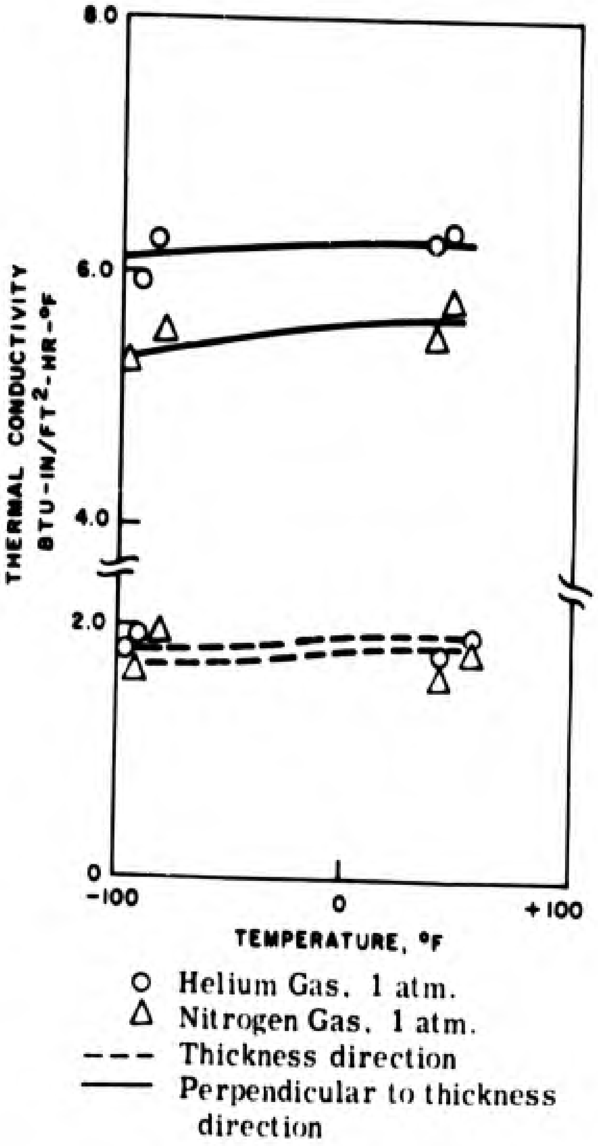


Figure 51. Thermal conductivity of TFE (L-material) in thickness and perpendicular to thickness direction (30)

SECTION VI. IONOMERS

A new family of thermoplastics, Ionomers, became commercially available in 1965. Because of this relative newness, very little low temperature data are available.

Properties of ionomers vary with composition and depend on molecular weight, degree of crystallinity and type of metal ion. "Unlike polyethylenes, density of ionomers is not a crystalline-dependent property and should not be used to predict other physical properties." (58)

Of the interested properties only the below thermal data was found:

Thermal Properties*	Film, Coating	Molding Grade	Electrical Grade
Brittleness temperature	<-160	<-160	<-160
Coef of thermal expansion (-4 to +90° F) in/in/°C x 10^{-5}	12 to 13	12 to 13	12 to 13

* Surlyn A Resins

SECTION VII. POLYAMIDES

Mechanical Properties

Tables 17, 18 and 19 present data on the mechanical properties of various nylon materials at three designated temperatures. Polyamides, more than other thermoplastics, are influenced by moisture content. Moisture content reduces strength and stiffness and increases elongation and impact resistance. Therefore, moisture, in addition to crystallinity (previously discussed), will affect low temperature properties.

Figures 52 to 56d show the ultimate tensile strength, yield point and modulus of elasticity of polyamides over a range of temperatures. From this data it can be seen that polyamides react normally to decreasing temperature (strength and modulus increase, elongation and impact strength decrease). Also, the previous statement concerning moisture is verified.

TABLE 17. PROPERTIES OF POLYAMIDES (ZYTEL, BLUE C)

Property(1)		ZYTEL*																				Blue C**	
		101		42		105		31		38		109		53		211		63		69		10 V	20 V
Water, %(2)	°F	0.2	2.5	0.2	2.5	0.2	2.5	0.2	1.5	0.2	1.5	0.2	2.5	0.2	3.5	0.2	3.0	0.3	3.4	0.2	3.4	0.25	0.25
Tensile Strength, Kpsi(3)	170	9.0	—	8.5	—	8.7	—	5.3	—	—	—	7.6	—	—	—	—	—	5.0	4.4	—	—	9.0	—
	73	11.8	11.2	12.6	11.2	13.1	9.0	8.5	7.1	8.2	6.6	12.4	9.6	—	10.2	9.0	8.7	7.4	7.5	8.0	4.7	11.9	11.0
	-40	15.7	13.5	—	16.1	18.7	17.0	12.0	12.1	—	—	10.2	16.0	—	—	16.1	13.1	13.0	12.7	11.8	8.7	16.0	13.7
Elongation at Break, %(3)	170	340	—	155	—	145	—	300	—	—	—	250	—	—	—	—	—	400	600	—	—	350	—
	73	80	300	90	300	30	200	85	220	240	300	40	330	300	400	290	285	300	370	300	330	65	300
	-40	20	20	—	35	10	15	20	30	—	—	10	20	—	—	25	36	100	20	60	70	22	—
Yield Stress, Kpsi(3)	170	8.7	—	8.5	—	8.9	—	4.2	—	—	—	6.6	—	—	—	—	—	—	—	—	—	6.9	—
	73	11.8	8.5	12.6	8.6	13.1	9.0	8.5	7.1	8.5	7.0	12.4	6.7	—	3.1	7.4	5.9	6.0	2.7	—	1.5	11.9	8.2
	-40	15.7	—	—	16.1	18.7	17.0	12.0	12.1	—	—	—	—	—	—	16.1	13.1	—	12.7	11.8	—	16.0	—
Elongation at Yield, %(3)	170	30	—	30	—	25	—	30	—	—	—	—	—	—	—	—	—	—	—	—	—	30	—
	73	5	25	5	30	5	25	5	30	—	—	—	—	—	—	20	30	—	—	—	25	4	25
	-40	5	—	—	5	5	5	10	13	—	—	—	—	—	—	10	8	—	—	—	—	4	—
Flexural Modulus, Kpsi(4)	170	100	—	100	—	—	—	70	—	70	—	87	—	—	—	80	80	18	—	—	—	110	—
	73	410	175	410	175	—	191	280	190	280	180	413	107	340	66	245	108	150	41	30	27	415	170
	-40	470	500	470	500	—	—	320	370	320	370	463	561	—	—	—	—	—	480	—	173	470	490
Brittleness Temperatures, °F(5)	—	-112	-85	-148	-121	—	—	-130	-85	-166	-130	—	—	—	—	—	—	—	—	—	—	-112	-85
Izod Impact Strength (6)	73	0.9	2.0	1.3	—	0.9	2.0	0.6	1.6	1.2	—	0.7	4.1	—	—	2.5	—	No(7)	—	No(7)	—	0.9	2.1
	-40	0.6	0.45	—	—	—	0.45	—	—	—	—	0.4	0.4	—	—	—	—	0.7	—	0.4	—	0.6	0.4

(1) Data shown are average values and should not be used for specifications. Many of these properties are determined on molded bars and are subject to variations caused by molding conditions, design, etc.

(2) Test values reported at 0.2% water are typical of those in the as-molded condition. Values at 1.5 to 3.4% water describe the states reached in equilibrium with the atmosphere at 50% R. H.

(3) ASTM D 638

(4) ASTM D 790

(5) ASTM D 746

(6) ASTM D 256

(7) No Break

*Ref - 31
**Ref - 5

NOTES: NYLON 6/6
"Zytel" 101 - General purpose.
"Zytel" 105 - Weather stabilized.
"Zytel" 42 - High viscosity for extrusion.
"Blue C" 10V - Unmodified, general purpose.
"Blue C" 20V - Unmodified, gen purpose, lubricated
NYLON 6/10
"Zytel" 31 - General purpose, unmodified
"Zytel" 38 - Same, higher melt viscosity

NYLON 6
"Zytel" 211 - General purpose
COPOLYMERS
"Zytel" 109 - General purpose
"Zytel" 53 - Heat stabilized.
"Zytel" 55 - Heat stabilized.
"Zytel" 63 - General purpose, low melting
"Zytel" 69 - Same

TABLE 18. MECHANICAL PROPERTIES OF PLASTON 8200, 8201 AND 8205 (DRY AS MOLDED)(33)

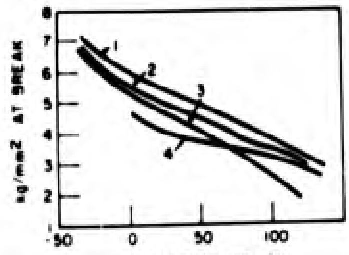
Property	Temperature		
	175°F	75° F	-40°F
Tensile Strength Ultimate, Kpsi(1)	9.9	11.8	17.4
Tensile Yield Stress, Kpsi(1)	4.3	11.8	17.4
Tensile Modulus, Kpsi(1)	60	380	445
Elongation, Ultimate, %(1)	310	200	5
Compressive Modulus, Kpsi(2)	75	248	252
Flexural Modulus, Kpsi(3)	75	395	435
Flexural Strength, Kpsi(3)	3.4	16.4	21.0

(1) ASTM D 638-58T
(2) ASTM D 695-54
(3) ASTM D 790-58T

TABLE 19. MECHANICAL PROPERTIES OF FE 2421(32)

	Temperature (° F)			
	73	0	-20	-40
Tensile Strength (p. s. i.)	8,320	14,820	16,570	17,990
Yield Strength (p. s. i.)	8,320	14,820	16,570	17,990
Elongation (%)	250	23	15	14
Flexural Modulus (p. s. i.)	66,500	336,000	426,000	463,000

Moisture: 0.19-0.23%
¹Data obtained in accordance with ASTM D 759.



1. Medium viscosity moulding grade, 2. Medium viscosity moderately flexible moulding grade, 3. Medium viscosity very flexible moulding grade and 4. Contains molybdenum disulphide.

Figure 52. Maximum tensile strength of nylon 11(16)

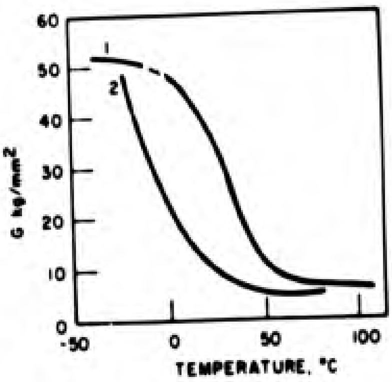


Figure 53. Variation of modulus of rigidity in torsion (G) for two typical grades of nylon 11 (16)

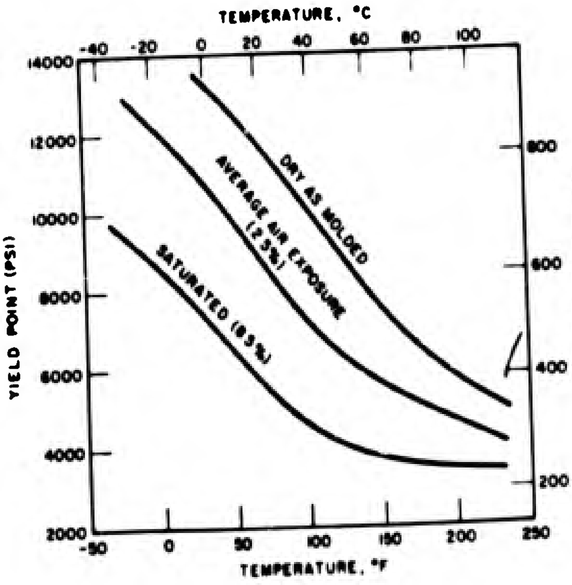


Figure 54. Yield point of nylon 6/6 (Zytel 101)versus temperature and moisture content(31)

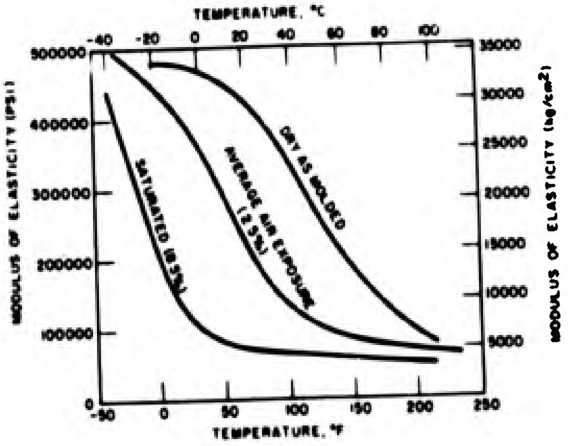


Figure 55. Modulus of elasticity of nylon 6/6 (Zytel 101)versus temperature at various moisture contents(31)

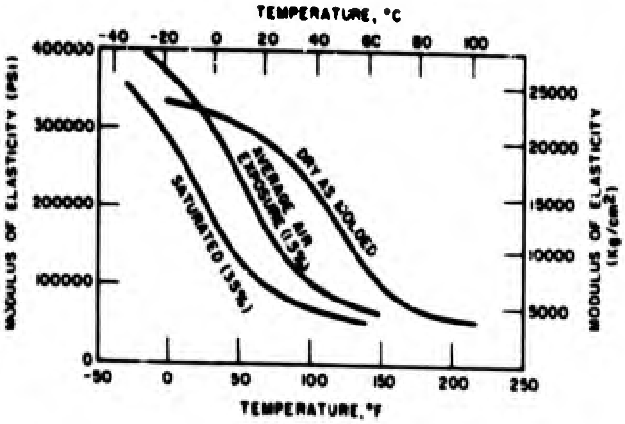


Figure 56. Modulus of elasticity of nylon 6/6 (Zytel 31) versus temperature at various moisture contents(31)

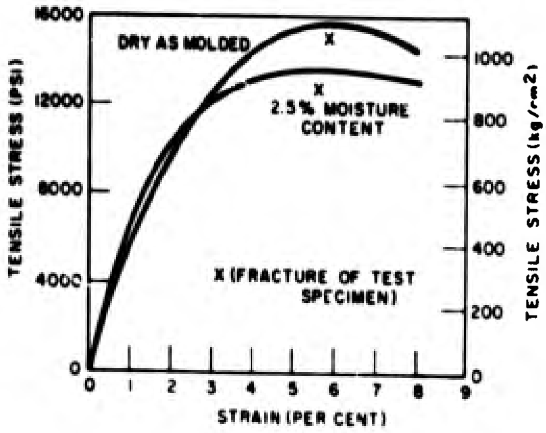


Figure 56a. Tensile stress versus strain of nylon copolymer (Zytel 109) at -68°F(31) (ASTM D-639)

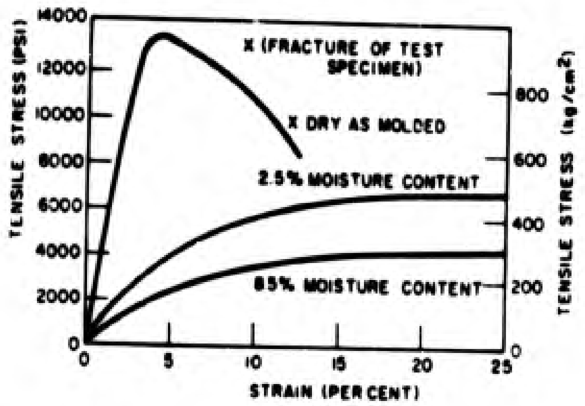


Figure 56b. Tensile stress versus strain of nylon copolymer (Zytel 109) at 73°F (31) (ASTM D-638)

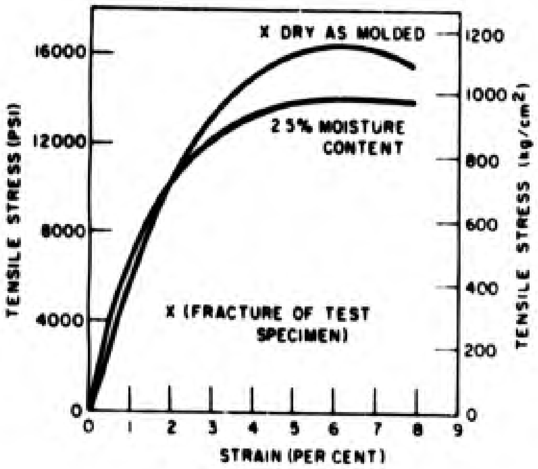


Figure 56c. Tensile stress versus strain of nylon 6/6 (Zytel 101) at -68°F (31) (ASTM D-638)

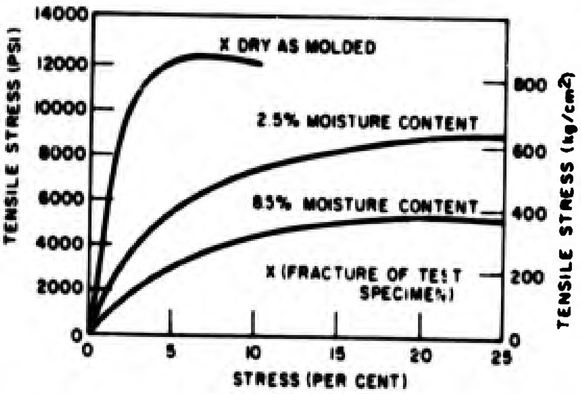


Figure 56d. Tensile stress versus strain of nylon 6/6 (Zytel 101) at 73°F (31) (ASTM D-638)

Data on the impact strength of nylon 6/6 and 6/10 are given in Table 20 and Figure 57.

TABLE 20. TENSILE IMPACT OF ZYTEL NYLON RESINS(31)

Property	ZYTEL				
	101(1)	101(1) WT07	31(2)	37(2)	42(1)
Tensile impact, ft lb sq in.					
Dry: at 73° F	82	74	79	103	155
at 0° F	93	80	73	90	105
at -40° F	85	88	68	94	107
Conditioned to 50% RH:					
at 73° F	156	164	121	135	300
at 0° F	113	90	73	96	118
at -40° F	79	71	86	88	110

(1) Nylon 66

(2) Nylon 610

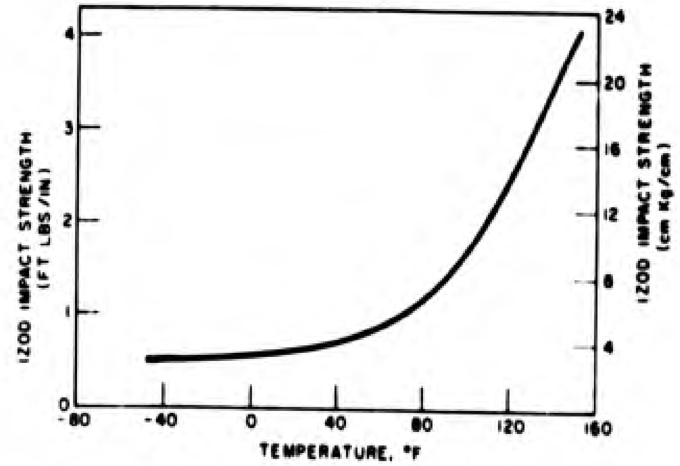


Figure 57. Effect of temperature of nylon 6/6 (Zytel 101) on Izod impact strength at 0.3 percent moisture content (31)

The mechanical properties of fiberglass reinforced nylon are listed in Table 21. Reinforcements are 30 to 40 percent by weight.

Tensile strength and rigidity improve with the reinforcement; percent elongation at break decreases. Unlike regular nylons, the impact strength of reinforced nylons improves from room temperature to -40° F. The improvement is attributed directly to the reinforcement. The best strength at both temperatures is obtained with the highest reinforced material, G-13/40.

TABLE 21. PROPERTIES OF (NYLAFIL) FIBERGLASS REINFORCED NYLON (53)

Property	°F	G-1/30 Type 6-6	G-2/30 Type 6-10	G-3/30 Type 6	G-10/40 Type 6-6	G-13/40 Type 6	G-1/30/(4) MS/5
Tensile Strength, Kpsi(1)	170 73 -40	18.5 20.0 21.0	16.2 19.0 20.0	16.0 21.0 23.0	23.4 30.0 36.6	25.0 30.0 39.0	18.0 20.0 21.8
Elongation, %(1)	170 73 -40	1.9 1.5 1.6	2.1 1.9 1.8	2.1 2.0 1.9	2.2 1.9 6.4	2.3 2.0 2.0	1.9 1.6 3.3
Flexural Modulus, Kpsi(2)	170 73 -40	740 985 1025	580 860 900	570 970 980	1400 1800 2000	900 1500 1900	790 1190 1260
Impact Strength, ft lb/in.(3) (1/2 x 1/4" bars)	73 -40	2.5 3.0	3.4 3.7	3.0 3.5	3.7 4.2	4.3 4.5	2.2 2.5

1. ASTM D 638
2. ASTM D 790
3. ASTM D 256
4. High lubricity grade with molybdenum-disulfide.

Figure 58 compares the impact strength of a 35 percent reinforced acrylic with 30 and 40 percent reinforced nylons. Again, the highest reinforced material is superior.

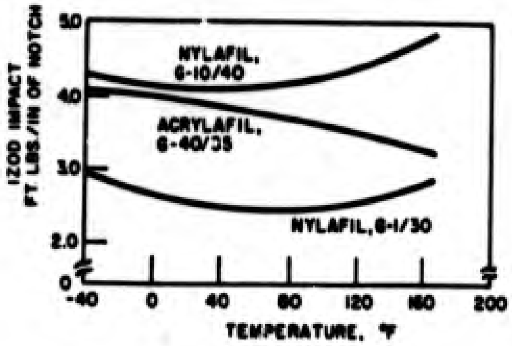


Figure 58. Izod impact strength of reinforced acrylic (Acrylafil and Nylafil) with variations in temperature (17)

Thermal Properties

The coefficient of linear expansion of two nylons 6/6 and 6/10 are given in Table 22 over the temperature range -40° F to 170° F.

TABLE 22. EFFECT OF TEMPERATURE ON LINEAR EXPANSION OF NYLON(31)

Property	°F	Zytel 101	Zytel 31
Coefficient of Linear Thermal Expansion (in./in./°F) x 10^{-5}	170	5	6
	73	4.5	5
	32	4	4.5
	-40	3.5	4

The reader is again reminded that the rates of thermal expansion of plastics are 5 to 10 times that for most metals. This difference must be allowed for in many design applications.

The brittleness temperature of unreinforced nylons (see Table 17 above) ranges from -85°F to -166°F depending on moisture content and resin formulation.

SECTION VIII. POLYCARBONATES

Mechanical Properties

The tensile and compressive strengths, the modulus of elasticity, and the torsional modulus for polycarbonate molding materials at various temperatures are given in Figures 59 - 62. The data at low temperatures reflect the normal trend for thermoplastics.

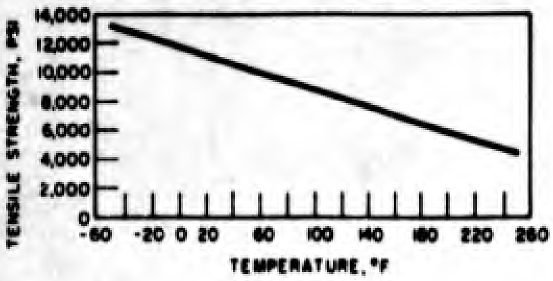


Figure 59. Tensile strength versus temperature of polycarbonate molding material (Merlon) (34)

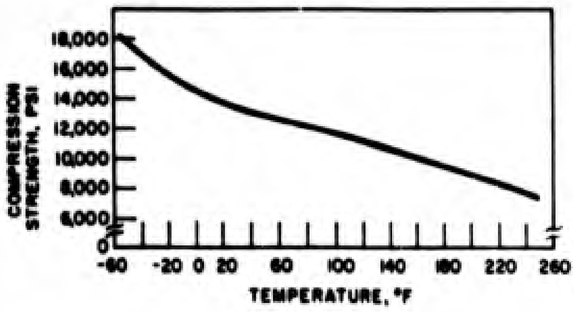


Figure 60. Compression strength versus temperature of polycarbonate (Merlon) (34)

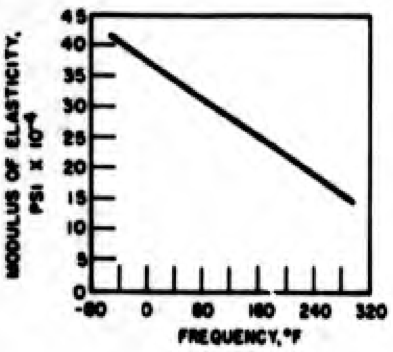


Figure 61. Effect of temperature on modulus of elasticity of polycarbonate resin(5)

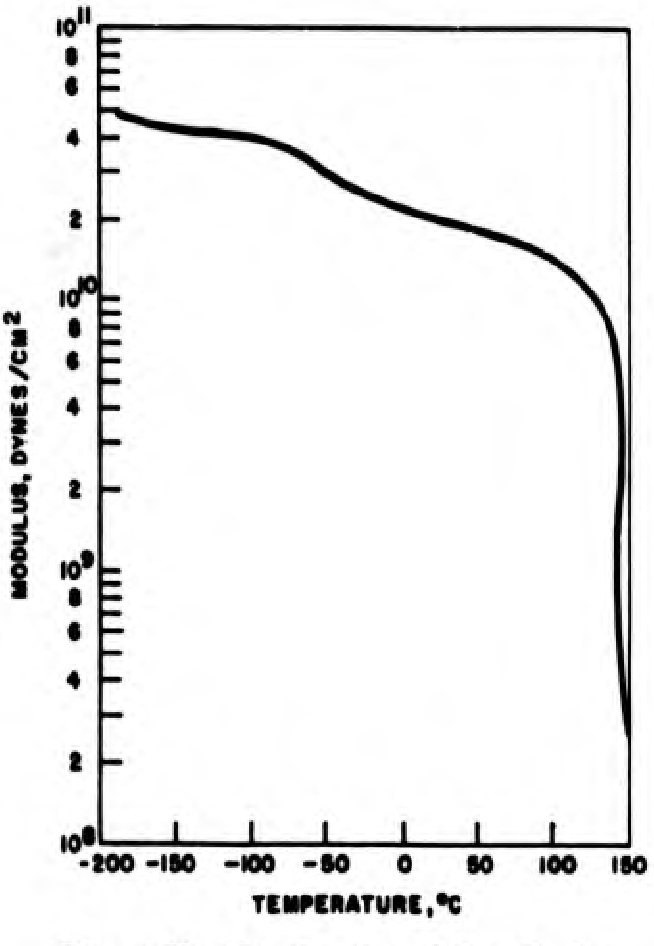


Figure 62. Torsional modulus of polycarbonate versus temperature (34)

Polycarbonates have very good impact strength over a wide temperature range. However, as can be seen in Figure 63, a tough-brittle transition of notched 1/8" thick specimens occurs at about -25°F. Even so, the impact strength at low temperatures is in the 2 to 3 ft/lb per inch of notch range, higher than many unreinforced thermoplastics of the same modulus.

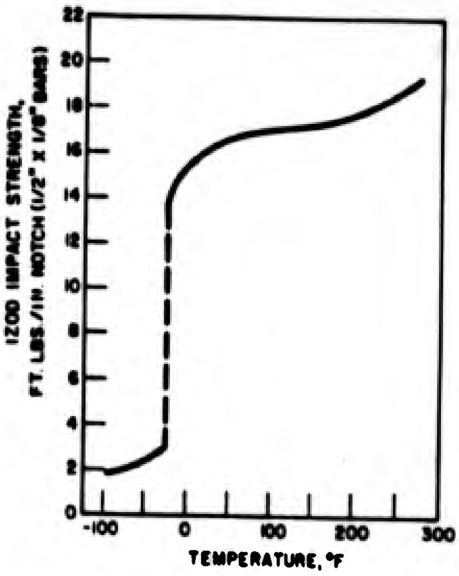


Figure 63. Izod impact strength versus temperature of (Merlon) polycarbonate (34)(ASTM D-256)

This transition in Izod strength is considered merely a change in response. In other words, it is a sudden and spectacular manifestation of a gradual change in properties and is not to be associated with fundamental transitions such as the glass transition or crystallization or phase change phenomena.(35) Vincent has explained the transition in terms of yield strength and brittle strength. The brittle strength is the stress which, if it could be applied, would cause the plastic to fail in a brittle manner.

"Both yield and brittle strength increase with decreasing temperature and increasing loading rate with yield strength increasing more rapidly. If the stress conditions of the test are such that the yield strength is less than the brittle strength, then yielding will occur and fracture will be ductile and tough. If the yield strength exceeds the brittle strength, then brittle fracture results before yielding can occur. A transition in the nature of the response is observed when changing test conditions (of loading rate or temperature) cause the yield strength to exceed the brittle strength." (35)

The impact strengths of polycarbonates are profoundly influenced by notch radius and specimen thickness. Impact strengths decrease with increasing thickness. For Izod notched impact tests the critical thickness is reported to be approximately between .13 and .17 inches at room temperature. (34) Below this thickness, ductile failure occurs; above this thickness brittle breaks occur. This means that a slight change in thickness results in a shift of the brittle fracture temperature from -25° F to above room temperature.

Electrical Properties

The electrical properties of polycarbonates are shown in Figures 64 to 68.

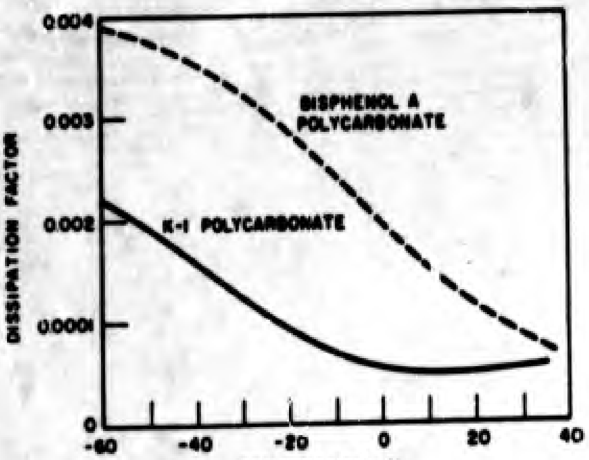


Figure 64. Dissipation factor of polycarbonates film at 1.0 kc

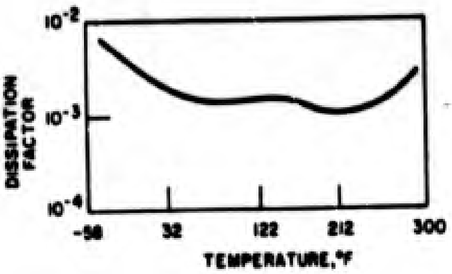


Figure 65. Dissipation factor of polycarbonate (Merlon) film versus temperature at 80 cycles/sec.

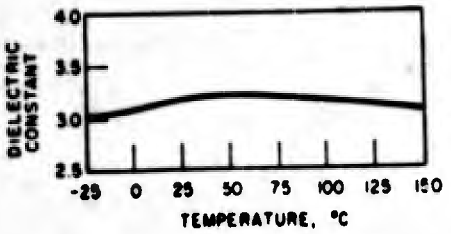


Figure 66. Dielectric constant of (Lexan) polycarbonate film versus temperature at 60 cycles(37)

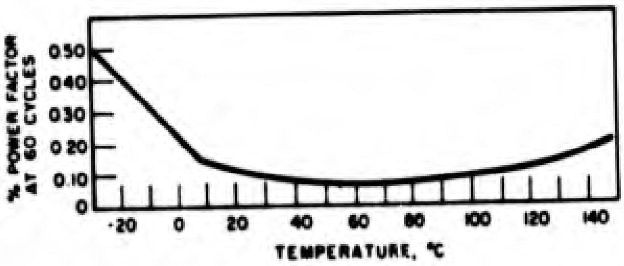


Figure 67. Power factor of (Lexan) polycarbonate film versus temperature at 60 cycles (37)

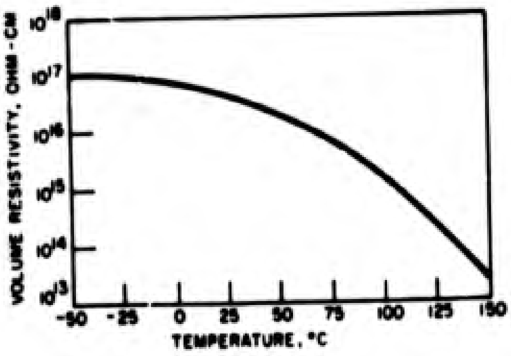


Figure 68. Effect of temperature on volume resistivity of (Lexan) film(37)

Thermal Properties

The coefficient of linear thermal expansion of polycarbonate molding compounds is reported to be 3.75 x 10^{-5} in/in/°F from -30 to +30C. (37)

Impact tests (ASTM D746) were conducted to determine the brittleness temperature of four compression molded disks cooled to -55°C. (4) Three specimens remained tough and one exhibited brittle behavior. Other specimens of the brittle failure material were tested to verify this finding and 60 percent of these failed at -55°C. Although brittle temperature of polycarbonates is reported to be -135°C, it is evident that some polymers resist shock loads better than others. No explanation was offered for the behavior of the sample which failed at -55°C.

SECTION IX. POLYETHYLENE AND POLYETHYLENE COPOLYMERS

Polyethylenes have a wide range of property values depending on their molecular structure - i.e., crystallinity, molecular weight and molecular weight distribution or any combination of these parameters. Density and melt index are methods used for indicating crystallinity and molecular weight: the higher the density the higher the percent crystallinity, the lower the melt index, the higher the molecular weight. Table 23 summarizes the effects of these parameters on the interested properties at low temperature.

Polyethylenes are normally divided into two or three groupings, depending on their densities: less than 0.940 g | cu. cm. is considered low and intermediate; over 0.940, high density. Typical nomenclature is given below:

ASTM Type	Common Designations	Density
I	Low-density, regular, conventional, high pressure	0.912 to 0.925 g/cu. cm.
II	Medium-density	0.925 to 0.940 g/cu. cm.
III	High-density, linear, low pressure	0.940 to 0.965 g/cu.cm.

Data on polyethylene copolymers is included where available. The high ethylene-low copolymer resins are similar to polyethylenes, while the high copolymer-low ethylene resins resemble gum rubber. The most prevalent copolymers are flexible ethylene vinyl acetate (EVA), ethylene acrylate (EEA) and ethylene-propylene; (See also Section XI for ethylene-propylene). Properties of these copolymers depend on percentage of comonomers, molecular weight and molecular weight distribution.

TABLE 23. EFFECTS OF MOLECULAR STRUCTURE ON LOW TEMPERATURE PROPERTIES OF POLYETHYLENES

Property	As Density Increased...	As Average Molecular Weight Increases...	As Molecular Weight Distribution is Narrowed...
Stiffness	Higher	Higher	Higher
Tensile Yield Strength	Higher	Higher	Higher
Tensile Ultimate Strength	Little Effect	Higher	Higher
Film Brittleness	Higher	Lower	Lower
Low Temperature Brittleness (ASTM D746)	Higher	Lower	Lower
Elongation %	Lower	Higher	——
Dielectric Constant	Higher	Little Effect	Little Effect
Dissipation Factor	Higher	Little Effect	Little Effect
Impact Strength			
Notched Unnotched	Higher * Lower *	Higher	Higher

* At temperatures below 18° F only.

Flexible ethylene copolymers are distinct exceptions to one fundamental relationship of ethylene homopolymers: with homopolymers stiffness increases with increasing density, and the stiffness modulus of any polyethylene can be approximated from the density value. This is definitely not true with ethylene alkyl-acrylates or ethylene vinyl acetate; in fact, as the density increases, stiffness decreases.

Cross-linked polyethylenes and copolymers, although not thermoplastic, are also included in this section. In general, cross-linking lowers the degree of polyethylene crystallinity.

Mechanical Properties

Figures 69 and 70 give the tensile properties of three types of polyethylenes at various temperatures. This data is on compression molded specimens which, for specification purposes, are preferred since the density can be accurately controlled and the effects of molecular orientation are minimized.

The mechanical properties of 20 and 40 percent fiberglass reinforced molding compounds are given in Table 24. Strength and modulus increases over the reinforced material. The increase in strength is the greatest with the highest quantity of reinforcement as indicated by the impact values.

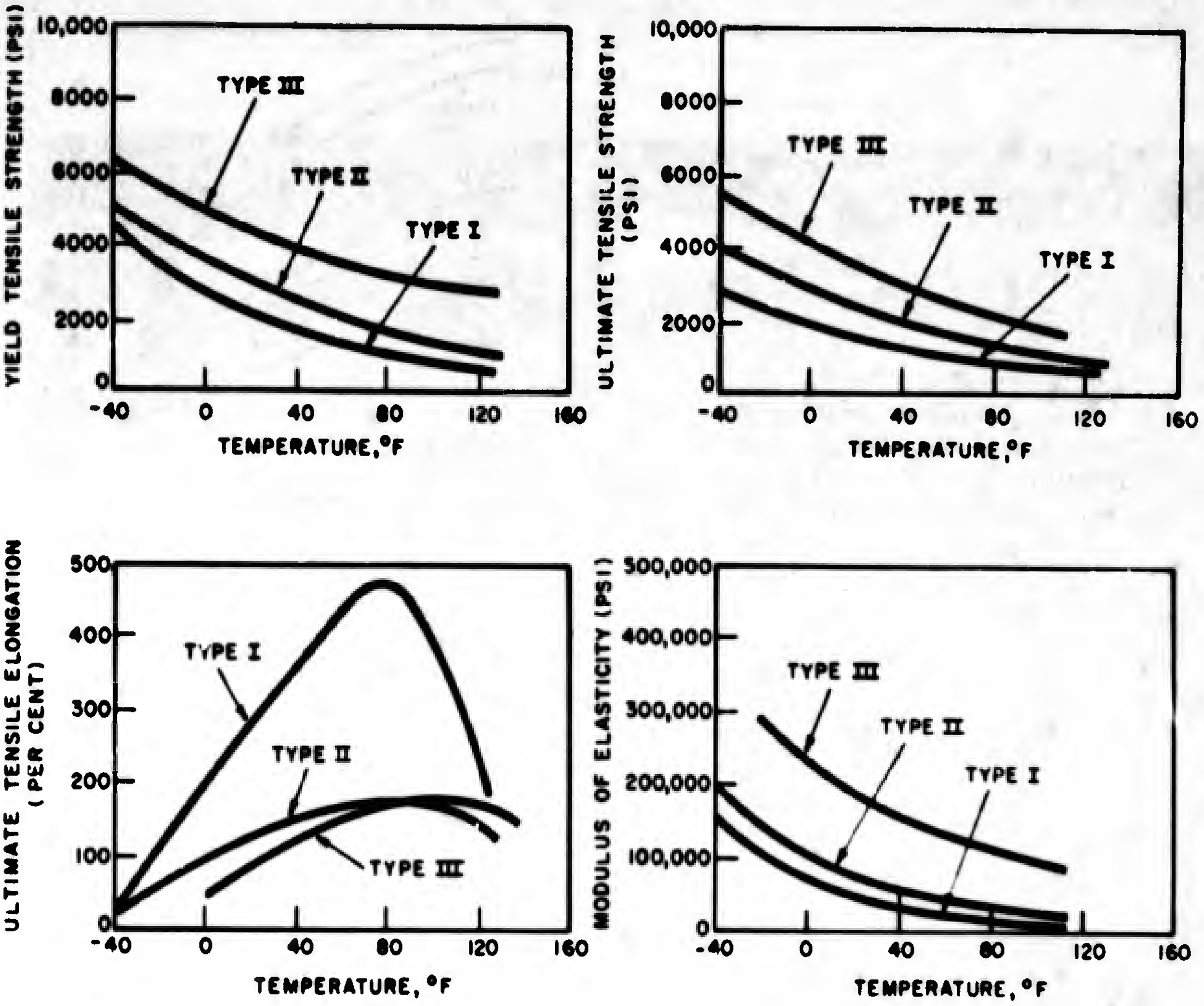


Figure 69. Strength of compression-molded polyethylene at various temperatures(38)

The tensile strength of polyethylenes is not as high as many other plastics. The tensile yield strength becomes greater than the ultimate breaking strength at the lower temperatures. In general, the tensile strengths and moduli increase with decreasing temperature and increased density; elongation decreases.

Tensile tests were conducted on low pressure (high density) polyethylene injected molded at 170°C along the longitudinal or working part of the dumbbell specimens.(40) The stress-strain curves are shown in Figure 71. As expected, there is an increase in the slope of the straight part of the elongation curve (elastic modulus in elongation) and in the height of the curves (tensile strength), a reduction in the deformation under tension, and a particularly sharp reduction in the breaking deformation with decreasing temperature.

1

Yield strength and elongations (by ASTM D-412) on compression moldings of polyethylenes having densities of 0.915, 0.930 and 0.960 (and melt indexes of 1.8, 2.1 and 0.5, respectively) at strain rates of 100 percent/min and 1000 percent/min.

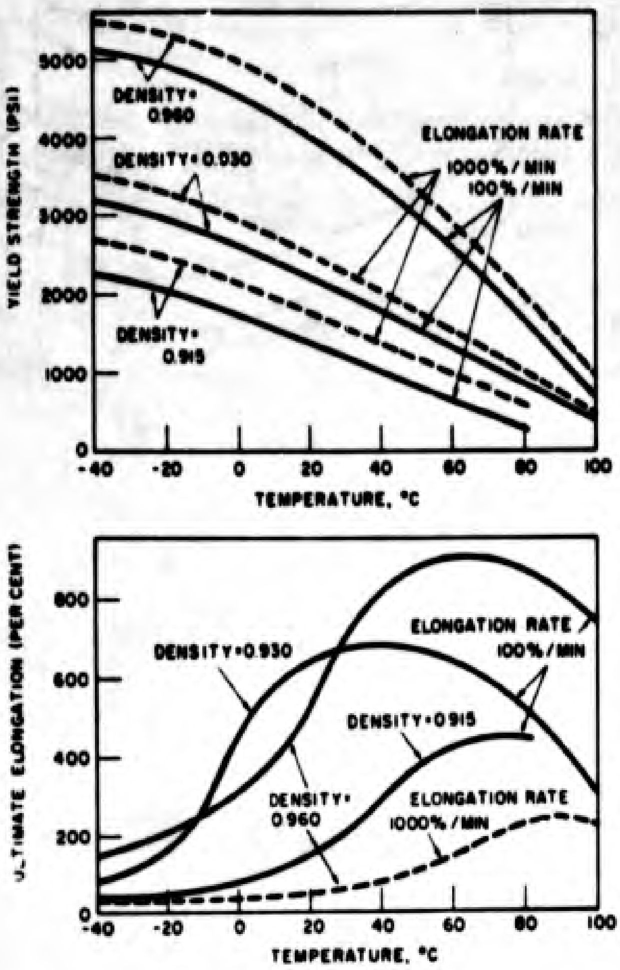


Figure 70. Tensile properties (ASTM D-412) versus temperature for polyethylenes (39)

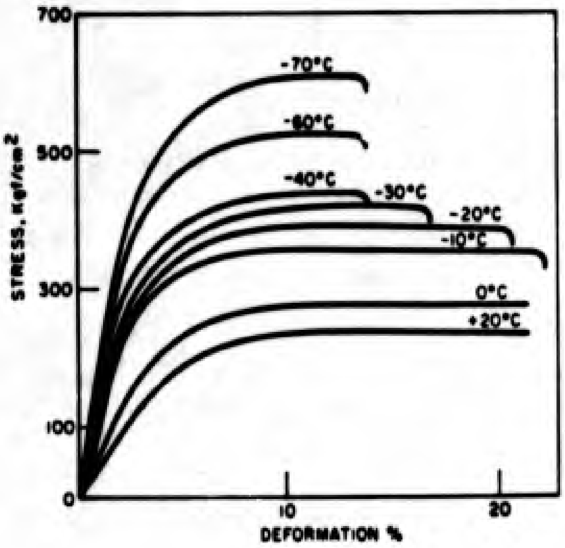


Figure 71. Stress-strain curves at various temperatures of low pressure polyethylene (40)

TABLE 24. MECHANICAL PROPERTIES OF FIBERGLASS REINFORCED POLYCARBONATE (POLYCARBAFIL) (17)

Property	°F	Polycarbafil	
		G-50/40	G-50/20
Tensile Strength, Kpsi(1)	170	18.0	—
	73	20.0	18.5
	-40	21.5	—
Elongation, %	170	0.7	—
	73	1.4	2.2
	-40	0.7	—
Modulus of Elasticity, Psi x 10^{-6} (2)	170	1.3	—
	73	1.7	1.2
	-40	1.85	—
Flexural Strength, Kpsi(2)	170	25.0	—
	73	27.0	25.0
	-40	32.0	—
Impact Strength, Izod, ft lb in. (3) (1/2 x 1/4" Bars)	73	4.0	3.0
	-40	4.0	3.0
Impact Strength, Unnotched (1/2 x 1/4" Bars)	73	12.0	9.0

(1) ASTM D 638 - 60T
(2) ASTM D 790 - 59T
(3) ASTM D 256 - 56

Figure 72 shows the tensile strength, elastic deformation and breaking elongation of injection molded, low-pressure polyethylene in both the longitudinal and transverse direction. As can be seen, the low-temperature resistance of an article can be improved if the direction of the maximum tensile force in service and the flow of the melt when the mold is filled are as close as possible. (40)

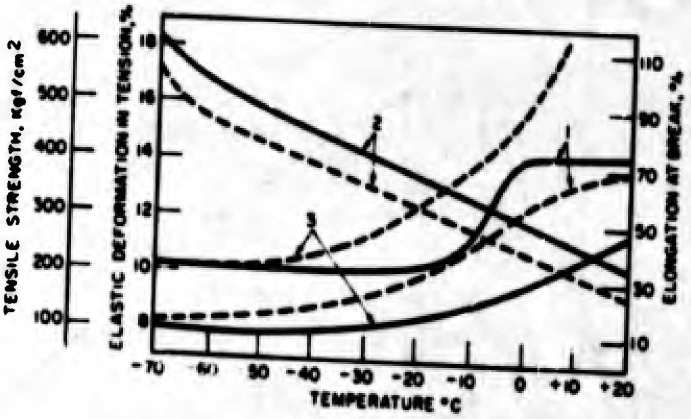


Figure 72. Dependence of the elastic deformation (1), tensile strength (2) and breaking elongation (3) of l.p. polyethylene on temperature. (Moulding temperature 170°C. ——— Longitudinal specimen;---------transverse specimen.) (40)

Figure 73 shows the tensile and yield strength of ethylene vinyl acetate copolymer (Montothene G50) at low temperatures. The decrease in elongation is plotted in Figure 74. Elongation at -50°C is about 200 percent.

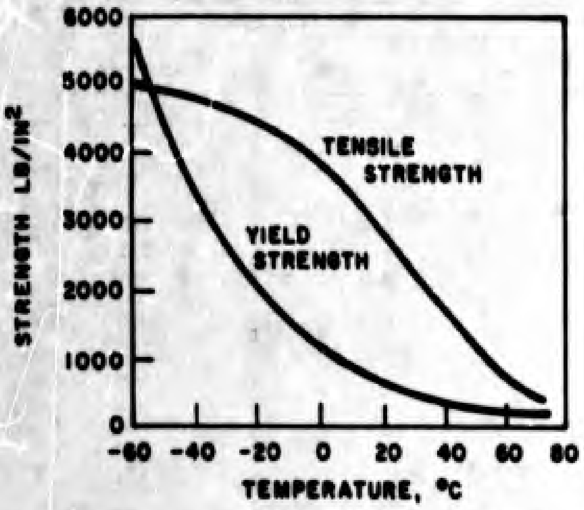


Figure 73. Variation of yield and tensile strengths of ethylene vinyl acetate copolymer (Montothene G50) with temperature (41)

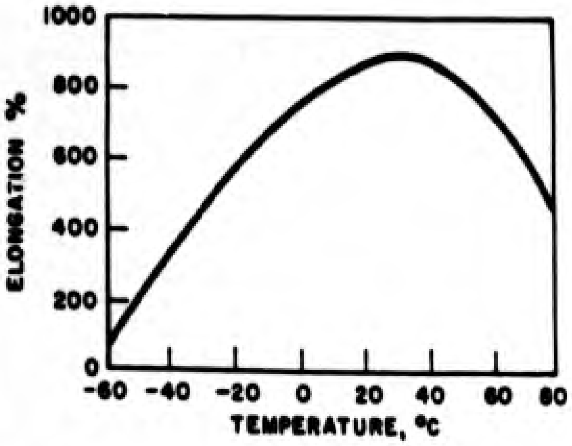


Figure 74. Elongation of the copolymer (Montothene G50) as a function of temperature (41)

Figure 75 shows stress-strain curves for a graft copolymer over a range of temperatures between -20°C to +100°C. Tensile strength at yield varies from a high value of 6300 psi at -20°C to approximately 1000 psi at +100°C. Also, elongation behavior is quite good at low temperatures.

The effect of low temperature on the flexibility and damping of different density polyethylenes are given in Figures 76 and 77.

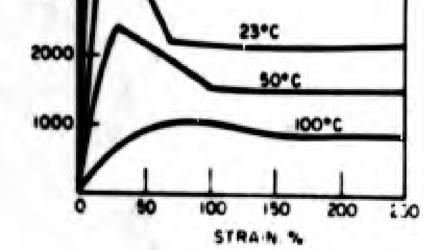


Figure 75. Stress-strain curves at various temperatures for QX-4262.6 high density polyethylene - 8 percent acrylic acid graft copolymer (42)

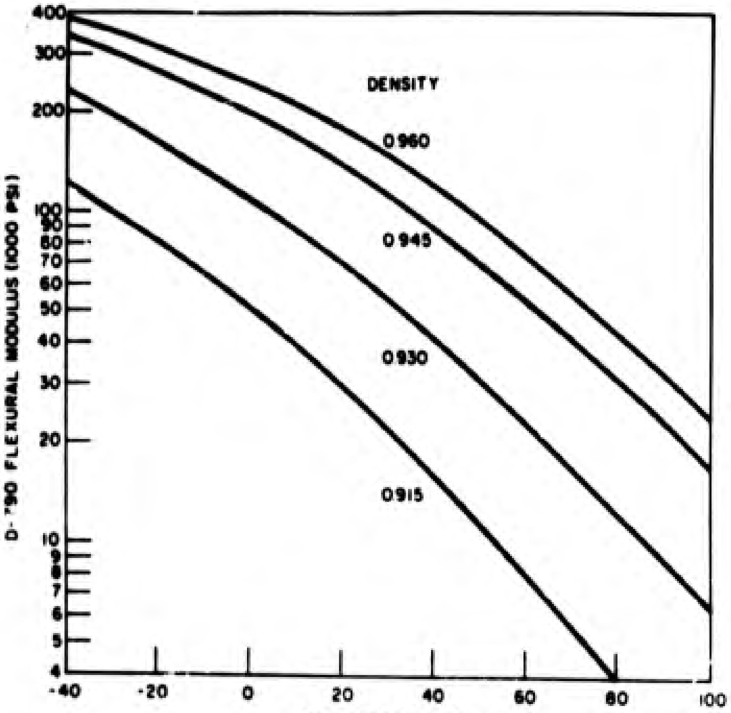


Figure 76. Effect of temperature on D-790 flexural modulus of polyethylenes of various densities (39)

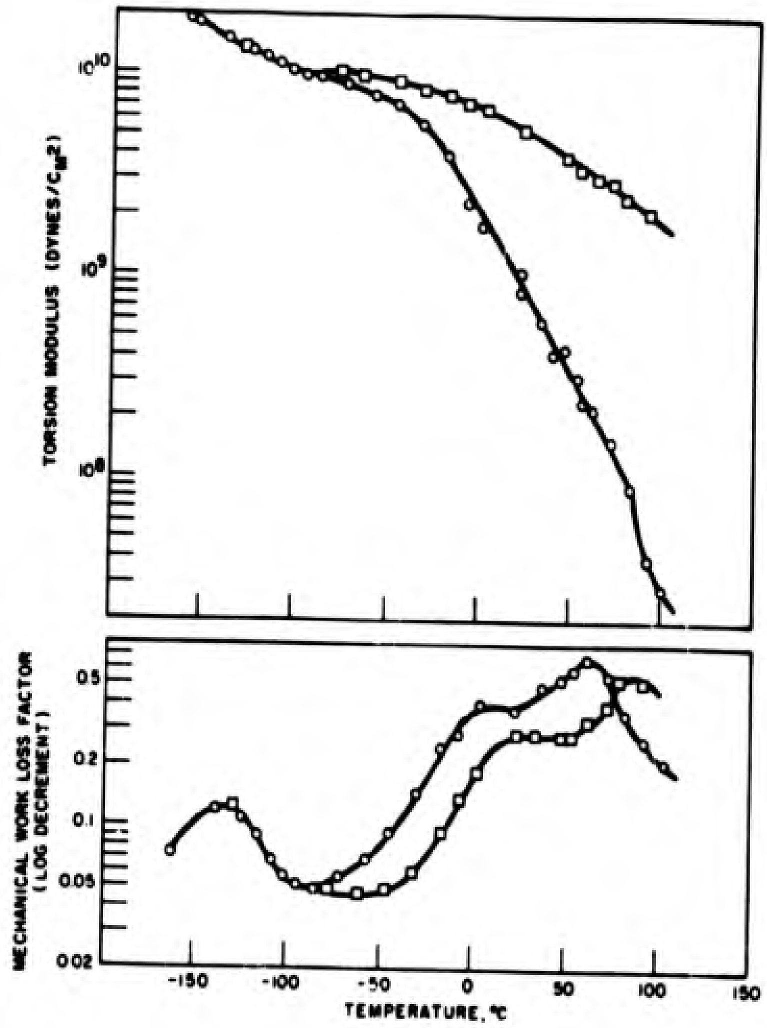


Figure 77. Dynamic mechanical properties of (Alathon 10) - (MI = 2.1; density = 0.923; point = ○) free radical polyethylene and (Alathon 7020) MI = 1.5; density = 0.954; point = □) linear polyethylene(39)

The relative twist moduli of polyethylenes over a range of temperatures, are compared to several thermoplastics in Figure 78. (43) The relative twist modulus refers to the ratio of the twist angle of a calibrated wire to the twist angle of the sample. The modulus of rigidity or torsion modulus was calculated from this relative twist modulus by the method given in the Gehman Low Temperature Flex Test D1053-61. The addition of a second protractor permitted operation with a total twist angle (sample + wire) of less than 180 degrees. The sample twist angles were kept to a maximum of about 20 degrees to minimize the creep effects from large deformations. Measurements were made 10 seconds after the total twist was applied to keep the creep consistent.

It was stated that the relative twist modulus must be multiplied by about 11,000 to convert it to psi. Then this value would be approximately 1/3 as large as the modulus of elasticity. The modulus coordinate uses a logarithm scale to cover a wide range of values which the modulus assumes as the plastic softens. A doubling of the stiffness occurs at about 3/4 of an inch on this scale. (43)

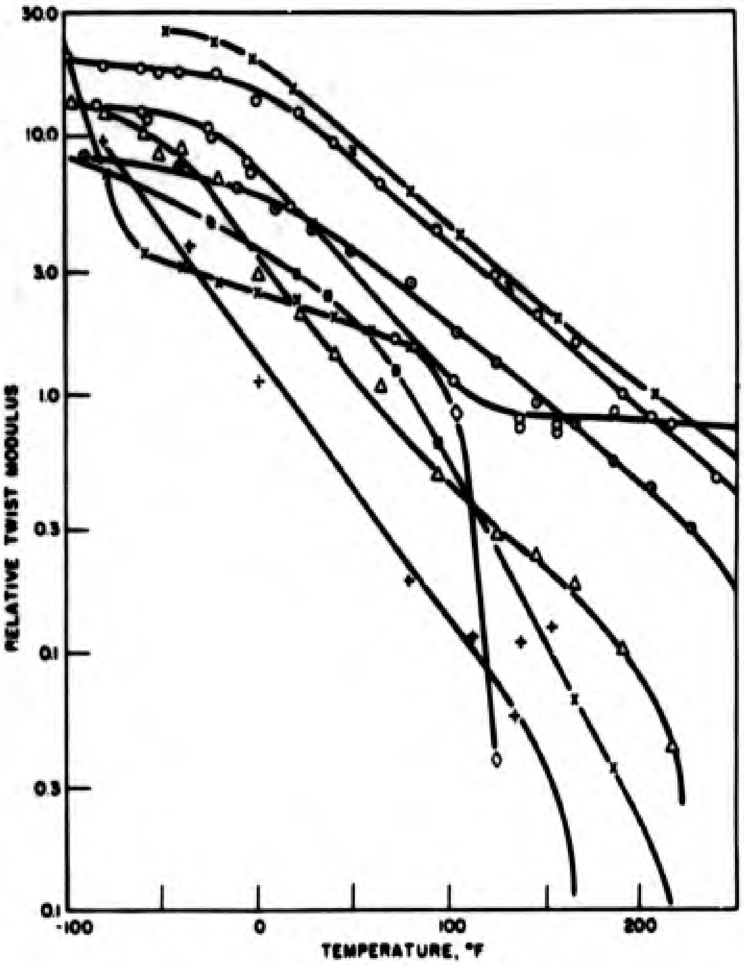


Figure 78. Modulus versus temperature characteristics of several "plastics" (43)

IDENTIFICATION OF MATERIALS

Material	Supplier and Identification		Molding Temperature (°F)	Molding Date
X Polypropylene	Eastman	Tenite 4251 A	439	12/11/61
O High Density Polyethylene	Phillips	Marlex 6002	384	1/10/62
Δ Low Density Polyethylene	Eastman	Tenite 810 A	373	1/5/62
+ Polyethylene Copolymer	Union Carbide	DPBE 6169	218	8/29/61
⊙ High Molecular Wt. Polyethylene	Hercules	Hi Fax 1906	455	1/16/62
✱ Ionomer	duPont	Surlyn A ER1552	326 307	3/23/65 10/29/65
8 Nylon 11	BCI (NY) (Current)	Rilsan Nylon 11	443	11/17/61
◊ Trans Polyisoprene	Acushnet	Purified Natural Balata	265	10/28/64

The data in Figure 78 show that the polyolefins harden gradually over a wide temperature range while the nylon 11 and the trans polyisoprene exhibit a "plateau" between the softening associated with the glass transition and that associated with melting crystallites.

At a relative twist modulus of about 10 or more the transition to a "rigid" material occurs and additional modulus increases are relatively minor with decreasing temperatures. This is clearly demonstrated by the data for the high density polyethylene and nylon 11 and it appears to be the general behavior of all polymers plotted.

Many plastics are rigid at room temperature and below, so flexibility measurements over this temperature range must be more precise than those measured by this technique if they are to be very informative.

A comparison of physical properties of ethylene copolymers; (namely, ethylene ethyl acrylate and ethylene vinyl acetate) with polyethylene and vinyl is given in Table 25. Again, it is pointed out that the copolymers differ from the ethylene homopolymers in that as the density of the copolymers increase, the stiffness modulus decreases. With homopolymers, stiffness increases with increasing density and the stiffness modulus can be approximated from the density value.

TABLE 25. COMPARATIVE PHYSICAL PROPERTIES OF POLYETHYLENE COPOLYMERS, POLYETHYLENE AND VINYL(44)

Property	°C	Copolymer			Vinyl
		EVA DQD-1868	Typical EEA	PE Low Density	90 Durometer
Tensile impact, (1) ft lb/cu in.	23	690	500	388	425
	-30	260	390	175	25
Brittleness Index, °C (2)					
20% failed	—	> -105	> -105	-95	-15
50% failed	-100	> -100	-100	-80	-15
Stiffness, Kpsi(3)	70	1.2	.40	3.0	.45
	60	1.7	.60	4.0	.60
	50	2.4	1.0	6.5	.90
	40	3.4	1.5	10.0	1.80
	25	5.1	4.5	18.0	4.5
	0	11.0	8.0	35.0	45.0
	-25	64.0	23.0	70.0	200.0
	-50	186.0	155.0	180.0	400.0

(1) ASTM D 1822
(2) ASTM D 746
(3) Secant modulus at 1% elongation (Instron)

The effect of temperature upon the stiffness of the copolymers is also shown in Figure 79 by plotting the stiffness modulus data measured on a tensile test apparatus at one percent elongation. The data reveal the similarities between the copolymers and polyethylene and the basic difference in behavior of the vinyl. The copolymer curves are very similar to the homopolymer (polyethylene) except for starting lower on the stiffness scale.

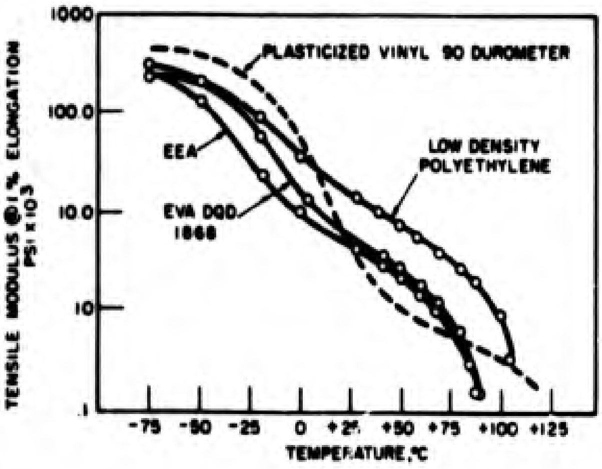


Figure 79. Stiffness modulus versus temperature of polyethylene, polyethylene copolymers and vinyl (44)

In the intermediate temperature range, both the homopolymers and the copolymers show a more gradual response to increasing temperature than the vinyl. The sharp change in stiffness of the vinyl at these temperatures is attributed to a higher glass transition temperature than the copolymers or polyethylene. This figure shows the advantage of the copolymer materials where low temperature, flexibility and dimensional stability are required.

The copolymers' higher polarity and initial low modulus also permit them to be highly filled with inorganic fillers without embrittlement or excessive loss of physical properties. Mixing conventional polyethylenes with inorganic fillers usually results in increased rigidity and lower elongation. Large quantities of carbon black, clays, calcium carbonate, asbestos, and similar materials can be mixed with EVA and EEA resins with little effects.

Flexible copolymers reveal the following characteristics:

- Flexibility over a wide temperature range, including good low temperature flexibility (better than comparable vinyls having the same room temperature stiffness modulus).
- Flexibility without plasticization and their related problems, such as migration.
- Excellent impact strength even at low temperatures.

The modulus of rigidity versus temperature of ethylene vinyl acetate copolymer (Monsanto's Montothene G50) and a plasticized PVC is shown in Figure 80. As can be seen, the copolymer shows a fairly gradual, almost linear increase of rigidity from +80°C to -50°C. In the interested temperature range -17° to -62°C the copolymer is more flexible than the plasticized PVC.

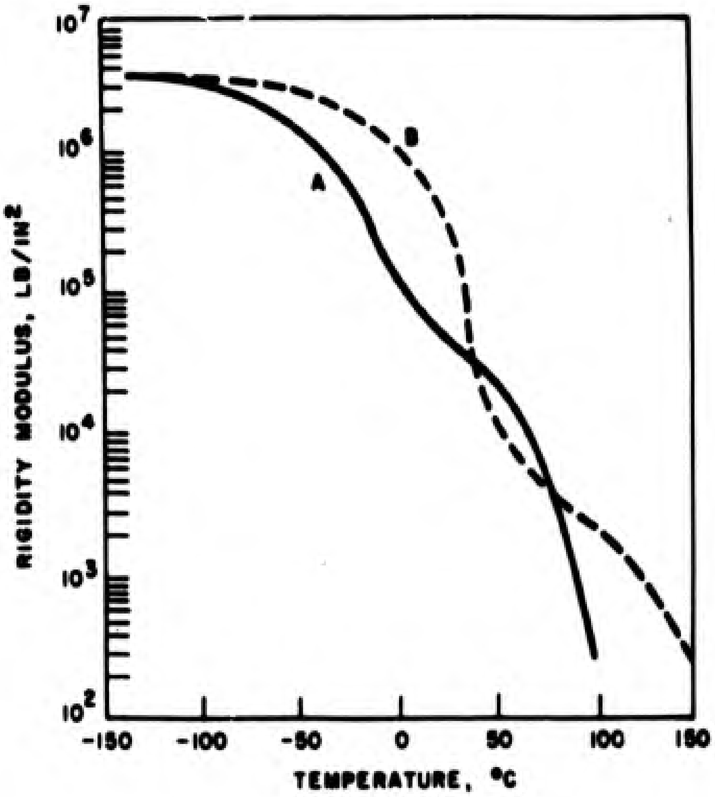


Figure 80. Modulus of rigidity versus temperature for A, ethylene vinyl acetate copolymer (Montothene G50); and B, plasticized PVC to 74 parts of suspension polymer (K-value 70) and 26 parts dioctyl phthalate (41)

Figure 81 compares the secant modulus of a high density polyethylene with an ethylene acrylate copolymer at low temperature.

The effect of cross-linking on modulus is shown in Figures 82 and 83. These crystalline products do not show a very sharp change in modulus in the interested temperature range. A gradual increase in stiffness is observed paralleling that of the uncross-linked parent down to -80°C. This behavior dominates even the relatively highly cross-linked products where 3 percent peroxide and 10 percent triallyl cyanurate were used. (46)

The impact strength of polyethylenes is affected by molecular weight and molecular weight distribution in addition to the usual factors previously discussed for thermoplastics (thickness, notch sensitivity and density or crystallinity). Impact strength increases as molecular weight increases and molecular weight distribution narrows, the former being more dominant. This is illustrated in Table 26 which presents Izod impact data on du Pont polyethylenes of various melt indexes (molecular weights), densities and molecular weight distributions at two temperatures.

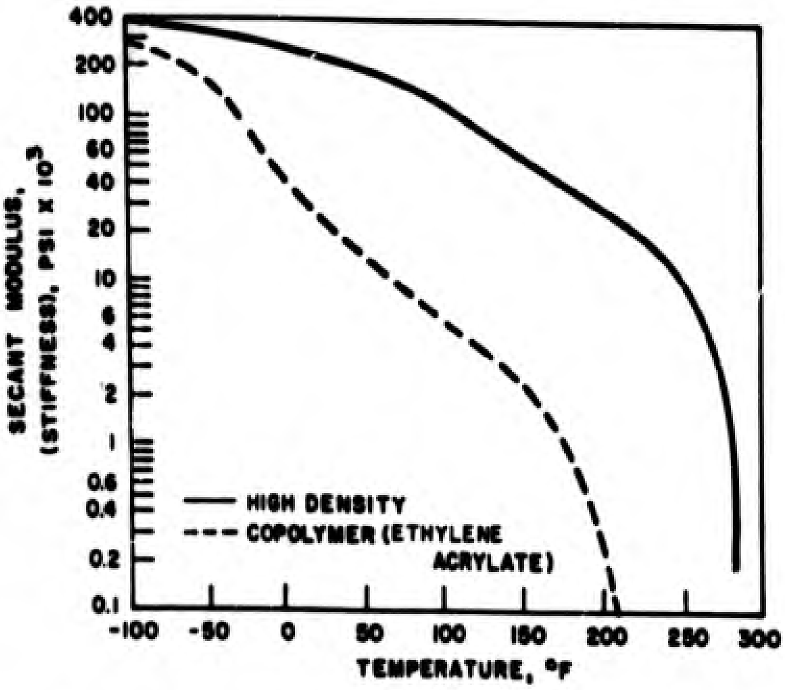


Figure 81. Modulus of polyethylene and ethylene acrylate copolymer as a function of temperature (45)

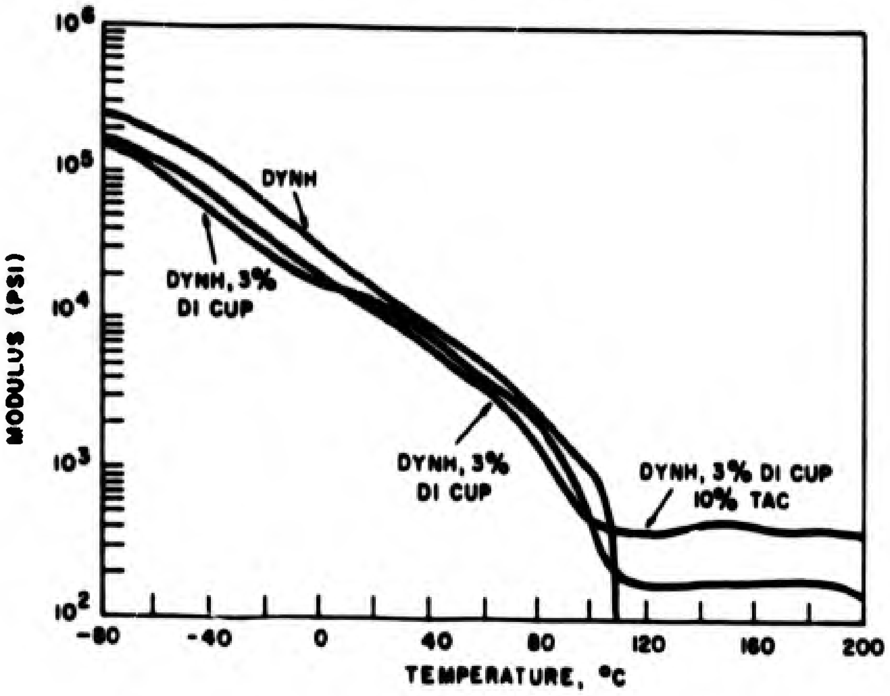
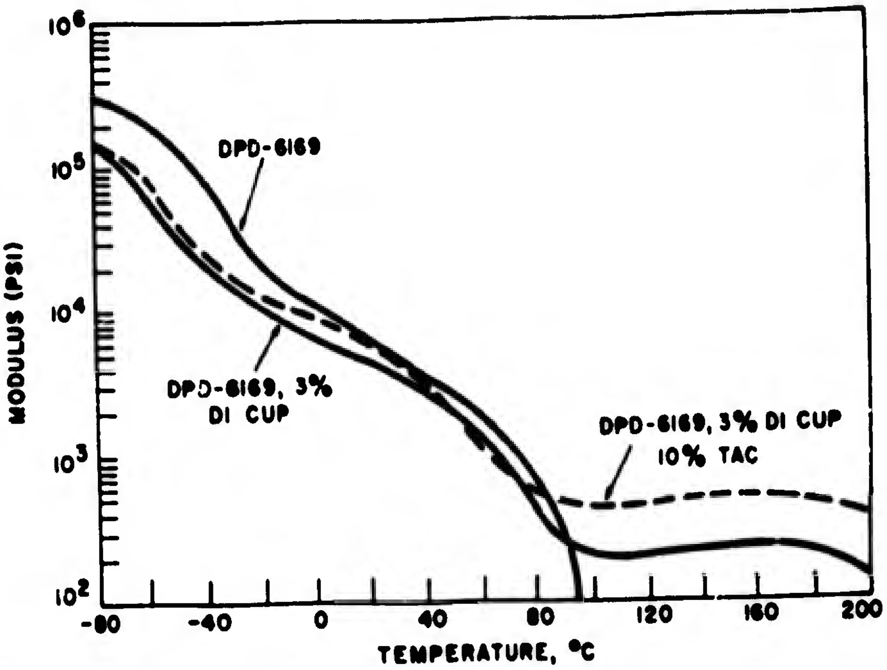


Figure 82. Modulus versus temperature - unfilled cross-linked low density PE (46)

DYNH - Cross-linked .919 density polyethylene
DI CUP - Dicumyl peroxide
TAC - Triallyl cyanurate



DPD-Ethylene ethyl acrylate copolymer
DI CUP-Dicumyl perioxide
TAC-Triallyl cyanurate

Figure 83. Modulus versus temperature - unfilled cross-linked acrylate copolymer DPD-6169 (46)

TABLE 26. NOTCHED IZOD IMPACT STRENGTH OF POLYETHYLENES (39)

Melt Index (g/10 min)	Density (G/ml)	Molecular Weight Distribution	Notched Izod Impact Strength (ft lb/in.)	
			-40°C	23°C
Free Radical Polyethylenes:				
1.3	.914	broad	0.7	>10 (NB)
2.1	.923	broad	0.8	>10 (NB)
0.5	.936	broad	0.95	>10 (NB)
Linear Polyethylenes:				
0.2	0.953	narrow	8	>10 (NB)
0.5	0.954	narrow	2.1	>10 (NB)
2.0	0.956	narrow	1.1	>10 (NB)
5.0	0.959	narrow	0.8	>10 (NB)
0.2	0.945	broad	1.4	1.9
2.0	0.962	broad	0.6	0.7

1 ASTM D-256-56

Figure 84 plots the impact strengths of low density, high density, and high melt index (low molecular weight) polyethylenes over a wide temperature range. It is interesting to note that in the notched Izod impact test the high and low density polyethylenes react differently. Between 0 and 20° F low density polyethylene goes through a tough-brittle transition where impact strength goes from less than one ft. lb./in. of notch to 18 ft. lbs./in. notch. The impact-strength of the high density polyethylene increases gradually with increase in temperature. As can be seen in Figure 84, the high density material is superior to the low density below 18° F; above 18° F the reverse is true.

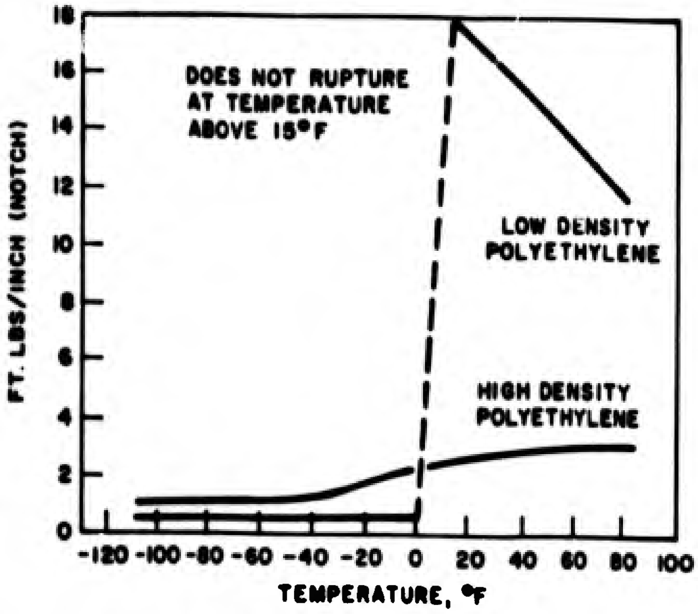


Figure 84. Effect of temperature on Izod impact strengths of (Marlex) polyethylene (47)

Data on unnotched specimens is quite different as shown in Figure 85. There is no transition point for low density polyethylene and little difference exists between the different density specimens. Also, unlike many thermoplastics, the impact strength of unnotched polyethylenes increases with decreasing temperature.

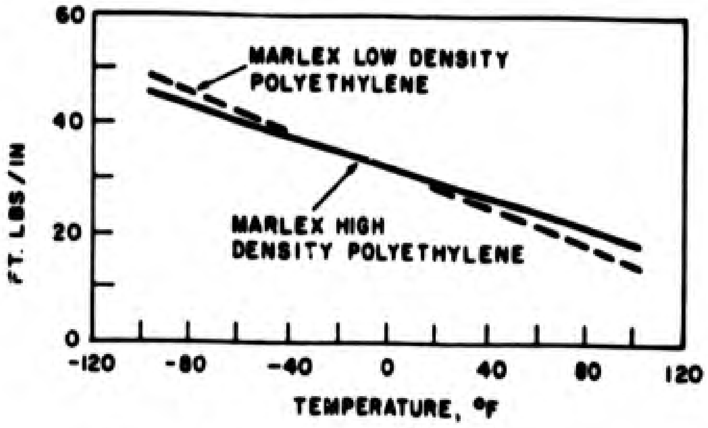


Figure 85. Effect of temperature on unnotched Izod impact strengths of (Marlex) polyethylene (47)

Tensile impact data for various high and low density polyethylene blends with flexible ethylene acrylate copolymers is plotted in Figures 86 and 87. At low temperature, the higher percent of low-density copolymer blends are superior. As previously stated, one of the outstanding properties of polyethylene copolymers is this improved impact strength. As an example, drop tests were made with a 5 lb. rod on a high density homopolymer and a 50/50 flexible ethylene acrylate copolymer blend injection molded dishpan. At room temperature the high density material withstood the rod dropping from a height of 2 ft., while a 50/50 blend did not fail within the limits of the test (a drop of 5 ft.). At low temperatures (-4° F) high density polyethylene withstood a drop of 1.5 ft., the 50/50 blend withstood a drop of 4 ft. At this same temperature, low density polyethylene homopolymer withstands a drop of 2.5 ft. while the 50/50 blend does not fail within the test limits.(45)

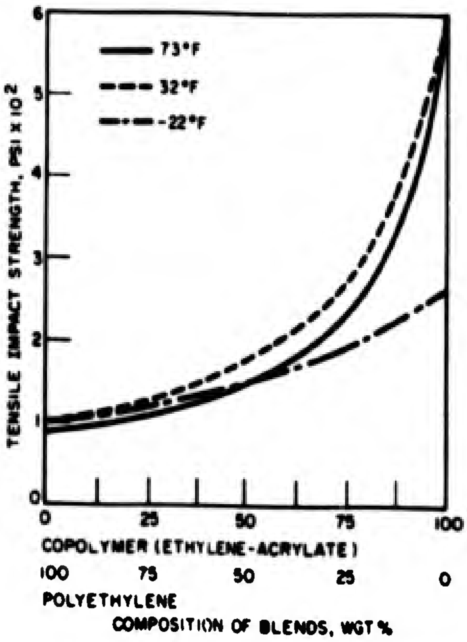


Figure 86. Tensile impact of copolymer-high density blends (45)

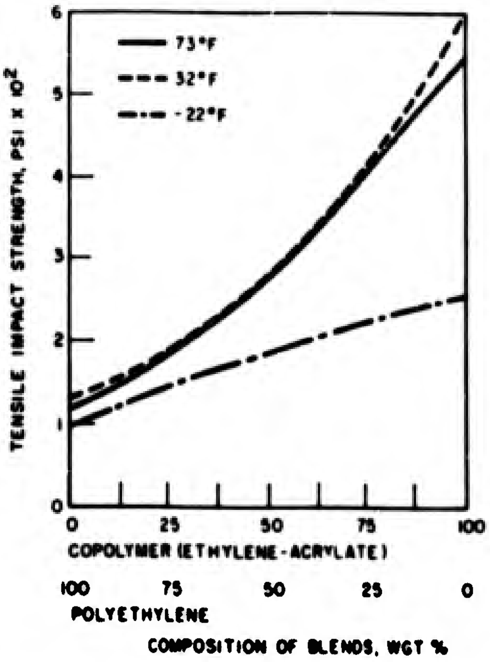


Figure 87. Tensile impact of copolymer-low density blends (45)

General Mills studied the effect of low temperature on polyethylene films for high altitude balloon applications. Two critical properties for balloon film are toughness and low gas permeability or porosity over a wide range of temperatures.(48)

Toughness was determined by measuring the cold brittleness temperature (the temperature at which the tear caused by a steel ball piercing the film changes from straight-lined tear to random shattering) and puncture resistance. The latter was determined by measuring the velocity of a steel ball dropped from a fixed height after breaking through a test sample by use of two photoelectric cells and a Berkley Time Interval Meter. The difference in velocity between free fall of the ball and after puncturing the film is considered to be the energy absorbed, although the relationship between velocity and energy absorbed is not strictly linear. (Table 27)

The authors report that the molecular weight of polyethylene resin had a decided effect on the brittleness and puncture resistance characteristics of the film. Although some exceptions were found, generally the higher molecular weight resulted in the lowest brittleness temperature and best puncture resistance.

TABLE 27. COLD BRITTLENESS TEMPERATURE AND PUNCTURE RESISTANCE OF POLYETHYLENE FILM(48)

Polyethylene Manufacturer's Grade	Melt Index Value	Film Thickness (inches)	Cold Brittleness Temperature (°C.)	Puncture Resistance (in. lbs./mil.) -20°C.)
A	0.41	0.002	-72	16
B	0.83	0.0015	-70	17
	0.98	0.001	-66	11
C	1.37	0.001	-67	10
	2.17	0.0015	-60	9
D	2.21	0.0015	-52	10
E	2.93	0.0015	-62	10

The deviations from perfect correlation are attributed to other factors besides molecular weight which influence low temperature toughness. Also, orientation seems an important factor in puncture strength but does not appear to influence cold brittleness temperature to any marked degree.(48)

Blends of polyethylene and polyisobutylene film were found unsuitable since small quantities of polyisobutylene raised the brittleness temperature. (See Table 28). However, they did improve the puncture resistance at low temperatures. Some other plastics film reportedly good at low temperature are listed in Table 29.

TABLE 28. LOW-TEMPERATURE TOUGHNESS OF POLYETHYLENE-POLYISOBUTYLENE BLENDS (48)

Per Cent Polyisobutylene	Cold Brittleness Temperature	Falling Ball Puncture Resistance at -20°C. (in. lbs./mil.)
0	-71°C.	19
3	-63°C.	21
15	-57°C.	26
30	-46°C	29
50	-40°C	23

TABLE 29. LOW-TEMPERATURE TOUGHNESS OF MISCELLANEOUS PLASTIC FILMS (48)

Film	Thickness	Cold Brittleness Temp. °C	Puncture Resistance (in. lbs./mil. -30°C.)
Vinyl Chloride Copolymer (plasticized)	0.002	0 to -15	5
Polychlorotrifluorethylene (unplasticized)	0.002	+30	4
Polychlorotrifluorethylene (plasticized)	0.002	-71	16
Polyethylene Terepthalate (2-way oriented)	0.0005	-73 to -75	10
Polytetrafluoroethylene	0.001	below -74	13

Permeability tests were conducted as follows: The film sample was clamped between two steel flanges. The flanges were held together by means of a screw press. The film was supported on each side by porous stainless steel discs which were inserted in the face of each steel flange to support the film and act as diffusers for the gas. A manometer or pressure gauge was used to measure the pressure difference, and the temperature of the surrounding air was measured on a thermometer. The pressure on the inlet side was held constant by a gas pressure regulator. After dynamic equilibrium was reached, the rate of gas flow was determined by observing the rate at which a column of mercury traveled through a 1 mm calibrated 30" long glass capillary. A vibrator was used on the glass capillary to prevent sticking.

Figure 88 plots the permeability versus temperature of polyethylene film for various gases. The permeability for helium at room temperature was 1,000 times that at -70°C. This decrease in permeability with decreasing temperature is so substantial that the property is unimportant. In addition, the authors state that neither molecular weight or film orientation appear to affect the permeability of polyethylene films to any significant degree. However, film thickness is very important.

True porosity of any given film is very difficult to evaluate due to lack of uniformity over the film's surface. Also, handling greatly increases the number and size of holes and affects porosity. This is especially serious at low temperature where flexing causes pinholes.

Unlike permeability, porosity varies depending on hole size, shape and absolute pressure. Temperature has no affect on gas loss, only on pinholes. Hole sizes 0.1 mm and greater are considered porosity whereas under .1 mm they are in the permeability range. (48) Porosity then is more serious at low temperatures than permeability which normally decreases with temperature.

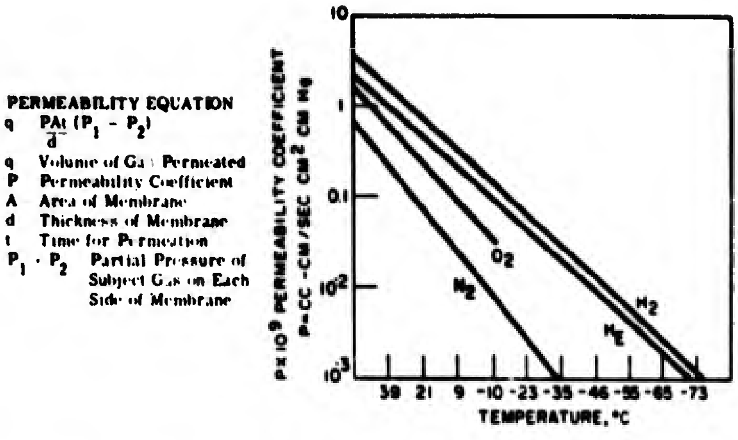


Figure 88. Permeability for polyethylene film(48)

Electrical Properties

Polyethylenes have good electrical insulating properties. The power factor and dielectric constants are low over a wide range of temperatures and frequencies. Data are given in figures 89 and 90.

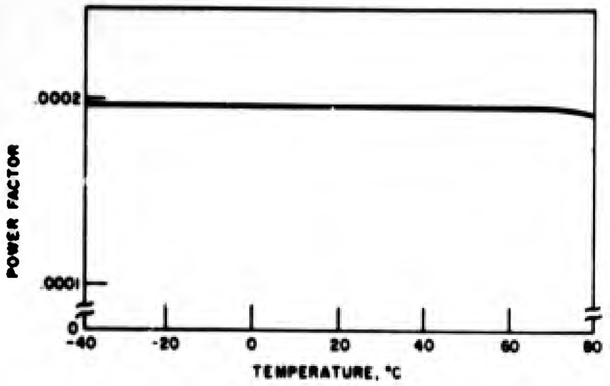


Figure 89. Effect of temperature on power factor for ("Alathon" 5, NC-10, 0.923 density) polyethylene (49)

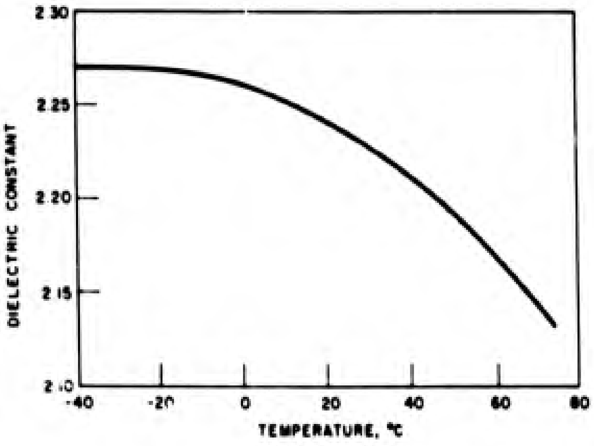


Figure 90. Effect of temperature on dielectric constant for ("Alathon" 5, NC-10, 0.923 density) polyethylene (49)

Figure 91 shows the dissipation factor of different polyethylenes over a broad temperature range. Despite the significance of the absorption peaks, the maximum values of the dissipation factor shown in Figure 91 (in our temperature range) are generally too small to be of direct engineering significance, except possibly at the highest frequencies. At very high frequencies, however, even a small increase in dissipation factor is considered important.(29)

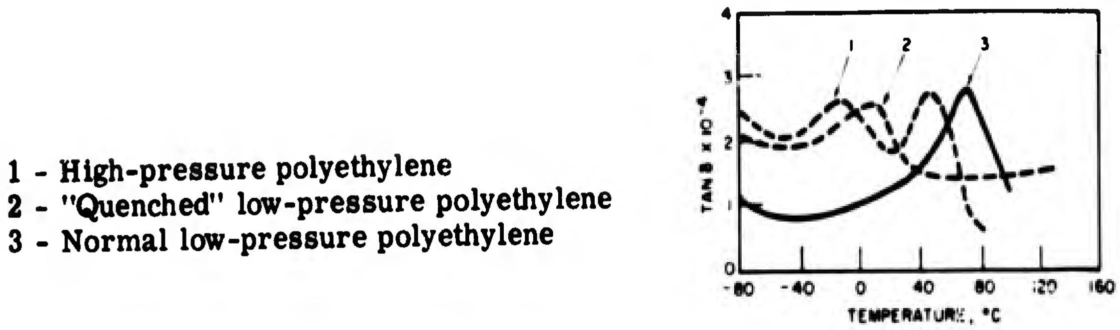


Figure 91. Dissipation factor versus temperature at 1000 cps for different types of polyethylene(29)

A contour map indicating the dissipation factor at low temperature for various frequencies for high density polyethylene is given in Figure 92.

The electric strength of polyethylene versus temperature and frequencies is plotted in Figure 93.

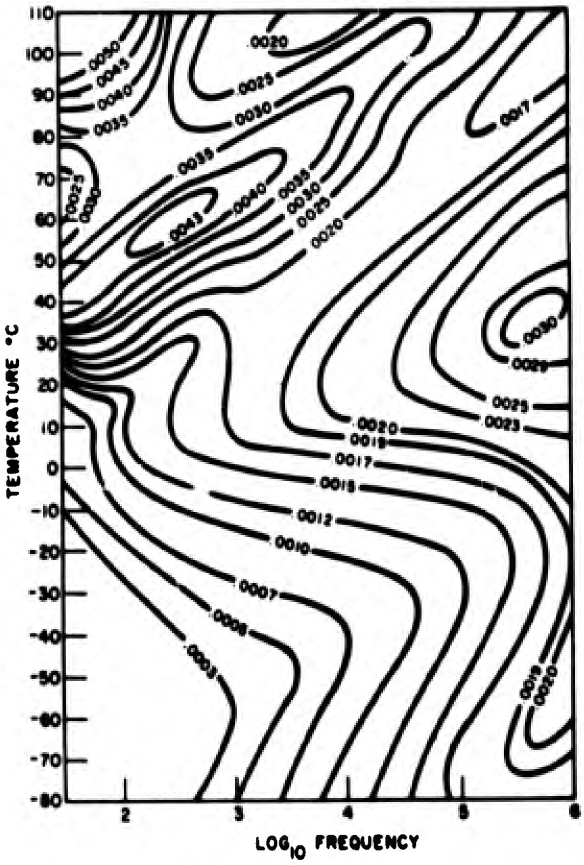


Figure 92. Tan δ contour map for high-density polyethylene (29)

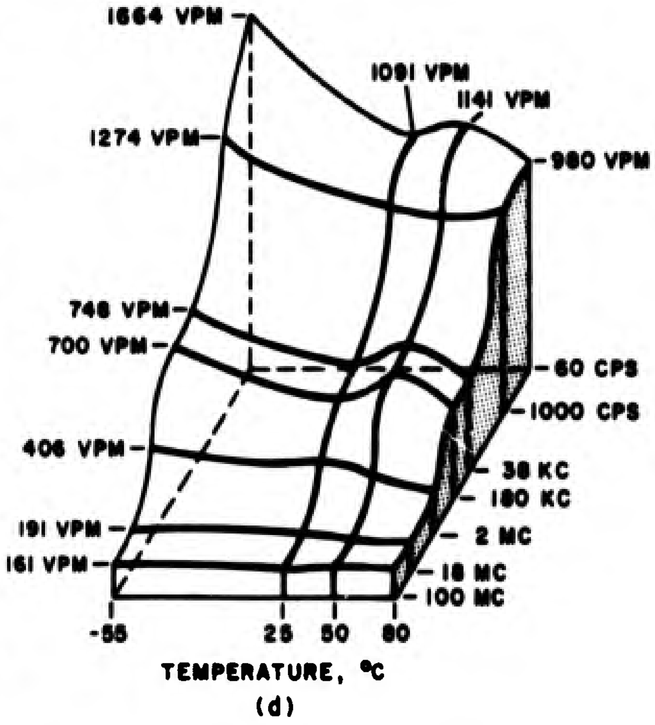


Figure 93. Electric strength of polyethylene (29)

Thermal Properties

The linear coefficient of expansion of several polyethylenes, a 50/50 copolymer of polypropylene and polyethylene-propylene blends, was determined. (50) The test procedure utilized a linear variable differential transformer. A least-square numerical differentiation procedure based on moving arcs was used to yield the coefficients of thermal expansion as a function of temperature. The results are shown in Figures 94 - 98.

The data above 0° C were erratic and inconsistent with the adjoining region. The authors were unsuccessful in eliminating these erratic results and therefore discounted this data. However, the literature reports the coefficient of linear expansion at room temperature to be approximately 9.5 x 10^{-5} for type I; 8 x 10^{-5} for type II; and 7 x 10^{-5} for type III. (51) This is higher than many thermoplastics.

The three polyethylene samples tested appear to have three transitions in our interested temperature range: two between 0 and -40° C and one about -62° to -67° C. Transitions in the 0 to -40° C range were attributed to branching. Bohn found that the transition temperature in polyethylene around 0° C was lowered as branching increased (crystallinity decreased). Similar results were reported by Tanaka and Kline et al. (50)

The lower transitions for samples L and M compared to J implies that they are more branched and therefore less crystalline than the 54 percent reported for sample J.

The 50/50 propylene-ethylene copolymer gave erratic results above -40° C but showed a major transition at -61° C. The authors stated that Kontos and Slichter, using dilatometry, reported a T_g value of -58° C for this composition, and Manaresi and Giannella's results on other copolymer compositions predict a value of -53° C. The erratic results obtained above -40° C were attributed to trapped stresses in the sample.

A major transition occurs in polyethylene-polypropylene blends in the -9 to -14° C portion of our interested temperature range. Also, the data for the 88 percent polyethylene are similar to 100 percent polyethylene suggesting transitions near -30 and -60° C. The magnitude of the change in the linear expansion coefficient at the transition decreases with increasing polyethylene content.

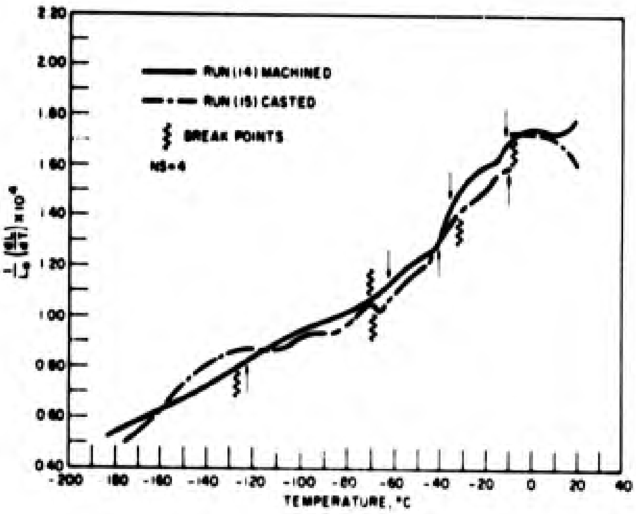


Figure 94. Linear expansion coefficient as a function of temperature for polyethylene samples L and M (50)

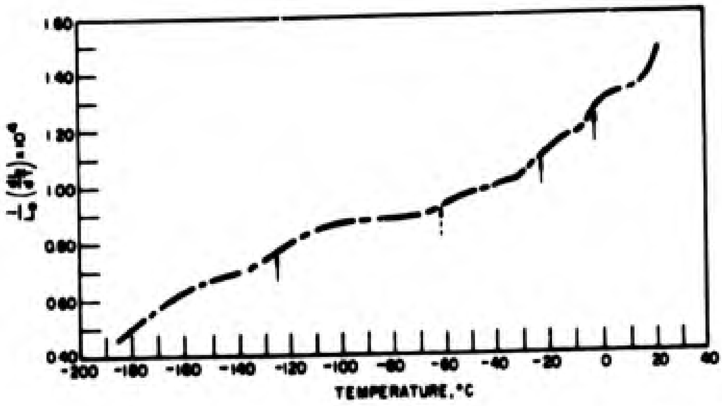


Figure 95. Linear expansion coefficient as a function of temperature for polyethylene, sample J (54 percent crystallinity, 0.929 density (50)

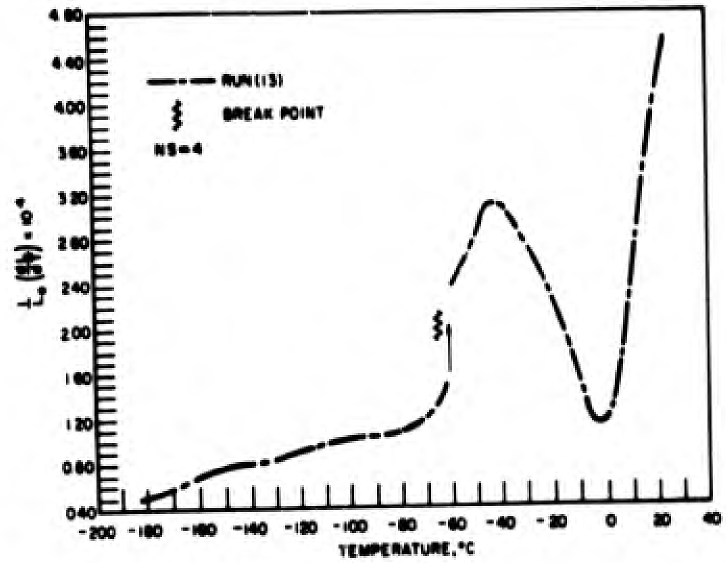


Figure 96. Linear expansion coefficient as a function of temperature for polyethylene (50 mole - percent) - polypropylene copolymer (non crystalline) density - 0.854 (50)

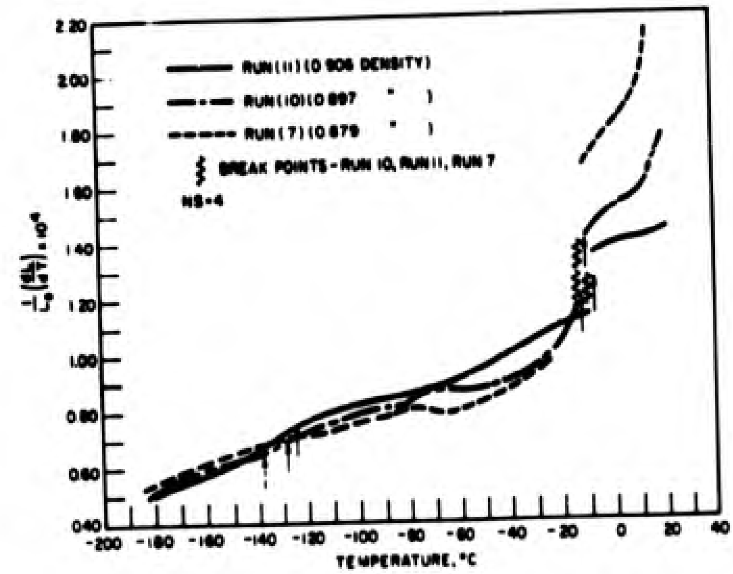


Figure 97. Linear expansion coefficient as a function of temperature for a polyethylene blends (64 moles percent - 0.897 density; 88 mole percent - 0.906 density 32 mole percent - 0.879 density) (50)

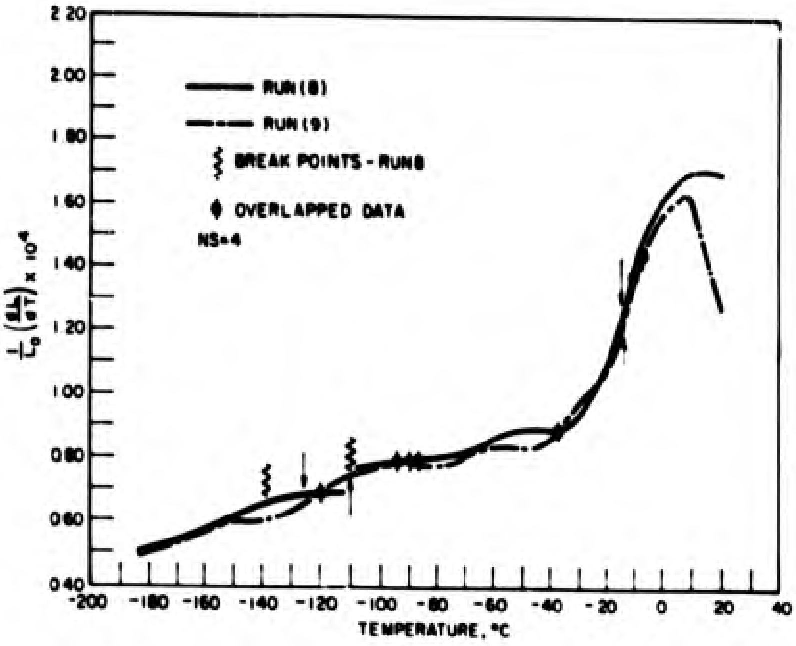


Figure 98. Linear expansion coefficient as a function of temperature for a polyethylene (48 mole percent) - polypropylene blend (0.888 density) (50)

The thermal conductivity as a function of temperature for various density polyethylenes is shown in Figure 99. As can be seen, the thermal conductivity changes vary little for low and medium density polyethylenes over a wide temperature range. The increase in thermal conductivity becomes significant at the lower temperatures with increasing densities.

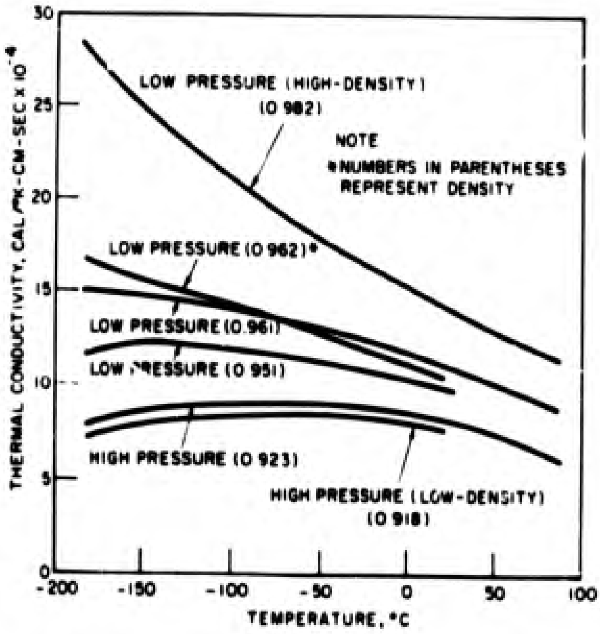


Figure 99. Effect of temperature on high and low-pressure polyethylenes of varying density(39)

As would be expected, density, melt index and molecular weight distribution also affect brittleness temperature. Figures 100 to 102 illustrate these factors. As can be seen, superior low temperature toughness would be found in a very dense polyethylene of extremely low melt index and the narrowest molecular weight distribution possible.

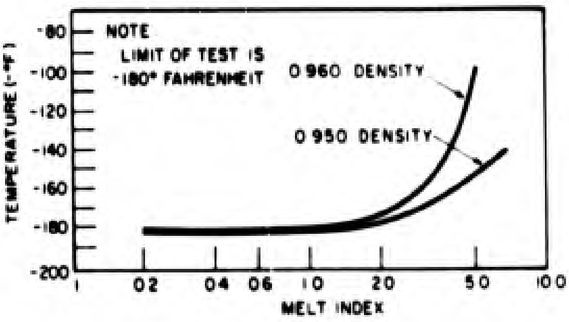


Figure 100. Melt index versus brittleness temperature of polyethylene (47)

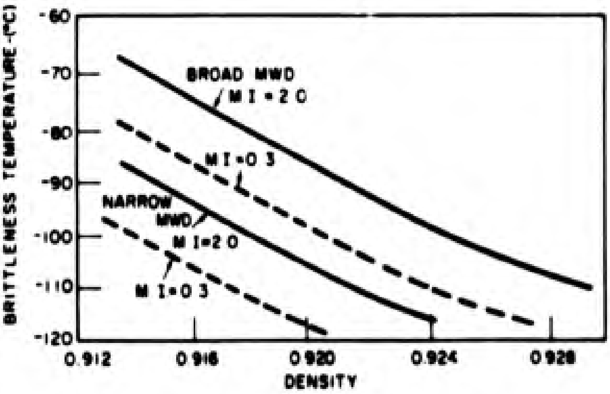


Figure 101. Effects of variables on low temperature brittleness of polyethylenes (49)

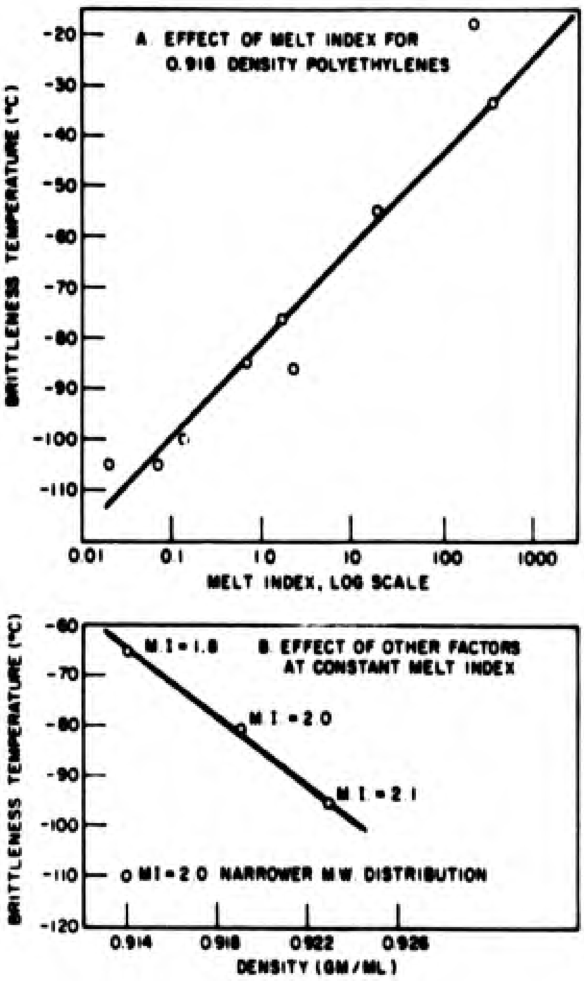


Figure 102. Effect of polymer parameters on brittleness temperature (ASTM D-746-57T) of polyethylene (39)

The term brittleness applied to polyethylene film means splitting along a crease when rapidly stressed. Figure 103 shows the effects of density and melt index on balanced film.

Balanced film is oriented equally in both directions. Most films are oriented more in the machine direction than in the transverse or circumferential direction. As this orientation becomes more unbalanced, the film becomes more brittle when stressed in the direction perpendicular to the orientation. Figure 104 shows the advantage of a narrow molecular weight distribution in the brittleness temperature of unbalanced films. Also, films made from a resin of narrow molecular weight distribution are tougher than similar film made from a resin of the same melt index and density but broader molecular weight distribution. (49)

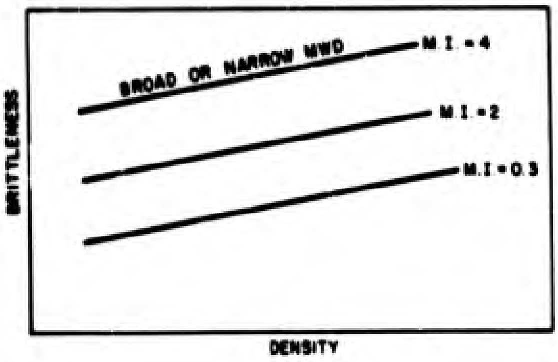


Figure 103. Effects of density and melt index on the brittleness of film having "balanced" orientation (49)

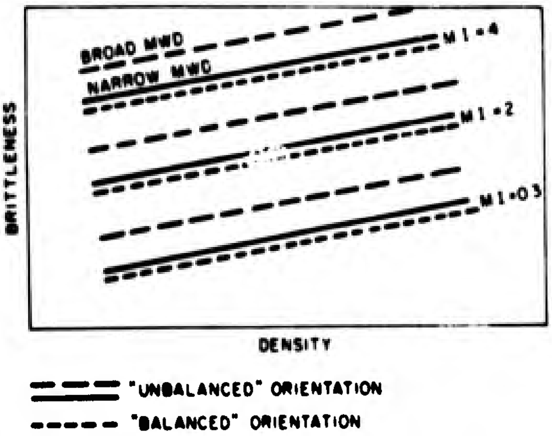


Figure 104. Effects of density and melt index on the brittleness of film having "unbalanced" orientation (49)

The brittleness temperatures for some typical polyethylenes are given in Table 30. Average values for EVA and EEA copolymers and cross-linked polyethylenes are given below.

TABLE 30. POLYETHYLENE BRITTLENESS TEMPERATURES(5)

GRACE RESINS			
	Properties		
No.	Melt Index (gms 10 min.)(1)	Density (gms/cc)(2)	Brittleness Temperature, °F(3)
DD50-150(4)	15.0	0.95	< -106
DD60-150(4)	15.0	0.96	< -92
DD60-220(4)	22.0	0.96	< -50
DD60-300(4)	30.0	0.96	< -66
SS50-010(4)	1.0	0.95	< -106
SS50-035(4)	3.5	0.95	< -106
SS50-050(4)	6.0	0.95	< -106
SS50-090(4)	9.0	0.95	< -106
SS60-015(4)	1.5	0.96	< -106
SS60-035(4)	3.5	0.96	< -106
SS60-050(4)	6.0	0.96	< -106
PP50-004(5)	0.3	0.95	< -106
PP60-002(5)	0.3	0.96	< -106
PP60-007(5)	0.8	0.96	< -106
PP60-007(5)	0.8	0.96	< -106
3001 Natural(6)	0.4	0.95	< -76
3002 Black(6)	0.3	0.949(14)	< -76
3003(6)	0.3	0.95	< -76
SF60-060(7)	6.0	0.96	< -106
SF50-060(7)	6.0	0.95	< -106
PP60-002(8)	0.3	0.96	< -106
DD60-003(8)	0.3	0.96	< -106

MARLEX RESINS			
	Properties		
No.	Melt Index (gms 10 min)(1)	Density (gms/cc)(2)	Brittleness Temperature, °F(3)
1478(9)	7.8	0.914	< -105
1531(9)	3.1	0.915	< -105
1702(9)	0.2	0.917	< -105
1712(9)	1.2	0.917	< -105
2130(9)	3.0	0.921	< -105
2320(9)	2.0	0.923	< -105
2331(9)	3.1	0.923	< -106
2520(9)	2.0	0.926	< -106
3250(9)	5.0	0.932	< -180
3328(9)	2.8	0.933	< -106
4270(9)	7.0	0.942	< -180
5002(9)	0.2	0.948	< -180
5003(9)	0.3	0.950	< -180
5005(9)	0.5	0.950	< -180
5012(9)	1.2	0.950	< -180
TR-704(7)(8)	0.4	0.917	< -106
TR-904(8)	0.2	0.917	< -106
TR-906(8)	0.2	0.920	< -106
TR-906(8)	0.3	0.927	< -105
TR-330(7)	8.0	0.930	-72
TR-882(10)	22	0.923	-72
TR-871(10)	55	0.925	-13
6040(10)(11)	4.0	0.950	-100
6065(10)(11)	6.5	0.950	-120
6090(10)(11)	9	0.950	-45
5005(10)(11)	0.5	0.950	-35
6003(10)(11)	0.3	0.956	< -100

(1) ASTM D1238
(2) ASTM D1505
(3) ASTM D746
(4) Injection molding
(5) Blow molding, extrusion
(6) Electric grade
(7) Film
(8) Sheet
(9) Film, sheet, blow molding
(10) Molding
(11) Copolymers, ASTM Type III
(12) ASTM Type I
(13) ASTM Type II
(14) Base resin

TABLE 31. BRITTLENESS TEMPERATURE OF COPOLYMERS AND CROSS-LINKED POLYETHYLENE (5)

Material	Brittleness Index (^{o}C) (1)
Copolymer EVA	-90
Copolymer EEA	-95
Cross-linked, high density, unfilled	<-65
Cross-linked, low density, 37 1/2% carbon black	<-70
Cross-linked, low density, 70% carbon black	<-15

(1) ASTM D 746 - 64T

SECTION X. POLYPHENYLENE OXIDE

Mechanical Properties

Polyphenylene oxide is relatively new (becoming commercially available in 1964) and little data are available in the interested temperature range.

The tensile properties of polyphenylene oxide (PPO) molding materials are shown in Figures 105 to 107. Tensile strength and modulus are very good compared to other thermoplastics and change little over the interested temperature range. Like other thermoplastics, strength and rigidity increase with decreasing temperatures.

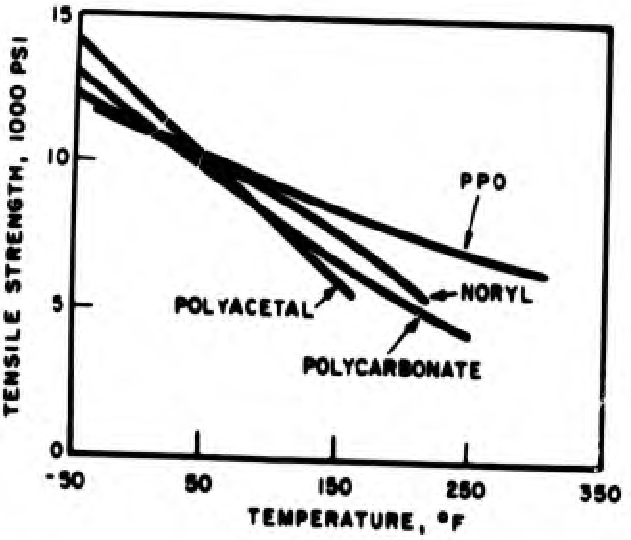


Figure 105. Tensile strength versus temperature of several thermoplastics (52)

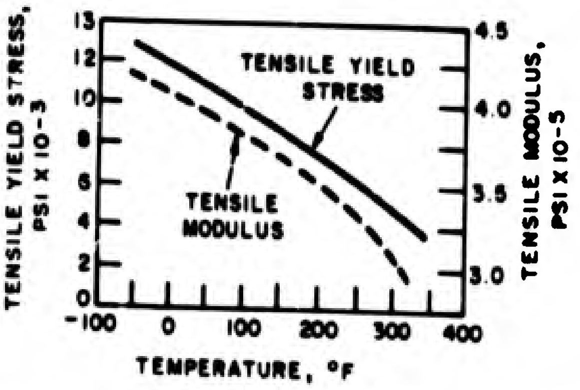


Figure 106. Effect of temperature on modulus and strength of polyphenylene oxide (5)

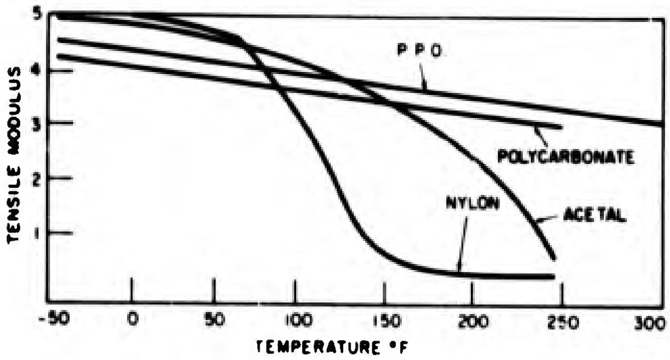


Figure 107. Tensile modulus versus temperature of several thermoplastics(53)

The notched impact strength of PPO is compared to some other thermoplastics and Noryl (a modified PPO) in Figure 108. As Figures 108 and 109 show, PPO's impact strength is stable over a wide temperature range.

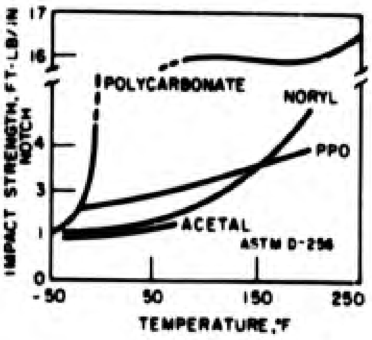


Figure 108. Izod impact strength versus temperature of several thermoplastics (52)

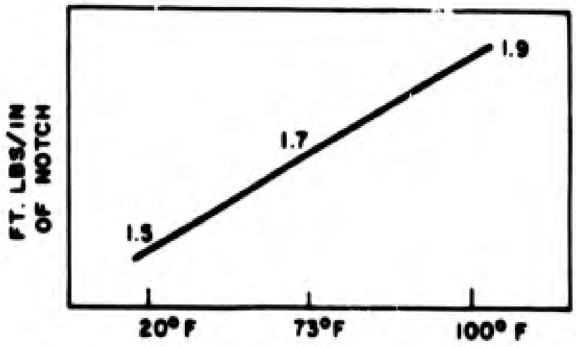


Figure 109. Izod impact versus temperature of PPO (53)

Electrical Properties

Tables 32 and 33 list the dissipation factor and dielectric constant of PPO (C-1001 molding grade material) at high and low temperatures over various frequencies. From this data it can be seen that the dissipation factor and dielectric constant are relatively unaffected by variations in temperature or frequency.

TABLE 32. DISSIPATION FACTOR OF PPO VERSUS TEMPERATURE AND FREQUENCY (53)

TEMPERATURE °C	100CPS	1KC	10KC	1MC
90	0.0003	0.0003	0.0003	0.0003
23	0.0003	0.0003	0.0003	0.0006
-6	0.0003	0.0003	0.0003	0.0009
-71	0.0006	0.001	0.001	0.0009

TABLE 33. DIELECTRIC CONSTANT VERSUS TEMPERATURE OF PPO(53)

TEMPERATURE °C	100CPS	1KC	10KC	1MC
90	2.51	2.51	2.51	2.51
23	2.53	2.53	2.53	2.53
-6	2.54	2.54	2.54	2.53
-71	2.55	2.55	2.54	2.54

Another property which is relatively stable as temperature changes is volume resistivity. Data are given in Table 34.

TABLE 34. VOLUME RESISTIVITY VERSUS TEMPERATURE(53)

TEMPERATURE °C	
90	8.0×10^{16}
23	1.3×10^{17}
-6	2.8×10^{17}
-71	6×10^{16}

Thermal Property

Polyphenylene oxide has a wide usable temperature range. The brittle temperature is reported to be -275° F.(5)

SECTION XI

POLYPROPYLENE, BLENDED PROPYLENES AND PROPYLENE COPOLYMERS

The physical properties of polypropylene and propylene copolymers are influenced by molecular structure (stereoregularity, crystallinity, molecular weight, molecular weight distribution and orientation) in addition to such parameters as temperature, rate of loading, time, etc. Generally, interested properties are affected as shown in Table 35.

TABLE 35. HOW MOLECULAR PROPERTIES AFFECT PHYSICAL PROPERTIES OF POLYPROPYLENES (54)

Physical Properties	As Density (Crystallinity) Increases...	As Average Molecular Weight Increas's...	As Molecular Weight Distribution is Narrowed...
Tensile Strength			
Yield	Higher	Lower	-
Break	Lower	Lower	Higher
Elongation	Lower	Lower	-
Flexural Stiffness	Higher	Lower	-
Flexibility	Lower	-	-
Low Temperature Brittleness	Lower	Lower	Lower
Electrical Properties	Higher	-	-
Impact Strength			
Unnotched	Lower	Higher	Higher
Notched	Lower	Higher	Higher

The degree of molecular alignment or orientation induced during fabrication also affects the properties of polypropylenes. Because of orientation, molded parts may possess different property values in different directions. For example, strength and impact properties are better parallel to the direction of orientation than they are perpendicular to this direction.

Mechanical Properties

Figure 110 presents the average tensile yield strengths for a general purpose and a polypropylene blend* molding materials. The yield strength increases with lowering temperature and is the highest for the unmodified homopolymer.

The tensile and flexural modulus of various polypropylenes are given in Figures 111 and 112. As can be seen, the values for flexural moduli of elasticity are quite different than the tensile moduli. Moduli of all compositions increase with decreasing temperature with the unmodified (general purpose) normally being the stiffest.

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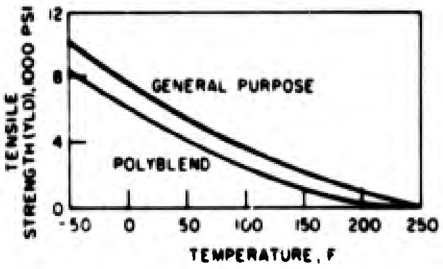
* Polypropylene blend (polyblends) usually consist of polyisobutylene dispersed in polypropylene.



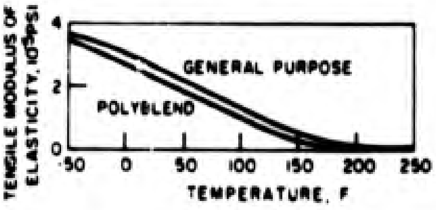


Figure 110. Tensile yield strength of polypropylenes (54)

Figure 111. Flexural modulus vs temperature of polypropylenes (54)

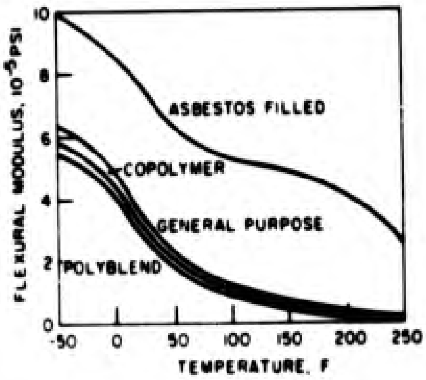


Figure 112. Flexural modulus vs temperature of polypropylenes (54)

The apparent modulus of rigidity of 1/4 x 1/16 x 2" general purpose polypropylene specimens was determined according to ASTM D1043 (4). It was stated that as the temperature was raised, the specimens bowed slightly between the clamp. Therefore, the values will be in error and should be taken with reservations. A graph of the results is contained in Figure 113.

As previously stated many factors affect the impact properties of thermoplastics. The common factors affecting impact performance of polypropylenes are the base resin type, crystallinity, molecular weight, molecular weight distribution, part configuration, fabrication history, additives and pigments, rate of loading, and of course temperature.

Impact tests were conducted to determine the effects of orientation and strength (4). Izod specimens were prepared from injected molded flex bars (5 x 1/2 x 1/8") which were cut in half and notched (ASTM D256) in both the gated and dead ends. Additional Izod specimens were prepared from the reduced sections of Type 1 tensile bars. Tests were run at room temperature and at 0^{o} C.

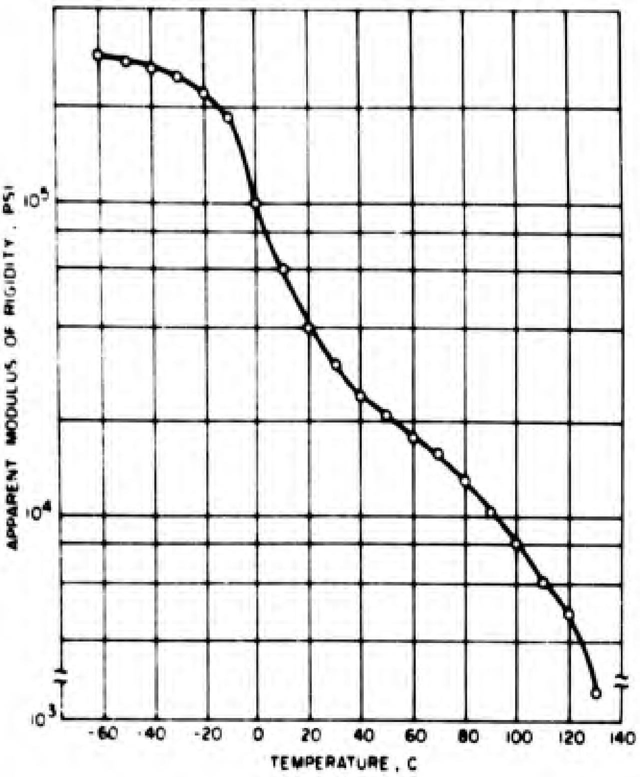


Figure 113. Modulus of rigidity for a typical general polypropylene ASTM D1043 (4)

This was compared to similar unnotched impact specimens tested at -18°C. The results are listed in Table 36.

TABLE 36. EFFECT OF ORIENTATION ON IZOD IMPACT STRENGTH (4)

	Izod Impact Strength, ft-lb inch (ASTM D 256)								
	Room Temperature, Notched Specimens			0 C, Notched Specimens			-18 C, Unnotched Specimens		
Sample	Gated End, Flex Bar	Dead End, Flex Bar	Reduced Section, Tensile Bar	Gated End, Flex Bar	Dead End, Flex Bar	Reduced Section, Tensile Bar	Gated End, Flex Bar	Dead End, Flex Bar	Reduced Section, Tensile Bar
A(1)	0.98	0.93	1.15	0.78	0.72	0.83	3.55	3.71	4.28
B(1)	0.85	0.84	0.96	0.72	0.70	0.76	2.97	3.08	3.47
C(1)	0.82	0.80	0.99	0.70	0.61	0.78	3.15	3.01	3.85
D(1)	0.75	0.71	0.88	0.62	0.61	0.72	2.86	2.69	3.13
E(1)	1.02	0.92	1.24	0.74	0.73	0.83	3.23	3.34	6.00
F(2)	0.84	0.80	0.92	0.70	0.67	0.72	3.19	2.88	3.86
G(2)	0.84	0.80	0.97	0.73	0.64	0.78	3.19	3.09	3.50
H(2)	0.75	0.72	0.87	0.61	0.60	0.72	2.98	2.73	3.45
I (2)	0.95	0.85	1.21	0.75	0.68	0.80	2.90	2.86	3.59
J (3)	0.85	0.83	1.00	0.72	0.69	0.75	2.89	2.94	3.37
K(3)	0.80	0.81	0.97	0.70	0.68	0.77	2.97	2.95	3.37
L(3)	1.63	1.50	1.83	1.00	0.97	1.07	8.55	8.28	9.80
M(4)	1.12	1.14	1.30	0.77	0.77	0.88	8.02	7.38	10.77
N(4)	1.58	1.46	1.73	0.88	0.87	0.89	10.04	8.89	12.98
O(4)	3.56	3.81	3.46	0.89	0.82	0.93	9.86	9.83	13.83

(1) General Purpose
(2) Heat Resistant
(3) Light Resistant
(4) Impact Resistant

As can be seen, the Izod values for notched specimens drop from tensile bars to gated end to dead end. The difference attributed to orientation is small but could increase with different molding conditions. (4)

Two additional points are interesting to note. One, at 0°C the Izod notched values of the regular specimens are about the same as those at room temperature. Also, there is no significant improvement in the impact grades at 0°C over the regular grades. Secondly, the impact strengths of the unnotched impact grades are about three times those of the unmodified specimens at -18°C. Comparing the impact strengths of the notched and unnotched at 0° and -18°C indicates that the presence of a stress raiser (notch) apparently eliminates the advantages of the impact-resistant materials.

Figure 114 illustrates the notch sensitivity of a general-purpose polypropylene at low temperatures.

Figures 115 through 118 present impact data on extrusion and molding grades of various polypropylenes and copolymers.

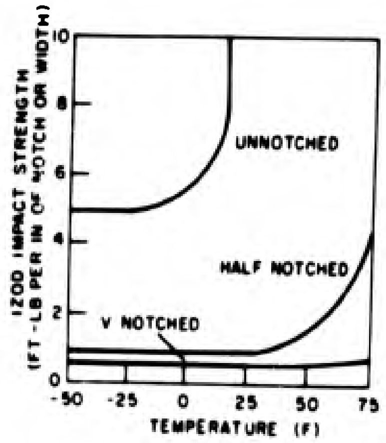


Figure 114. Effect of temperature on impact strength of general-purpose polypropylene for various test speciments. (55)

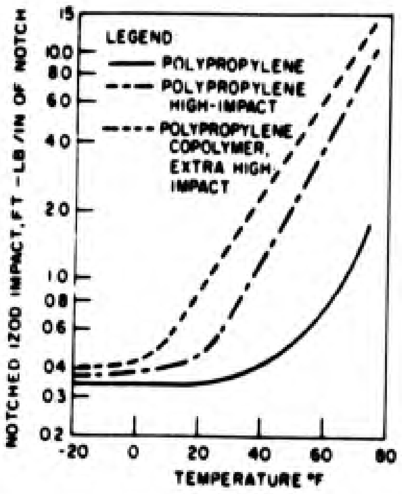


Figure 115. Effect of temperature on notched Izod impact strength on three types of extrusion grades. (5)

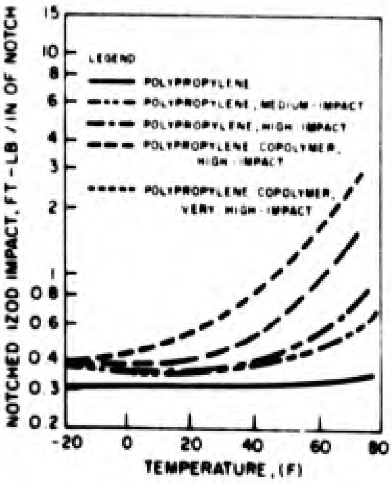


Figure 116. Effect of temperature on notched Izod impact strength of molding grades of polypropylene resins. (Pure resin has poorest strength.) (5)

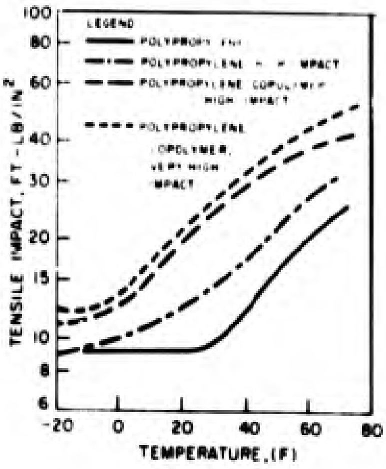


Figure 117. Effect of temperature on tensile impact of molding grades. (Modified types have better low temperature strength.) (5)

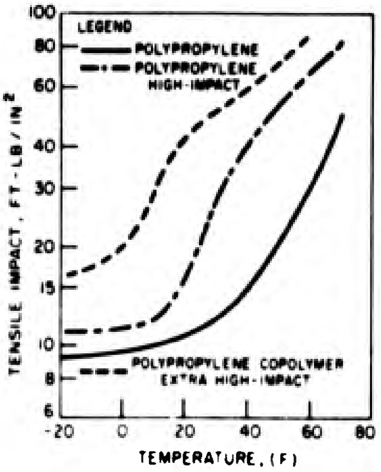


Figure 118. Effect of temperature on the tensile impact strength of extrusion grades. (Effect of Modification is similar to that in molding grades.) (5)

The number of additives, pigments and colors in plastics today is almost unlimited. These also have a significant effect on impact strength as illustrated in Figures 119 and 120 and must be considered in selecting materials for many applications. Figure 119 shows the effect of carbon black content on a polypropylene alloy at -18°C. The effect of the per cent loading and particle size of a metallic pigment is plotted in Figure 120.

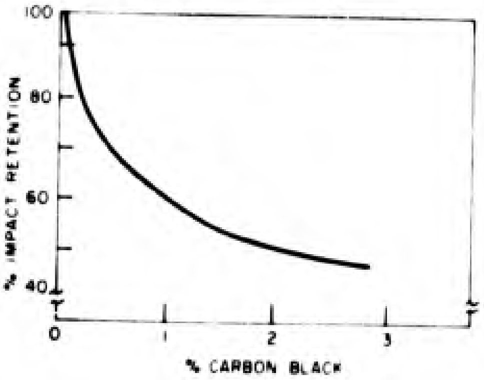


Figure 119. Effect of carbon black content on drop weight, -18°C (56)

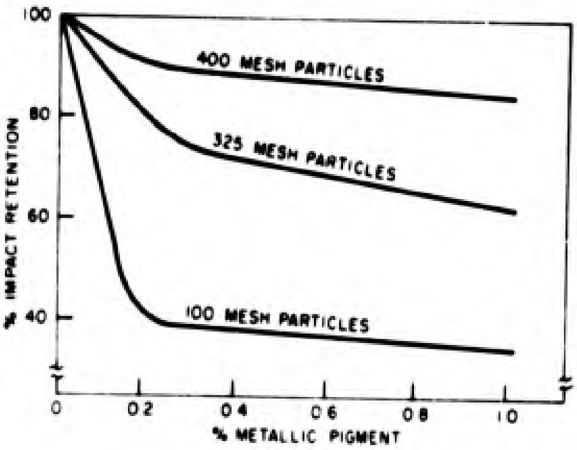


Figure 120. Effect of metallic pigment content on drop weight impact, -18°C. (56)

An illustration of the impact strength advantages to be gained in going to higher molecular weights (lower melt flow rates) is shown in Figure 121.

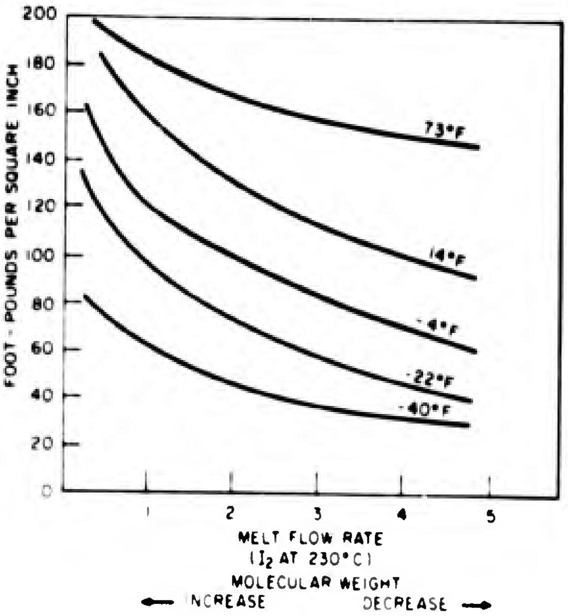


Figure 121. Propylene copolymer, tensile impact resistance (56)

This data represents tensile impact values for polypropylene copolymers with melt flow rates from 4 to 0.4 at test temperatures from -40 to 73°F. If, for example, the performance temperature has been established as -22°F, a resin with a melt flow rate of 4 will have a tensile inpact strength of 45 ft. -lbs/sq. in. However, if one were to go down to the 2 melt flow range, such a product would have a 70 ft. -lb/sq. in. tensile impact, a very marked increase in resistance to impact failures.

The data presented in Figure 121 also points up another interesting facet of the effect of polypropylene molecular weight on low temperature impact properties. Dropping stepwise from melt flow rates of 4 through 2 to -.8, which are commonly accepted melt flow types, equal impact performance can be obtained at about 18°F lower temperatures. For this series of resins, the 4 melt flow type has a tensile impact of 100 ft. -lbs/sq. in. at +14°F, the 2 melt flow resin has 100 ft. -lbs./sq. in. at -4°F, and the 0.8 melt flow type is 100 at -22°F. (56)

Thermal Properties (See also Section IX)

Brittleness temperature tests (ASTM D746) were conducted on four types of polypropylenes. (4) Specimens were die cut from compression-molded disks and evaluated at -55°C, 0°C, +10°C and +20°C. At -55°C, all the polypropylenes failed, and at 0°C only an impact resistant type (sample O) showed any resistance. Even at +10°C most of the samples had brittle behavior as shown below, two materials (samples L & O) had no failures.

Code	Type	Brittle Behavior at 10°C Percent Failure
A-E	General purpose	100
F-H	Heat resistant	100
I	Heat resistant	75
J, K	Light resistant	100
L	Light resistant	0
M	Impact resistant	13
N	Impact resistant	25
O	Impact resistant	0

The authors report that polypropylene containing about 5 percent of hexane-soluble material has a brittle temperature of +20°C. This could be reduced to +10°C if the hexane-soluble fraction was increased to around 17 percent. Even though the polypropylenes have relatively low Izod impact strength and high brittleness temperature, they are still used in certain low-temperature applications. In some areas, polypropylene has been found suitable at temperatures as low as -80°C. This points out the fallacy of relying on any single test. (4)

It is possible to improve the brittle temperature of polypropylenes by the addition of plasticizers. However, this will also significantly reduce its strength and elongation properties. Therefore, the plasticized polypropylenes were modified by cross-linking agents during processing to produce a superior low-temperature resistant material with the strength of the original polypropylenes. (57) Figure 122 shows the brittleness temperature of modified propylenes as a function of cross-linking agents.

As can be seen, a 15 percent dioctyl sebacate plasticized propylene with 4 percent cross-linking agent improves the brittle temperature from -18°C to -65°C. Also the tensile strength increases from 3,058 psi to 3,556 psi.

Figure 123 plots the coefficient of linear expansion of a general purpose polypropylene from -40 to 104°F. Values range from about 2.0 at -40° to 4 in/in/°F x 10^{-5} at 100°F.

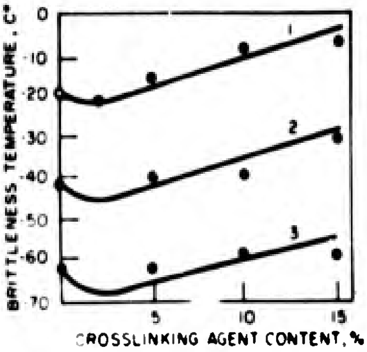


Figure 122. Changes in the brittleness temperature of modified polypropylene as a function of the content of cross-linking agents. 1 - original polypropylene; 2 - polypropylene +7% dioctyl sebacate; 3 - polypropylene +15% dioctyl sebacate. (57)

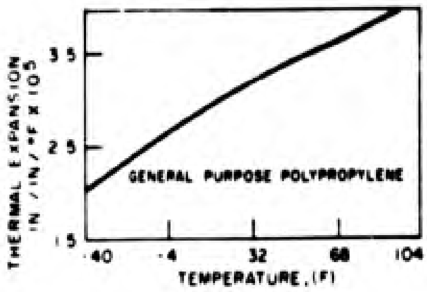


Figure 123. Variation in coefficient of linear thermal expansion of a general purpose polypropylene. (54)

SECTION XII. POLYSTYRENE POLYMERS AND COPOLYMERS

Mechanical Properties

The mechanical properties of general purpose and impact grades of compression molded polystyrenes are plotted in Figure 124. The shaded areas represent the range of values and the solid line, typical values.

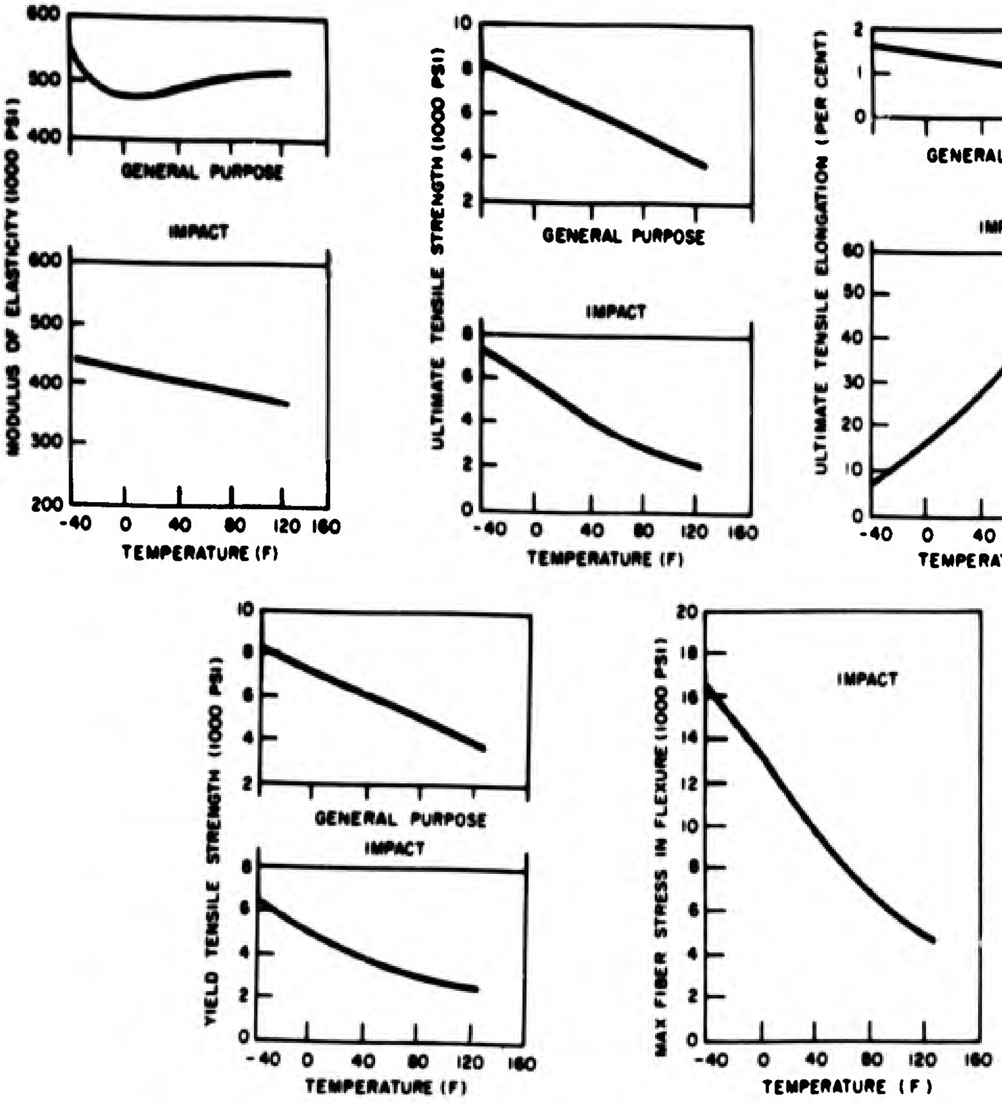


Figure 124. Temperature vs mechanical properties for typical general-purpose and impact grades of compression-molded polystyrenes (38)

Figure 125 shows the mechanical properties of two polystyrene copolymers.

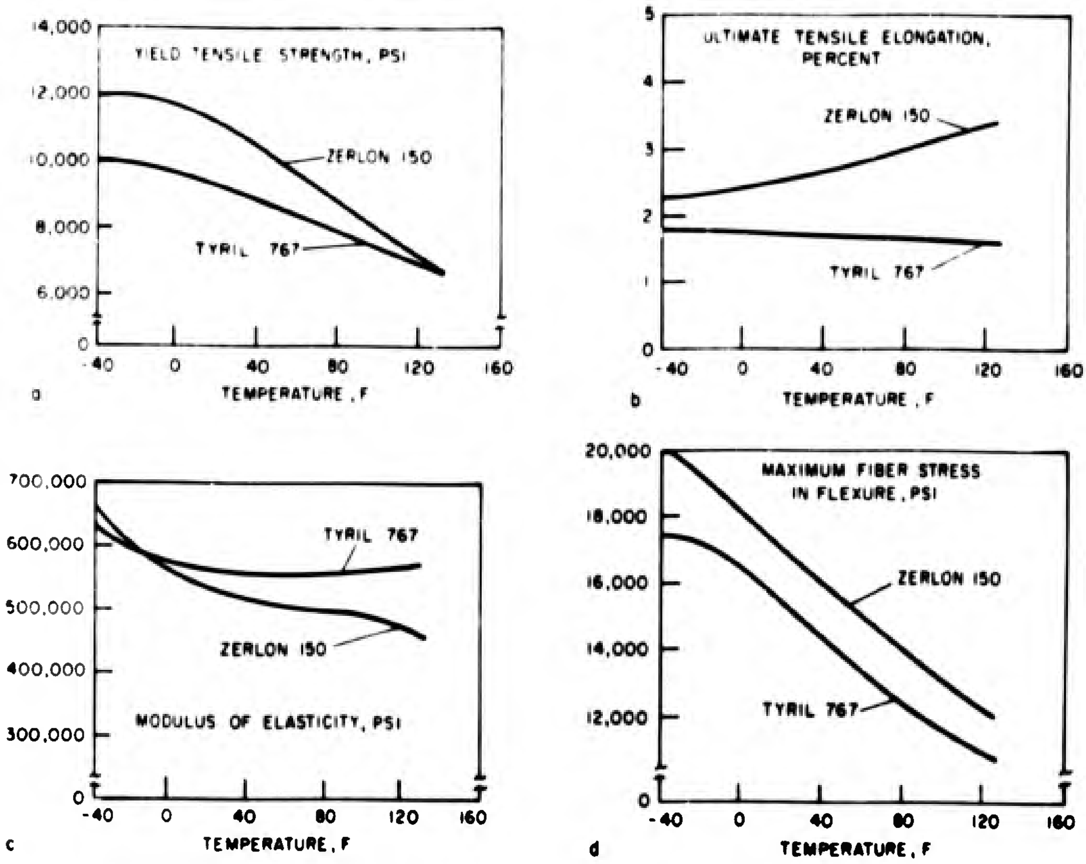


Figure 125. Mechanical properties of polystyrene copolymers as a function of temperature (19)

Note: Tyril - acrylonitrile-styrene copolymer
Zerlon - methyl methacrylate-styrene copolymer

Tensile tests (18) were conducted on polystyrene and polystyrene copolymers at +77, +10, -40 and -65 ° F. Tensile values were obtained on a Tinius-Olsen Plastiversal testing machine. Determinations were made using a constant rate of load increase of 2500 psi/min. Load-elongation graphs were automatically recorded to the point of fracture using Baldwin Models PS-6, PS-7 and PS-8 Microformer-type plastics extensometers coupled to a Baldwin Microformer-type recorder. The results are given in Figure 126.

The notch sensitivity of general purpose polystyrenes was determined (59). Specimens were cut from compression-molded sheets and machined into dumbbell test bars having cross sectional dimensions of 0.125" x 1.00" in the test area. Each bar was carefully drilled in the center of the test area or notched on both sides. The bars were tested in an Instron Model TTB universal testing machine at a crosshead speed of .05"/min.

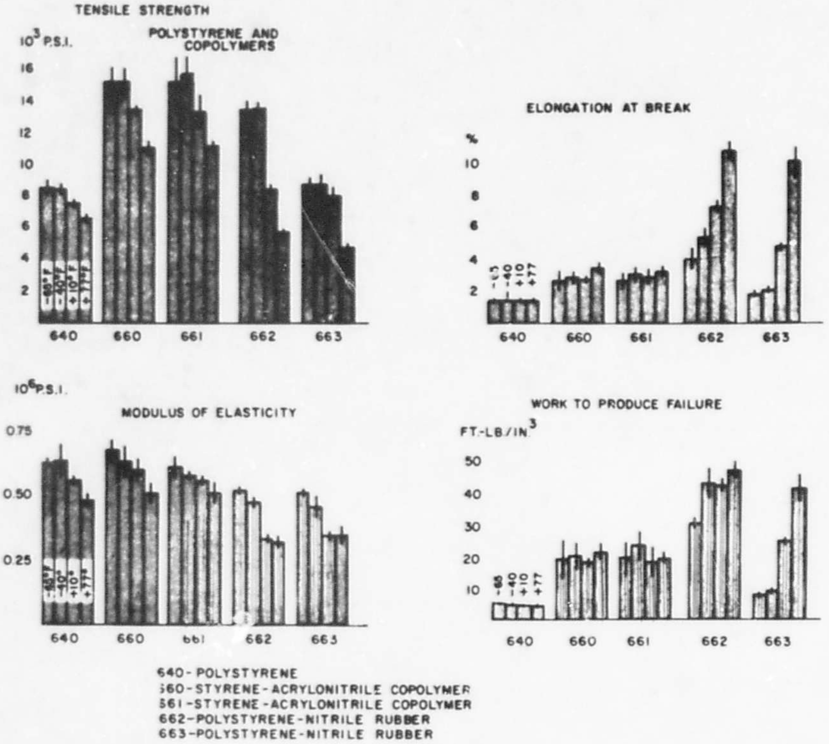


Figure 126. Tensile properties of polystyrene and polystyrene copolymers at -65, -40, +10 and +77 ° F. (18)

The notch sensitivity index in static tension was calculated as follows, based on the remaining cross sectional area:

$$q' = \frac{Kr - 1}{Kt - 1}$$

where $Kr = \frac{\text{tensile strength at rupture of unnotched specimen}}{\text{tensile strength at rupture of notched specimen}}$

Kt = theoretical stress - concentration factor.

Figure 127 shows the notch sensitivity of a general purpose polystyrene at -40 and +73 ° F. The sensitivity at the low temperature (-40 ° F) decreases at a greater rate with decreasing hole size, than at room temperature. In fact it crosses the 73 ° F curve at a hole radius of about 0.10 in. Since lowering the temperature tends to inhibit flow and cause brittle failure, this crossover was not expected.

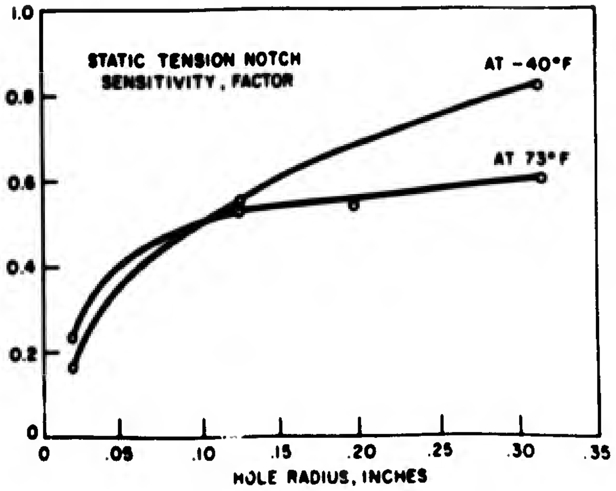


Figure 127. Notch sensitivity of general purpose polystyrene C at -40 and +73° F. (59)

The unexpected effect of temperature is again dramatically shown in Figure 128 for a second general purpose polystyrene. The notch sensitivity was higher at the elevated temperature than at room temperature. This was attributed to local heating which resulted from the local high strain rate added to the already existing elevated temperature, causing a local hot spot at the discontinuity, and thus lowering the tensile strength of the notched specimen. Using this lowered value in the notch sensitivity calculation will result in a high strength-reduction factor, and consequently a high notch-sensitivity factor. As additional work is generated in this and related fields it is felt that this apparent contradiction will be resolved (59).

The general-purpose material GP-P/S-C in Figure 129 was reported to

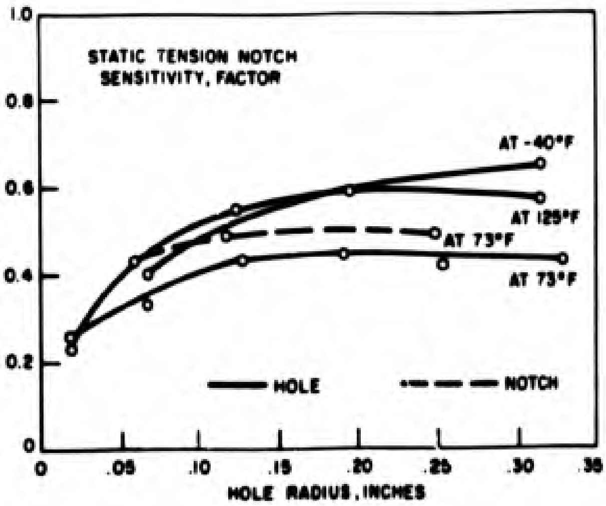


Figure 128. Notch sensitivity of general purpose polystyrene D at -40, +73 and 125° F. (59)

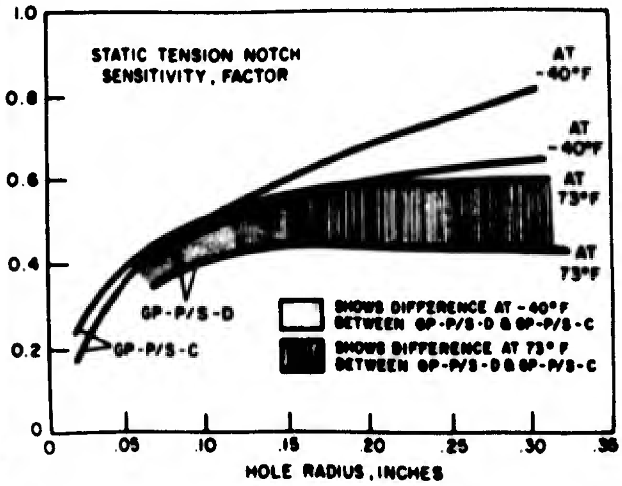


Figure 129. Comparison of two general purpose polystyrenes at two temperatures (59)

have a higher yield strength, higher ultimate strength, slightly higher modulus and about the same elongation as GP-P/S-D. However, the authors report that field experience with these two materials showed that material C would fail when a stress concentration was present before GP-P/S-D. Figure 129 verifies that the C-material is more notch sensitive. This difference in notch sensitivity eliminates the difference in tensile strength when a stress concentrator is present.

The impact strength of general purpose unmodified polystyrene is very low; however, strength is increased by modification and/or glass-reinforcement. Some representative data are given in Figures 130 and 131 and Tables 37-40.

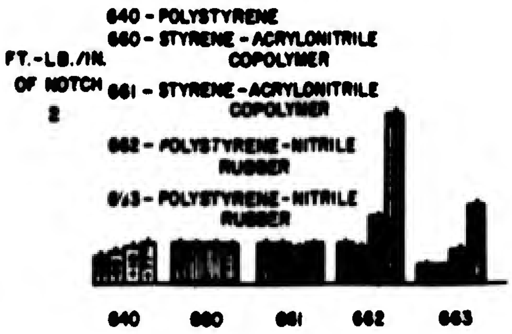


Figure 130. Izod impact strength (18)

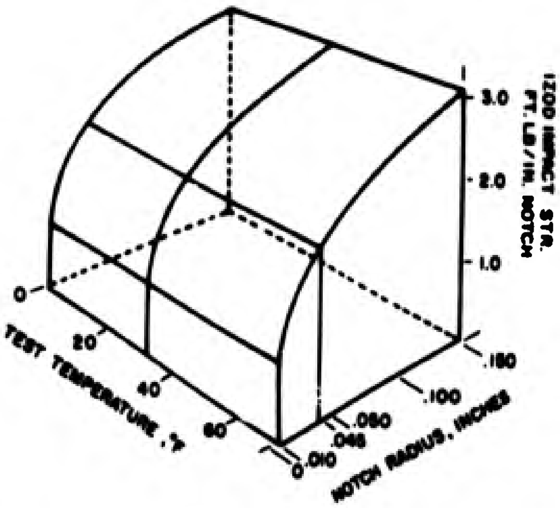


Figure 131. Izod impact strength/ notch radius/temperature relationship for a typical HI polystyrene (16)

TABLE 37. POLYSTYRENE IZOD IMPACT STRENGTH - FT. LB./IN. NOTCH (60)

Grade*	Test Sample	73°F	0°F
Y-14.1 med. Impact, closure grade	1/8" bar inj. mold	1.5	1.1
	1/4" bar inj. mold	0.7	0.6
Y-16.1 med. Impact	1/8" bar inj. mold	1.3	0.65
	1/4" bar inj. mold	0.65	0.45
315 med. Impact, high heat	extruded sheet	0.77	0.63
	inj. molded	0.79	0.57

TABLE 37. POLYSTYRENE IZOD IMPACT STRENGTH - FT. LB/IN. NOTCH (60) (CONT)

Grade*	Test Sample	73 °F	0 °F
324 high Impact, appl. grade	extruded sheet	1.2	0.95
	inj. molded	1.1	0.8
Y-20.1 high Impact	extruded sheet 0.115"	0.95	0.70
Y-28.1 high Impact	extruded sheet 0.115"	1.5	1.1

* Shell Chemical Co.

TABLE 38. POLYSTYRENE FALLING WEIGHT IMPACT STRENGTH - FT. LB./IN. OF THICKNESS (60)

Grade*	73 °F	0 °F
Y-20.1 high impact (0.011" sheet gauge extrusion)	500	350
Y-28.1 high impact (0.115" sheet gauge)	410	380
Y-28.1 high impact (0.011" sheet gauge)	690	420
315 med. impact (extruded sheet)	220	140
324 high impact (extruded sheet)	380	310

* Shell Chemical Company compounds

As shown in Table 39 adding 30 and 20 percent fiberglass to general purpose polystyrene increases strength, stiffness and impact resistance at low temperatures.

Table 40 gives the low temperature properties of glass reinforced styrene acrylonitrile. The data are for two grades, 35 percent and 20 percent fiberglass reinforced.

TABLE 39. PROPERTIES OF FIBERGLASS REINFORCED POLYSTYRENE (STYRAFIL) (17)

Property	°F	ASTM Test	Styrafil G - 30/30	Styrafil G - 32/30	Styrafil G - 37/20
Tensile Strength, K psi	170	D638	12.8	10.9	7.5
	73		14.0	11.0	11.5
	-40		14.3	11.2	14.0
Elongation, %	170	D638	1.1	1.1	2.4
	73		1.1	1.1	3.5
	-40		1.3	1.0	2.5
Modulus of Elasticity, Psi x 10^5	170	D638	10.6	9.9	6.5
	73		12.1	11.6	8.4
	-40		13.2	12.5	9.0
Impact Strength, Izod, (1/2 x 1/4" bars), ft. lb/in	73	D256	2.5	1.4	1.5
	-40		3.2	1.9	1.6

TABLE 40. PROPERTIES OF FIBERGLASS REINFORCED STYRENE - ACRYLONITRILE (ACRYLAFIL) (17)

Property	°F	ASTM Test	Acrylafil G - 40/35	Acrylafil G - 47/20
Tensile Strength, K psi	170	D638	17.5	9.0
	73		19.5	13.0
	-40		20.0	14.0
Elongation, %	170	D638	1.4	2.6
	73		1.4	2.2
	-40		1.2	1.6
Flexural Modulus, Psi x 10^5	170	D790	11.5	8.8
	73		14.5	10.6
	-40		15.0	10.9
Impact Strength, Izod, (1/2 x 1/4" bars), ft. lb/in.	73	D256	3.0	1.2
	-40		4.0	1.4

Electrical Properties

The dielectric constant of an NBS casting resin is shown in Figure 132. The data was obtained on a modified Schering bridge at frequencies from 10^2 to 10^5 cps and a series resonance circuit at 10^6 and 10^7 cps. The specimen was approximately 2.0" in diameter and 0.2" thick.

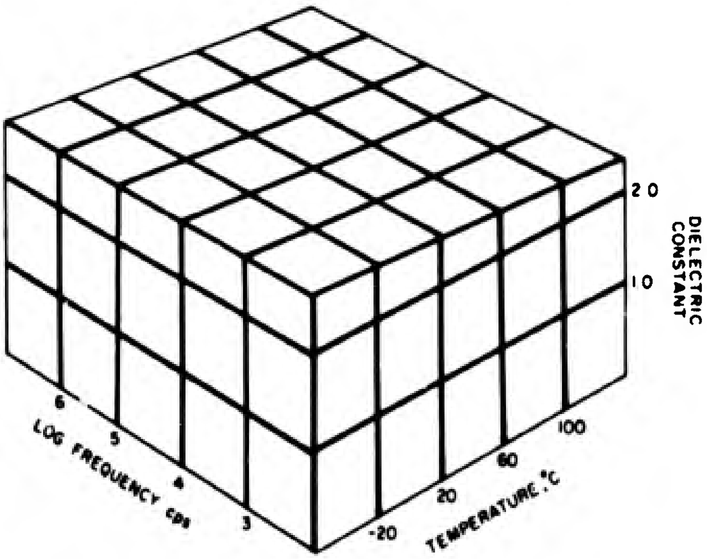


Figure 132. Dielectric constant of a modified polystyrene resin (NBS* casting resin) as a function of frequency and temperature (61)

		Parts by weight
* Modified polystyrene (NBS casting resin)	2, 5-dichlorostyrene	33
	Poly-2, 5-dichlorostyrene	21.5
	Styrene	21
	Polystyrene	11
	Hydrogenated terphenyl	13
	Divinylbenzene, 40% sol'n	0.5
	Cumene hydroperoxide	0.1

SECTION XIII. POLYSULFONES

Polysulfone is a relatively new family of heat-resistant, high performance thermoplastics. Since they were only introduced in 1965 and interest lies mainly in high temperature applications, little low temperature data is available. However, information is included to the extent available.

Mechanical Properties

Figures 133 - 135 plot yield stress, elongation and rupture energy versus temperature for a special Union Carbide extrusion grade polysulfone P-2300. Yield strength is good and it increases with decreasing temperature and increasing loading rate. Figures 134 and 135 show that the material is ductile at temperatures above -75° C.

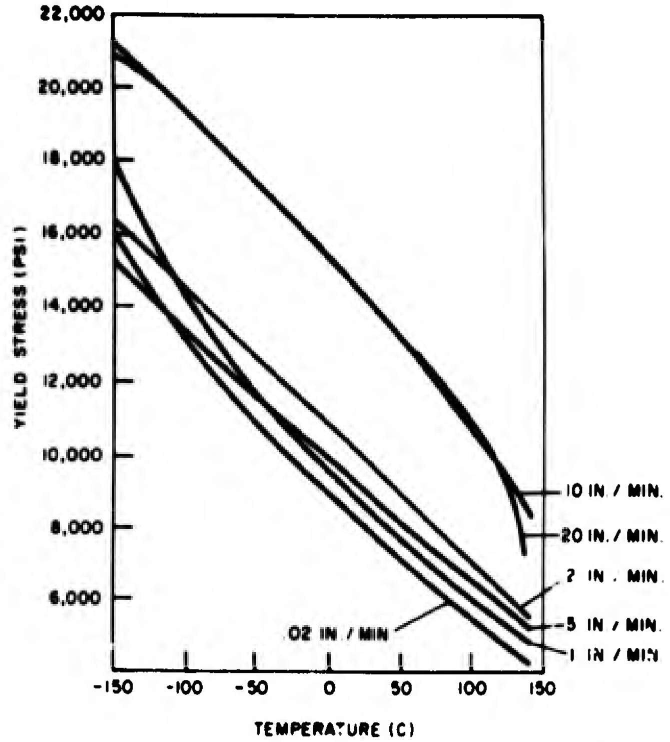


Figure 133. Yield stress vs temperatures for polysulfone at various loading rates (62)

Polysulfones are notch-sensitive as most thermoplastics. Notched Izod Impact strength changes little with decreasing temperature, being 1.3 ft. lb. /in. for 1/8" bar at room temperature and 1.2 at -40° F. (63)

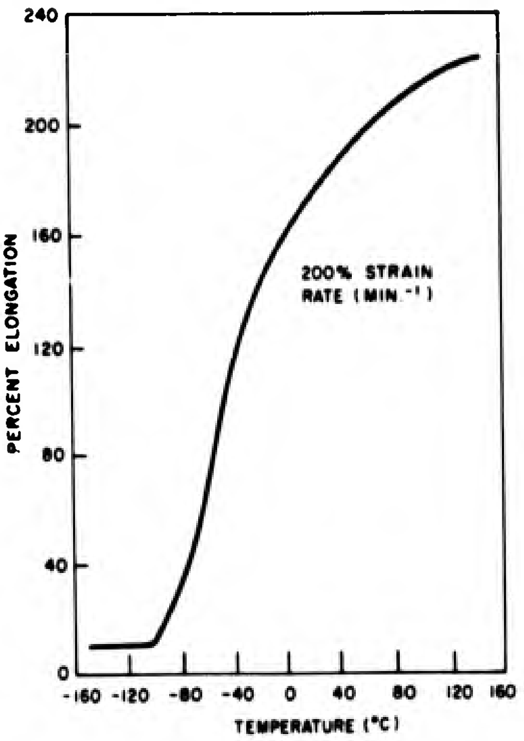


Figure 134. Percent elongation vs temperature for polysulfone (62)

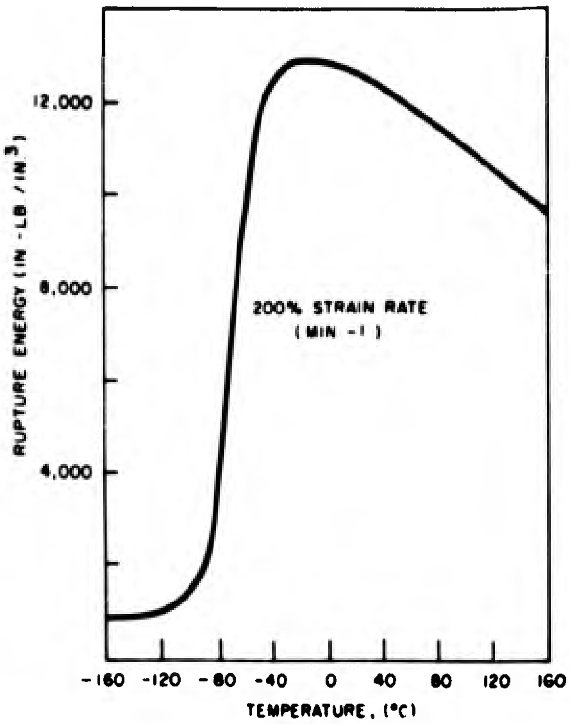


Figure 135. Rupture energy vs temperature for polysulfone (62)

Thermal Properties

The coefficient of thermal expansion is 3.1 x 10^{-5} in/in/°F over the temperature range -22 to +300° F. (63)

SECTION XIV. VINYLS

The vinyl family as generally considered by usage consists of: homopolymers polyvinylchloride, polyvinyldichloride; copolymers of vinyl chloride with vinyl acetate, vinyl alcohol, vinylidene chloride, vinyl butyral, etc.; and mixtures of polyvinylchloride or vinyl copolymers with other polymers. Because of its many variations and innumerable formulations within each variation, vinyls are available in many forms and have a wide range of properties.

Mechanical Properties

Tensile tests were conducted on the following PVC formulations (64):

PLASTICIZED PVC FORMULATIONS

Ingredients	Parts							
	1	2	3	4	5	6	7	8
GEON 101EP	100	100	100	100	100	100	100	100
Ba/Cd Stabilizer	2	2	2	2	2	2	2	2
Epoxy Stabilizer	4	4	4	4	4	4	4	4
DOP*	30	40	50	60				
610P**					30	40	50	60

* di-2-ethylhexanol
** n-hexanol, N-octanol, N-decanol; 20/45/35 ratio

Tests were run in a temperature cabinet on an Instron tensile tester at a crosshead speed of 1, 10 and 20 inches per minute at +10, -5, -20 and -35° C. No extensometer was used and the jaw separation of 2 inches was assumed to be the gauge length. This is accurate at room temperature but at the lower temperature, the per cent elongation and 100 per cent modulus are reported lower than they actually are. In addition, the authors point out that the more plasticizer in the formulation, the less effect the lowering of temperature will have on the accuracy of the 2 inch gauge length.

Table 41 gives the tensile data. From this data the following conclusions can be drawn:

- Tensile break and 100 per cent modulus decrease as the plasticizer is raised from 30 to 60 phr, but the per cent elongation increases.

- Tensile break and 100 per cent modulus increase as the temperature is lowered from 10° C to -35° C while the per cent elongation decreases. At the lower temperatures, a yield point appears in the stress-strain curve. (See also Figure 140.)

- The tensile break and 100 per cent modulus increase, the per cent elongation decreases and the yield strength increases when the rate of loading is raised from 1 to 20 inches per minute.

Figure 136 shows the per cent elongation versus rate of loading and temperature for 30 and 60 phr of DOP and 610P plasticized PVC.

TABLE 41. TENSILE TEST DATA ON PVC FORMULATIONS (64)

Property	Temp °C	Rate of Loading	DOP Plasticizer 30 phr	DOP Plasticizer 40 phr	DOP Plasticizer 50 phr	DOP Plasticizer 60 phr	610P Plasticizer 30 phr	610P Plasticizer 40 phr	610P Plasticizer 50 phr	610P Plasticizer 60 phr
Tensile, psi	10	1"/min	3872	3352	3017	2870	4086	3424	2750	-
	-5	1"/min	5140	4332	3567	3136	4756	4016	3300	-
	-20	1"/min	5952	5514	5685	3756	4954	4890	4112	-
	-35	1"/min	7370	6614	5800	5196	6996	6100	5002	-
	10	10"/min	4067	3912	3346	2964	4224	3862	3298	2727
	-5	10"/min	4484	4240	3786	3442	4530	4004	3442	3110
	-20	10"/min	5126	4736	4236	3670	4420	4124	4206	3510
	-35	10"/min	7383	4950	4840	4368	5410	5158	4806	4340
	10	20"/min	4380	3676	3170	2814	4110	3550	3178	2685
	-5	20"/min	4522	4336	3962	3416	4764	4114	3790	3366
	-20	20"/min	4970	4726	4450	3956	5356	4802	4224	3808
	-35	20"/min	8112	5120	4786	4184	5300	5272	4825	4584
Elongation, %	10	1"/min	193	220	284	364	220	266	305	-
	-5	1"/min	167	212	251	300	172	212	244	-
	-20	1"/min	143	182	272	270	152	198	215	-
	-35	1"/min	86	131	177	220	109	151	189	-
	10	10"/min	206	332	306	368	223	271	312	345
	-5	10"/min	159	204	270	326	183	228	245	289
	-20	10"/min	77	155	205	238	132	198	213	221
	-35	10"/min	13	70	159	181	31	140	149	203
	10	20"/min	232	277	299	362	231	252	317	343
	-5	20"/min	172	209	255	293	174	222	272	293
	-20	20"/min	70	124	203	235	101	179	215	239
	-35	20"/min	16	61	141	186	38	120	185	192
100% Modulus, psi	10	1"/min	2962	2122	1430	1114	2922	1934	1310	-
	-5	1"/min	4390	3178	2196	1596	4014	2850	1968	-
	-20	1"/min	5576	4450	4075	2300	4366	3768	2920	-
	-35	1"/min	-	6170	4720	3764	6850	5414	4158	-
	10	10"/min	3290	2702	1818	1362	3242	2312	1730	1268
	-5	10"/min	4118	3454	2522	1920	3924	3016	2320	1798
	-20	10"/min	-	4442	3602	2902	4352	3594	3252	2634
	-35	10"/min	-	-	4542	3826	-	4972	4236	3550
	10	20"/min	3532	2548	1864	1354	3294	2430	1766	1302
	-5	20"/min	4337	3742	2958	2162	4390	3376	2094	2108
	-20	20"/min	-	4632	3962	3212	5325	4418	3518	2868
	-35	20"/min	-	-	4766	4142	-	5182	4510	4114
Yield, psi	-5	1"/min	2962	-	-	-	2320	-	-	-
	-20	1"/min	5152	2400	-	-	3174	1732	-	-
	-35	1"/min	6833	5608	3264	-	7082	3940	-	-
	-5	10"/min	3832	-	-	-	3014	-	-	-
	-20	10"/min	7178	4100	-	-	4946	-	-	-
	-35	10"/min	8970	6778	4144	6024	5094	-	-	-
	-5	20"/min	4630	3010	-	-	3836	1180	-	-
	-20	20"/min	6930	4530	-	-	5686	-	-	-
	-35	20"/min	8112	6812	5096	2826	7200	5814	3452	-

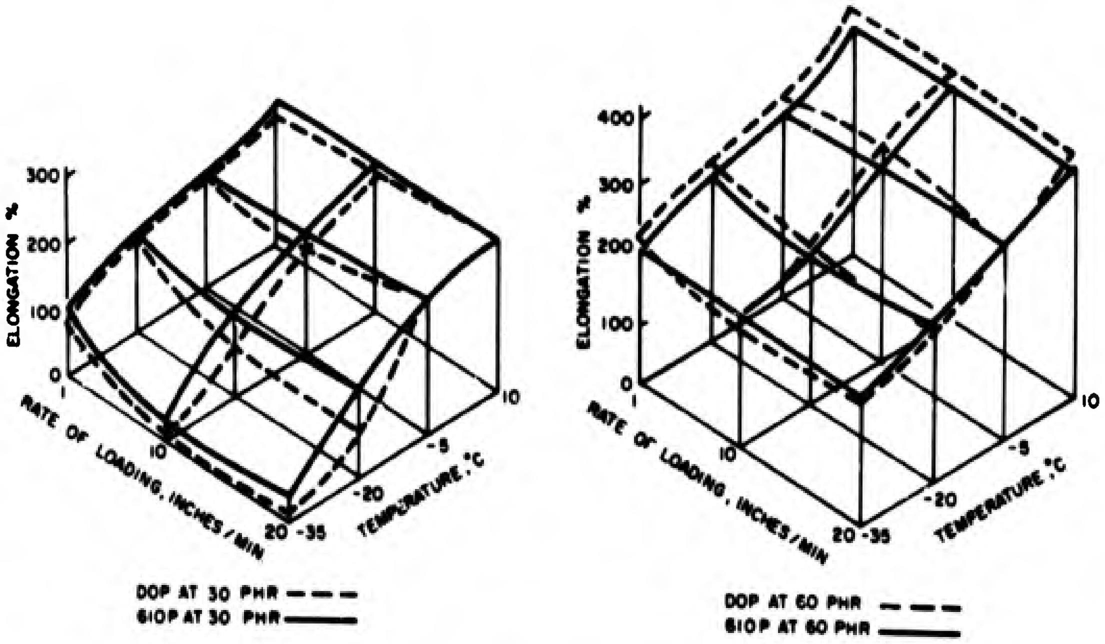


Figure 136. Percent elongation of plasticized PVC resin (64)

The changes with rate of loading and temperature are greater for the 30 phr than with 60 phr. The 30 phr graph shows the 610P plasticizer has greater elongation than the DOP over the complete range of conditions. This is also true for 40 and 50 phr. (See Table 41.) The 610P and DOP change positions over the range of conditions in the 60 phr graph. This indicates that the difference in plasticizers may well be masked when the plasticizer content of the formulation is relatively high.

The per cent elongation at two temperatures versus rate of loading and plasticizer content is shown in Figure 137.

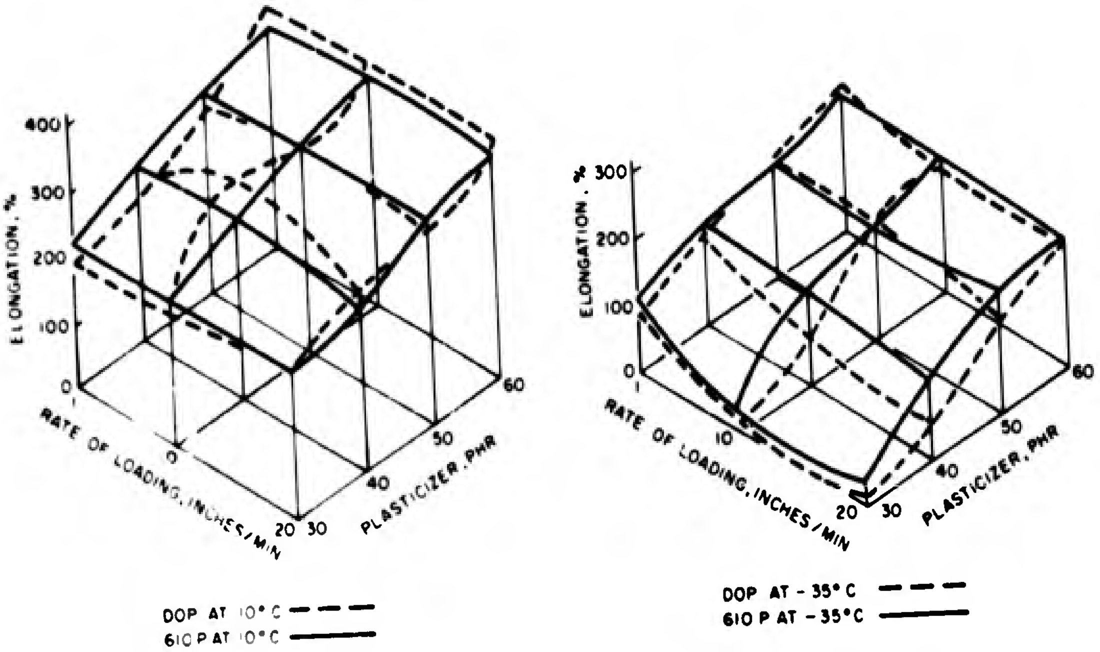


Figure 137. Percent elongation of plasticized PVC resin at 10 and -35° C. (64)

The -35° C graph shows a marked increase in elongation as the plasticizer is raised. The decrease in elongation with increasing loading rate is greater than at 10° C.

It should be noted that the 30 phr graph in Figure 136 has the approximate shape and magnitude of the -35° C graph in Figure 137. This also applies for the 60 phr graph in Figure 136 and the 10° C graph in Figure 137. This indicates that plasticized PVC behaves the same when the temperature is lowered as it does when the amount of plasticizer is decreased (64).

Figure 138 plots the 100 per cent modulus as a function of loading rate and temperature for 30 and 60 phr of DOP and 610P plasticized vinyls. From this graph, 610P is the more efficient plasticizer since it has lower modulus value than DOP and more of the plane is shown in the 30 phr graph than the DOP. The relatively high level of 60 phr of plasticizer, however, masks the difference between the two.

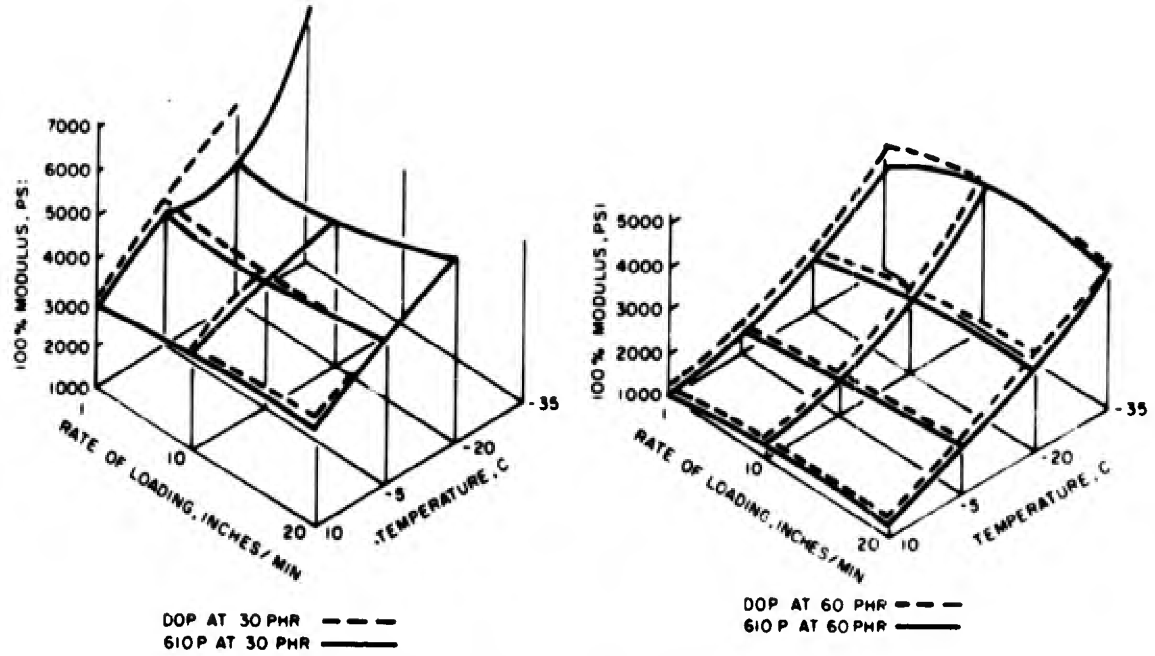


Figure 138. Modulus of plasticized PVC resin (64)

Note: The incomplete planes reflect a failure of the sample to elongate 100% before tensile failure.

Figure 139 shows the modulus of DOP and 610P at 10 and -35°C as a function of plasticizer phr and loading rate.

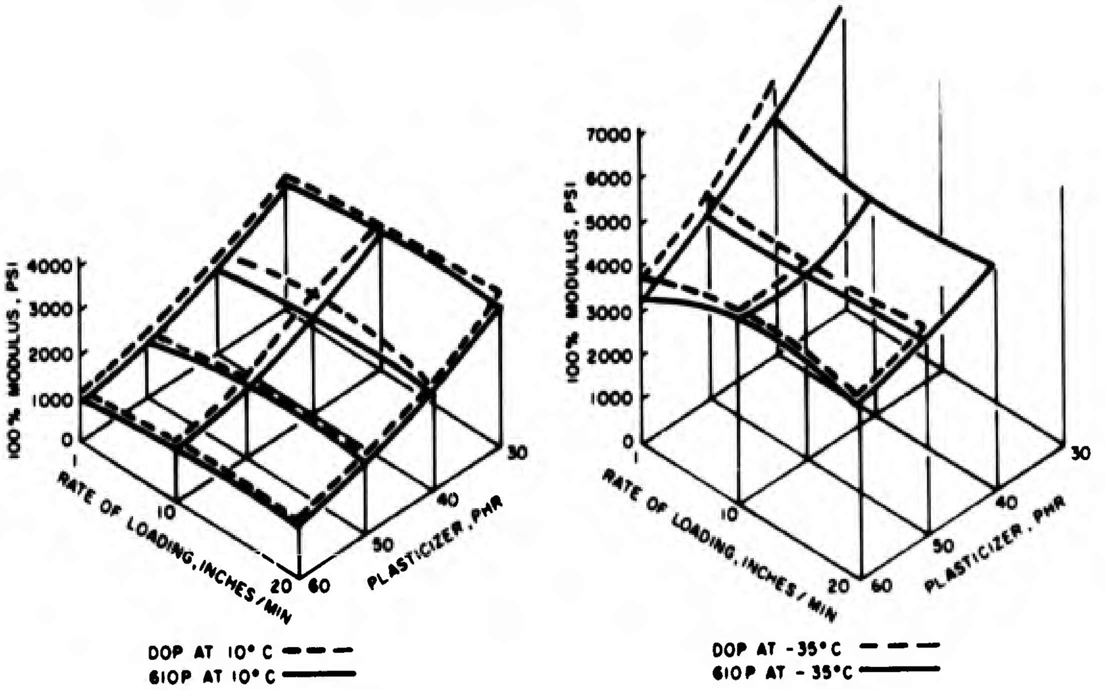


Figure 139. Modulus of plasticized PVC resin at 10 and -35°C (64)

As can be seen, the 10° C graph does not show the difference found in the -35° C plot. This illustrates the advantages of testing plasticizers at different temperatures. Like Figures 136 and 137 the same conclusions as for elongation can be drawn; namely, the 100 percent modulus of plasticized PVC behaves the same when temperature is lowered as when plasticizer content is decreased. This is evidenced by the similarity of 30 phr graph in Figure 138 with the -35° C temperature plot in Figure 139; and the 60 phr and 10° C as well.

The stress-strain curves are shown in Figure 140 for a 40 phr plasticized composition at four temperatures and three loading rates. From this data the following observations were made.

- Lowering temperature and increasing rate of loading have the same effect. The curve at -5° C and at a loading rate of 20 inches per minute is the same as that of -20° C at a loading rate of 1 inch per minute. Therefore, a 20 fold increase in loading rate is equivalent to a 15° C reduction in temperature on the tensile properties of this material.

- Decreasing temperature and increasing the rate of loading changes the curves from soft and weak, to soft and tough, to hard and tough, to hard and strong, to hard and brittle.

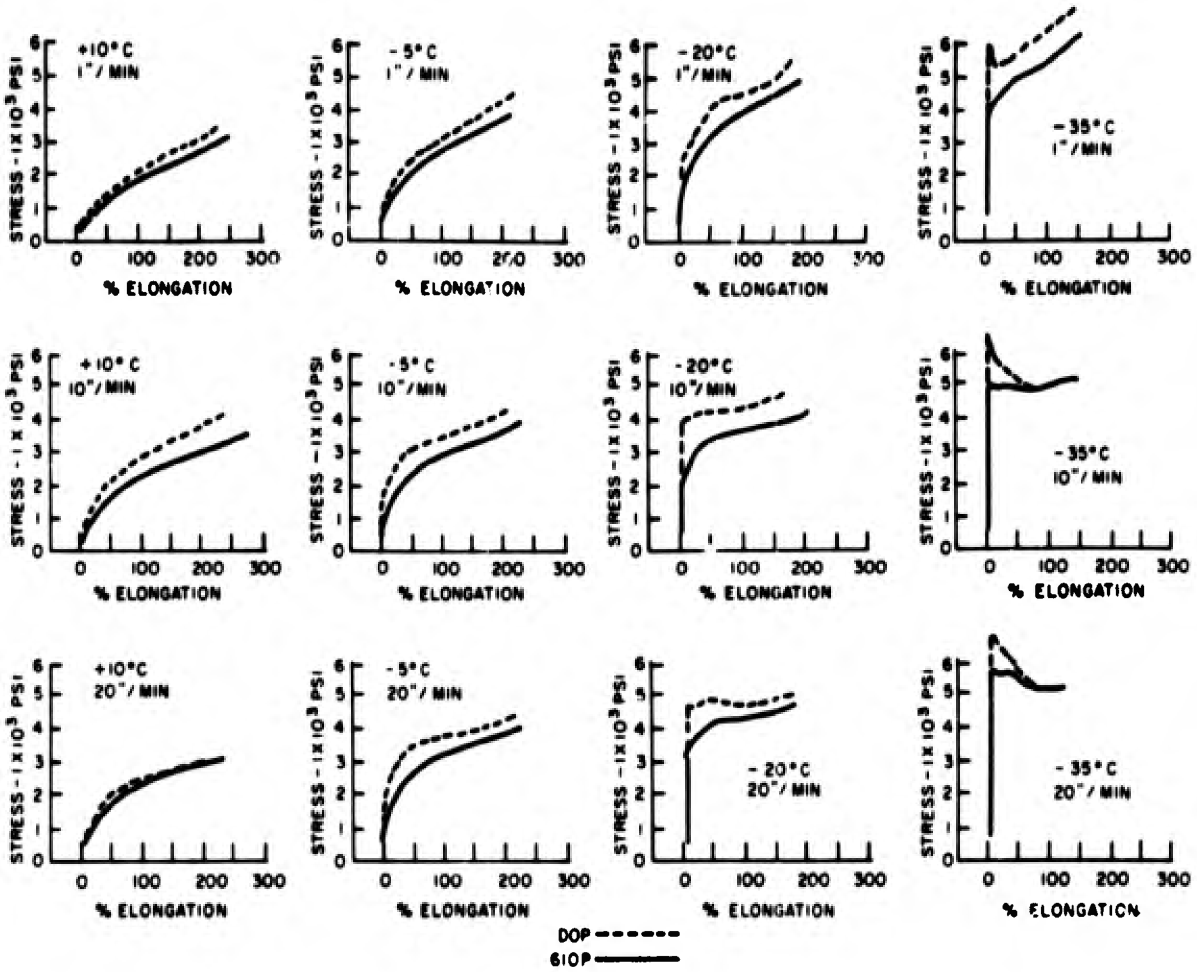


Figure 140. Stress strain curve - 40 phr plasticized PVC resin (64)

610P is a more efficient plasticizer than DOP as evidenced by comparing the DOP curve at -20 ° C and 10 inches per minute with the 610P at -35 ° C and 1 inch per minute loading rate.

The area under the stress-strain curve (a measure of toughness) was determined from the weight of the chart paper encompassed by the curve relative to the weight of a known area of the same paper. Results are given in Table 42.

TABLE 42. AREA UNDER STRESS-STRAIN CURVE (IN SQUARE INCHES) (64)

Temp °C	Rate of Loading	DOP Plasticizer				610P Plasticizer			
		30 phr	40 phr	50 phr	60 phr	30 phr	40 phr	50 phr	60 phr
10	1"/min	10.89	9.74	10.02	11.16	13.08	11.80	9.89	9.27
-5	1"/min	13.64	13.06	11.17	11.25	12.10	11.58	9.72	9.82
-20	1"/min	13.93	14.44	13.91	13.40	12.82	14.41	11.28	11.42
-35	1"/min	13.68	14.38	15.18	15.91	13.59	14.35	11.79	12.22
10	10"/min	12.66	13.07	13.38	13.45	13.94	13.42	12.69	11.33
-5	10"/min	13.39	13.42	13.52	14.05	13.69	13.19	11.31	11.59
-20	10"/min	9.09	13.66	13.82	13.07	11.71	13.92	11.11	13.80
-35	10"/min	1.84	7.88	14.36	13.31	4.51	14.09	11.83	14.05
10	20"/min	11.74	8.95	11.94	12.72	12.91	10.80	13.07	11.74
-5	20"/min	14.42	15.13	14.64	13.70	13.93	14.81	15.12	13.53
-20	20"/min	7.29	15.44	15.38	13.86	11.10	14.50	14.83	12.99
-35	20"/min	2.24	8.10	13.21	14.81	5.39	12.63	16.65	14.31

There is little change in toughness at: 1 in./min. and 30 phr plasticizer at all temperatures; 10°C over the different loading rates; and at various temperatures and loading rates for 60 phr. However, there is a sharp decrease in toughness as the loading rate is raised from 1 to 20 in./min. and at temperatures below -5°C.

From the above data it is obvious that the difference in tensile properties of plasticized compositions are greater at lower temperatures and plasticizer levels and that 610P is a more efficient plasticizer than DOP.

Figure 141 plots the tensile and flexural properties of a vinyl chloride molding copolymer.

Figure 142 plots the flexural stiffness of an elastomeric electrical grade polyvinyl chloride compound at various low temperatures.

Table 43 shows the effect of various low temperature plasticizers on polyvinyl chloride stiffness.

The effectiveness of low temperature plasticizers was determined by the Clash-Berg and brittleness temperature tests (65). Two points - T4, the temperature in ° C at which stiffness in flexure is 10,000 psi and Tf, the temperature in ° C at which stiffness in torsion is 135,000 psi - were reported in the Clash-Berg test. Results are given in Table 44.

Tests were also conducted on blends of plasticizers to determine any synergistic effects. Table 45 lists these results.

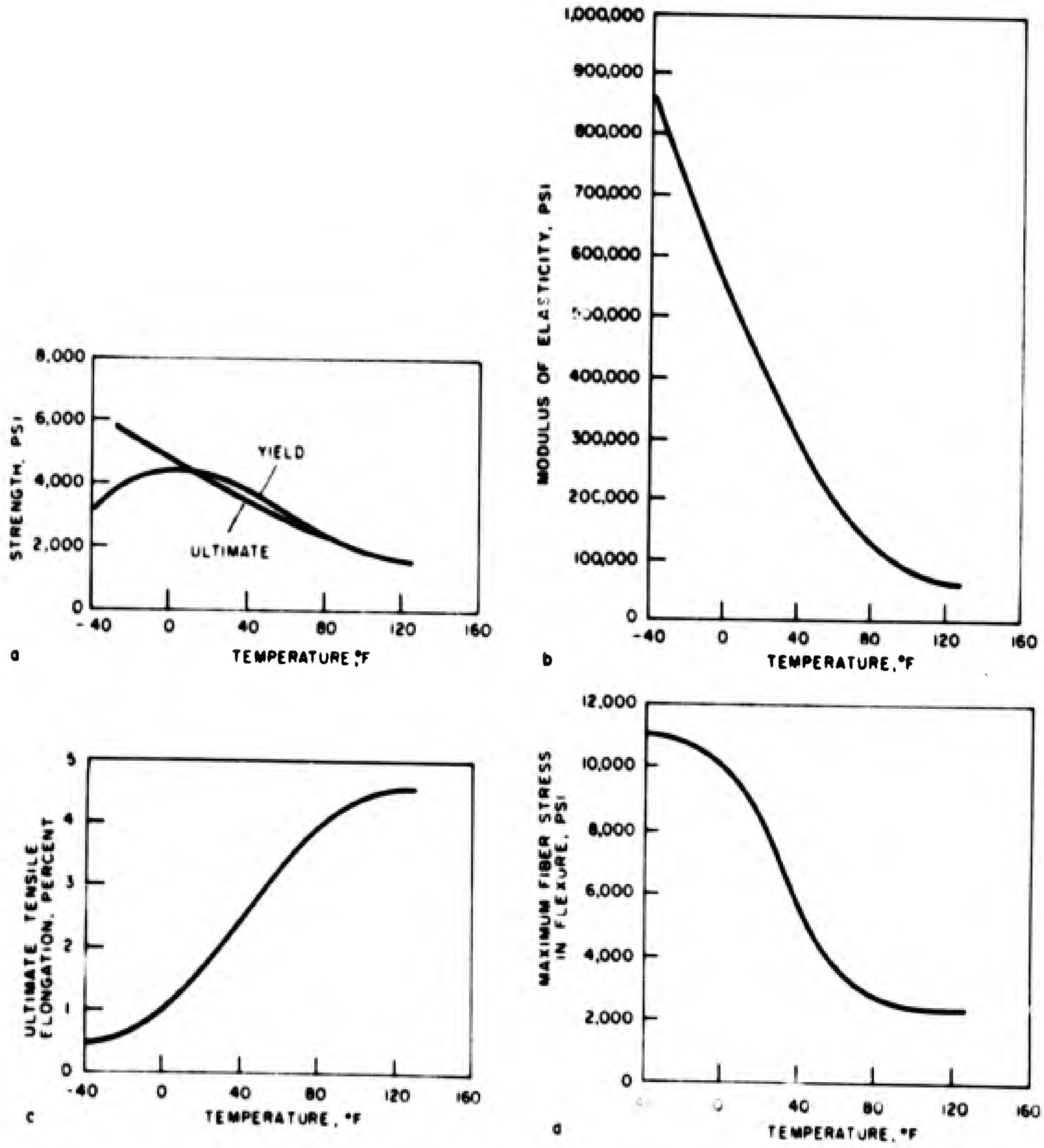


Figure 141. Tensile and flexural properties of (Saran 909) vinyl chloride copolymer (19)

TABLE 43. COMPARATIVE PERFORMANCE OF PLASTOLEIN LOW TEMPERATURE AND EPOXY PLASTICIZERS IN PVC (5)

	Plastolein 9051 DNHZ	Plastolein 9055 DGP	Plastolein 9057 DIOZ	Plastolein 9058 DOZ	Plastolein 9066 LT	Plastolein 9078 LT	Plastolein 9214 Epoxy	Plastolein 9232 Epoxy	Plastolein 9250 THFO	DOP
Plasticizer Concentration, phr	45	45	50	45	45	45	51*	54*	45	50
Clash & Berg T 10,000, °C	-8	-5	-5	-9	-3	-2	-2	+2	-4	+1
Clash & Berg T 45,000, °C	-28	-25	-32	-31	-26	-23	-20	-11	-22	-15
Clash & Berg T 135,000, °C	-49	-43	-52	-49	-45	-41	-36	-23	-43	-28

* As a 50 50 blend with DOP.

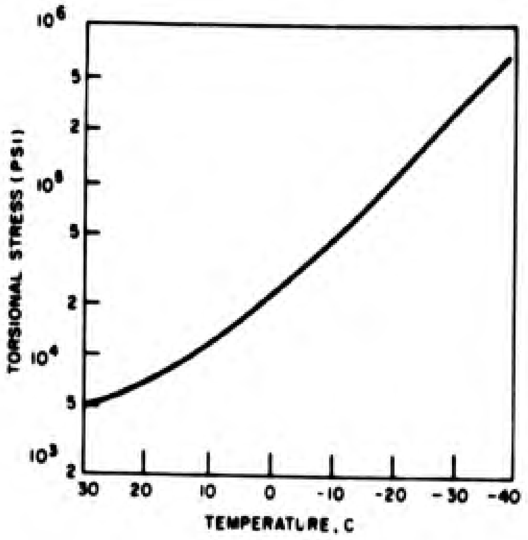


Figure 142. Flexural stiffness vs temperature for elastomeric vinyl compound (38)

TABLE 44. EFFECTIVENESS OF LOW TEMPERATURE PLASTICIZERS (65)

Code	T_B	T_4	T_f	Code	T_B	T_4	T_f
A-10	-42	-17	-39	A-30	-53	-19	-44
B-10	-42	-17	-40	B-30	-50	-17	-42
C-10	-38	-17	-40	C-30	-49	-19	-45
D-10	-44	-17	-34	D-30	-52	-18	-46
E-10	-38	-16	-31	E-30	-48	-19	-38
F-10	-36	-17	-31	F-30	-46	-19	-39
A-20	-46	-19	-43	A-40	-54	-18	-43
B-20	-44	-17	-40	B-40	-52	-18	-45
C-20	-46	-18	-41	C-40	-58	-20	-46
D-20	-50	-18	-40	D-40	-53	-20	-52
E-20	-45	-18	-35	E-40	-48	-21	-42
F-20	-41	-18	-35	F-40	-44	-20	-43

Formulation
PVC Resin - 100
Di-2-ethylhexyl phthalate) 57
Low temperature plasticizer)
Epoxy stabilizer - 3
Ba-Cd-Zn stabilizer - 3

Code:
A = di-2-ethylhexyl adipate
B = diisodecyl adipate
C = di-2-ethylhexyl azelate
D = tri-2-ethylhexyl phosphate
E = 2-ethylhexyl epoxytallate
F = epoxy ester

TABLE 45. EFFECTIVENESS OF BLENDS OF LOW TEMPERATURE PLASTICIZERS (65)

Code	T_B	T_4	T_f	Code	T_B	T_4	T_f
A-1	-39	-13	-40	A-3	-45	-17	-46
B-1	-40	-14	-41	B-3	-48	-17	-47
C-1	-40	-14	-40	C-3	-47	-17	-47
D-1	-39	-14	-40	D-3	-46	-16	-46
E-1	-39	-14	-40	E-3	-45	-18	-47
F-1	-40	-13	-41	F-3	-48	-18	-46
A-2	-42	-15	-42				
B-2	-45	-15	-44				
C-2	-44	-15	-46				
D-2	-43	-15	-44				
E-2	-43	-16	-45				
F-2	-45	-15	-43				

Formulation	1	2	3
PVC Resin	100	100	100
Di-2-ethylhexyl phthalate	42	33	27
Low temperature plasticizer	15	24	30
Epoxy plasticizer	3	3	3
Ba-Cd-Zn stabilizer	3	3	3

Code:
A = di-2-ethylhexyl adipate/diisodecyl adipate
B = di-2-ethylhexyl adipate/tri-2-ethylhexyl phosphate
C = di-2-ethylhexyl adipate/di-2-ethylhexyl azelate
D = diisodecyl adipate/tri-2-ethylhexyl phosphate
E = diisodecyl adipate/di-2-ethylhexyl azelate
F = tri-2-ethylhexyl phosphate/di-2-ethylhexyl azelate

As can be seen, no synergism exists; the low temperature performance of all systems tested were equivalent. Therefore, the secondary effects such as efficiency, volatility, oil extraction, processability, temperature performance range, fungus resistance or flame retardancy must determine the selection of the plasticized compound.

Table 46 gives the physical properties of vinyl compounds made with different low temperature plasticizers at equal efficiency (80 Duometer "A" hardness) compared to a compound plasticized with di-2-ethylhexyl phthalate.

The effects of various secondary hydrocarbon plasticizers used for improving low temperature flexibility of plasticized polyvinyl chloride were studied (66). Formulations were prepared using secondaries as 25 percent replacements for DOP. The aromatic plasticizer (Code I) was evaluated throughout its complete range of compatibility. Results of the low temperature flexibility test (ASTM D736-54T) are shown in Table 47.

TABLE 46. PHYSICAL PROPERTIES OF VARIOUS PLASTICIZED VINYL COMPOUNDS AT "80A" HARDNESS (65)

	Di-2-ethylhexyl Phthalate	Di-2-ethylhexyl Adipate	Diisodecyl Adipate	Di-2-ethylhexyl Azelate	Tri-2-ethylhexyl Phosphate	n-Octyl, n-decyl Phthalate	2-Ethylhexyl Epoxy tallate	Epoxy Ester
PHR	48	40	52	46	47	52	50	40
"A"	80	80	80	80	80	80	80	80
T_4	+9	+2	0	-4	-4	+3	-6	+2
T_f	-20	-37	-48	-46	-44	-36	-37	-36
T_B	-22	-48	-50	-48	-55	-35	-42	-37
Volatility, % 24 hrs 70° C, 20 mil	1.0	2.4	0.6	0.8	1.6	0.5	0.6	1.2
Oil extraction K, 50°C*	1.6	4.8	9.9	14.0	7.0	5.6	6.9	5.7
MET**	106	120	148	141	110	110	125	114

* $K = \frac{\text{Wt Loss}}{a\sqrt{t}}$

** Minimum fluxing temperature

TABLE 47. LOW-TEMPERATURE FLEXIBILITY WITH SECONDARY HYDROCARBON PLASTICIZERS (50 PHR TOTAL PLASTICIZER)(66)

DOP Primary Plasticizer, %	Secondary Plasticizer %	Secondary Plasticizer Code*	Rating at ** 0° F.	Rating at ** -20° F.	Rating at ** -40° F.
100	-	-	P	P	P
75	25	A	P	P	F
75	25	B	F		
75	25	C	F		
75	25	D	P	F	
75	25	E	P	P	F
75	25	F	P	P	F
75	25	G	P	P	P
75	25	H	P	F	
75	25	I	P	F	
50	20	I	P	F	
25	75	I	F		
0	100	I	F		
75	25	J	P	P	P

** P = Pass; F = Fail

* Code: Typical secondary hydrocarbon plasticizers for vinyl resins:

Chemical Type	Letter Code	Specific Gravity	Viscosity, CPS. 100° F.	Viscosity, CPS. 210° F.	Approx. Molecular Weight
Special petroleum fraction	A	0.8899	8.89	2.33	267
Aromatic hydrocarbon	B	0.9786	----	20.7	5-8
Aromatic hydrocarbon	C	1.0140	143	6.8	270 avg.
Alkyl-aryl hydrocarbon	D	0.878	17.9	3.33	280
Partially hydrogenated alkyl-aryl hydrocarbon	E	0.970	9.38	----	200
Partially hydrogenated terphenyl	F	1.001	70*	----	237
Alkylated naphthalene	G	0.944	49.7	4.20	240
Aromatic hydrocarbon extender	H	1.02	92.6	5.74	275
Very aromatic hydrocarbon**	I	1.09	11.85	2.31	235
Chlorinated paraffin	J	1.15	17.4-21.6*	----	570

* At 77° F.

** Experimental at present.

The low temperature resistance of plasticized film was determined by bending a film specimen cooled from -20 to -60° C through 180°. (67)

Four specimens 150 x 100 mm were cut from each type of film in the horizontal and transverse directions. The film and a Kg weight used for stressing (bending through 180°) were cooled for 10 minutes. If a crack appeared at the point of bending, the specimen was not considered low temperature resistant.

The results in Table 48 show that transverse specimens break at higher temperatures. This is due to the orientation of molecular chains during processing in the longitudinal direction. As a result, it is necessary to break many more chemical bonds in the longitudinal direction than in the transverse.

TABLE 48. LOW TEMPERATURE RESISTANCE OF PLASTICIZED PVC FILM (67)

Plasticizer	Per Cent Plasticizer	Orientation Direction	Failure Temperature
Dibutyl phthalate	45	transverse	-42° C
Dibutyl phthalate Phthalic esters	12.5 12.5	transverse longitudinal	-20° C -30° C
Dibutyl phthalate Phthalic esters	22.5 22.5	transverse longitudinal	-30° C -50° C
Dibutyl phthalate Phthalic esters	27.5 27.5	transverse longitudinal	-40° C -50° C

Like the polycarbonates, rigid polyvinyl chlorides have a sharp transition from tough to brittle fracture in notched impact tests as the temperature is lowered. This change in fracture, as illustrated in Figure 143, is accompanied by a near ten-fold decrease in Izod value.

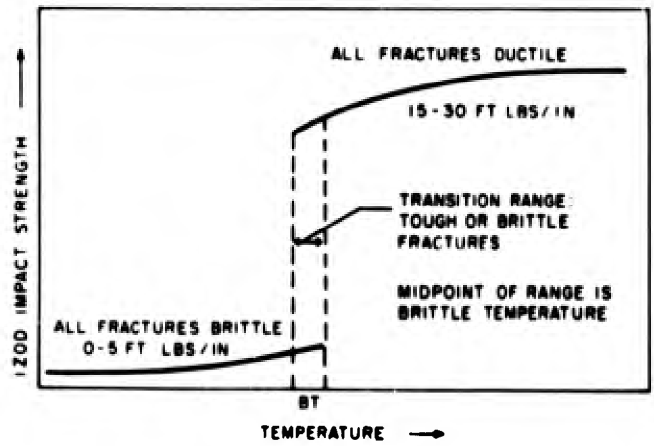


Figure 143. Notched izod response of rigid polyvinyl chloride (35)

Below a certain temperature (the brittle temperature) all specimens fail in a brittle manner with no yielding. Izod strengths range from 0 to about 5 lbs/in. Above this temperature fractures are ductile with yielding, and Izod values are from 15 - 30 lb/in. In other words, specimens fail in a tough or brittle manner; no intermediate types are observed. (35)

This transition in Izod response of polyvinyl chloride is simply a change in response. It is a sudden manifestation of a gradual change in properties and is not associated with fundamental transitions such as the glass transition or crystallization or phase change phenomena.

The impact strength of vinyls, as with other thermoplastics, are influenced by many factors in addition to temperature as previously stated. These have been discussed in previous sections and will only be mentioned here. Such factors are:

- Composition - effects of plasticizers, stabilizers, pigments, copolymers, alloys, etc.
- Processing - time, temperature, work history, method of formulating, orientation, etc.
- Thickness of specimen - notched impact strength decreases with increased thickness.
- Increasing loading rate.

The impact strengths of an acrylic-polyvinyl chloride alloy (Kydex 100) are given in Table 49 and a rigid polyvinyl chloride molding material in Table 50.

TABLE 49. IMPACT STRENGTH OF AN ACRYLIC-PVC ALLOY (KYDEX 100) (68)

Impact Test	Test Method	Impact Strength ft/lbs
Charpy Unnotched (ft. lbs./1/2" x 1" section)	ASTM D-256-56 (Modified to 2" span)	
73° F.		118
32° F.		80
0° F.		35
-20° F.		26
-40° F.		23
-65° F.		14
Falling Dart (ft. lbs.) * 2 lb. dart with 1/8" radius	R & H P-24F	
73° F.		> 36 (Indented - no break)
48° F.		> 36 (Indented - no break)
25° F.		> 30 (Indented - no break)
0° F.		12.0
-20° F.		4.4
-40° F.		4.0
-65° F.		3.2

* Sample .125" thick.

TABLE 50. IZOD IMPACT OF RIGID PVC MOLDING COMPOUNDS (ASTM 256) (69)

	Geon Vinyl ASTM Type I Grade I Normal Impact	Geon Vinyl ASTM Type II Grade I Normal Impact	Geon Vinyl ASTM Type II Grade I High Impact Trans	Geon Vinyl ASTM Type I Grade II Normal Impact Trans	Hi-temp Geon Vinyl 88876	Hi-temp Geon Vinyl 88861
Izod impact, ft.lb./in.notch						
at 73° F.	.80	15.0	13.0	.70	3.5	1.4
at 32° F.	.70	1.7	5.0	.65	---	---
at -10° F.	.55	1.0	.90	.55	---	---
at -40° F.	---	----	-----	---	0.5	0.5

Electrical Properties

The effect of plasticizer content and temperature on dielectric constant is shown in Figure 144. The power factor versus temperature for a plasticized PVC at 1000 c/s is plotted in Figure 145.

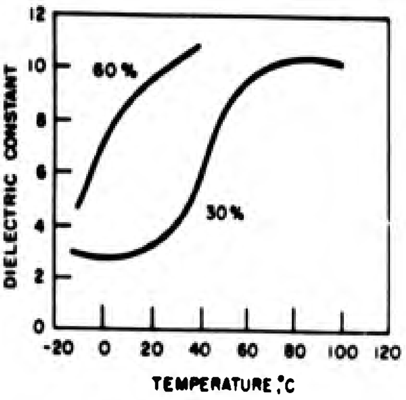


Figure 144. Effect of temperature and plasticizer content on the dielectric constant of PVC at 60 c/s (16)

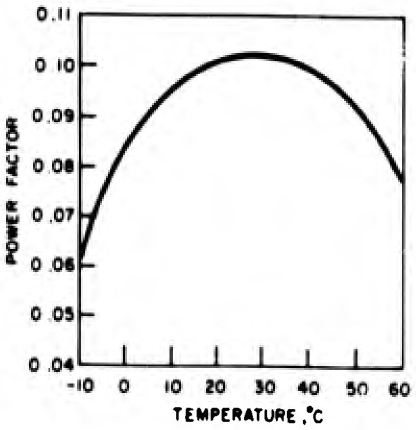


Figure 145. Power factor vs temperature for PVC compound containing 50 parts Bisoflex 791 per 100 parts polymer. Frequency = 1000 c/s (16)

Thermal Properties

Tables 51, 52 and 53 give some representative brittle temperatures of various plasticized compounds.

TABLE 51. COMPARATIVE PERFORMANCE OF (PLASTOLEIN) POLYMERIC PLASTICIZERS IN PVC (5)

Plastolein No.	Concentration (phr)	Brittle Point, ASTM D 746 (° C)	Masland Impact 20 Mil Sheet (° C)
9717	56	-32	-25
9720	56	-27	-25
9722	60	-23	-20
9730	70	-16	-10
9750	65	-23	-15
9765	68	-17	-10
9789	68	-22	-15
9051 (DNHZ)	45	-60	-55
9055 (DGP)	45	-60	-50
9057 (DIOZ)	50	-60	-50
9058 (DOZ)	45	-60	-55
9066 (LT)	45	-60	-55
9078 (LT)	45	-57	-50
9214 (Epoxy)	51*	-47	-35
9232 (Epoxy)	54*	-37	-25
9250 (THFO)	45	-52	-40
-- (DOP)	50	-29	-30

* Di-2-ethylhexyl phthalate used as control.

TABLE 52. BRITTLENESS TEMPERATURE OF (GEON) COMPOUNDS (5)

Geon Vinyl Specific Gravity	Brittle Temperature (ASTM D 746) (° F)
1.19	-50
1.27	-20
1.32	-10
1.34	0
1.39	15
1.48	-20 (Method A)
1.56	0 (Method A)
1.58	+20 (Method A)

TABLE 53. BRITTLENESS TEMPERATURE OF (BAKELITE) VINYLS FOR CONTOUR EXTRUSION (70)

Vinyl	Brittleness Temperature 50% Non-failure (ASTM D 746) (° C)
VFD-9914, Clear 111	-44
VFD-9918, Clear 221	-26
VFD-9411, Clear 181 Nat.	-20
VFDA-9932, Clear 111, Nat.	-16
VFD-9410, Clear 111	-30
VFD-9960, Nat.	-39
VFD-9970, Nat.	-32
VFD-9020, Nat. and Colors	-12
QFDA-9021, Nat. and Colors	-16
VFD-9980, Nat.	-22
VFD-9990, Nat.	-15
VFD-9953, Nat. and Colors	-36
VFD-9962, Nat. and Colors	-36
VFD-9963, Nat. and Colors	-32
QFDA-9121, Nat.	-44
VFDA-9973, Nat. and Colors	-28
VFD-9532, Nat. and Colors	-23
QFDB-9983, Nat. and Colors	-10

PART II. THERMOSETS

SECTION XV. EPOXY COMPOUNDS

Properties of epoxies at low temperature are influenced by many factors: the epoxy resin, addition of reactive diluents (flexibilizers), hardeners and curing temperature and time as well as reinforcements and fillers.

Mechanical Properties

Table 54 gives tensile property data for various epoxy resin cast systems at -25° C.

TABLE 54. TENSILE STRENGTH PROPERTIES OF (EPON) EPOXY RESIN CAST SYSTEMS AT -25° C. (71)

EPON 828/ EPON 871 Ratio	Curing Agent phr - based on resin blend	Tensile Properties at -25° C (Ultimate)				
		Unaged		Aged 3 days at 180°F		Loading Rate "/min
		K psi	Elong. %	K psi	Elong. %	
0/100	AEP - 10.8 (1)	4.9	50	3.3	62	2
20/80	AEP - 13.2 (1)	7.1	13	7.8	24	2
40/60	AEP - 15.5 (1)	6.1	3	11.0	8	.05
60/40	AEP - 18.0 (1)	13.1	6	11.7	4	.05
80/20	AEP - 20.2 (1)	10.8	3	11.7	4	.05
100/0	AEP - 22.6 (1)	14.2	8	12.3	4	.05
0/100	DTA - 5.2 (1)	3.3	62	3.5	52	2
20/80	DTA - 6.3 (1)	7.0	7	6.3	3	2
40/60	DTA - 7.4 (1)	3.8	4	2.8	1	2
60/40	DTA - 8.4 (1)	4.9	1	7.0	2	.05
80/20	DTA - 9.7 (1)	12.6	4	7.3	2	.05
100/0	DTA - 10.8 (1)	13.6	4	12.2	3	.05
0/100	Agent Z - 10.0 (2)	5.3	41	5.2	27	2
20/80	Agent Z - 12.3 (2)	8.0	9	8.6	10	2
40/60	Agent Z - 14.5 (2)	10.6	6	10.7	5	.05
60/40	Agent Z - 16.3 (2)	9.2	2	6.0	1	.05
80/20	Agent Z - 18.9 (2)	9.9	2	12.1	3	.05
100/0	Agent Z - 21.1 (2)	14.0	5	12.2	3	.05
0/100	NMA (3) - 44.8 (4)	7.5	11	7.1	10	2
20/80	NMA - 54.6 (4)	7.3	3	---	--	.05
40/60	NMA - 64.3 (4)	10.8	4	10.5	3	.05
60/40	NMA - 72.5 (4)	10.8	3	8.4	2	.05
80/20	NMA - 84.0 (4)	11.1	3	10.4	2	.05
100/0	NMA - 93.8 (4)	7.1	1	9.6	2	.05

(1) Cure, 4 hrs at 125° C.
(2) Cure, 2 hrs at 100° C + 3 hrs at 150° C.
(3) Nadic methyl anhydride; 1% wt. Benzyl dimethylamine used as accelerator in all cases.
(4) Cure, 4 hrs at 120° C + 4 hrs at 200° C.

Note:
AEP = N-aminoethylpiperazine
DTA = Diethylene triamine
Agent Z = Aromatic amine

Tables 55 and 56 present the mechanical properties of an epoxy-glass reinforced molding compound over our interested temperature range.

TABLE 55. MECHANICAL PROPERTIES VERSUS TEMPERATURE OF (SCOTCHPLY TYPE 1100) EPOXY MOLDING COMPOUND (EPOXY-FIBERGLASS 1/2" FIBER LENGTH) (72)

Property	Units	Temperature ° F.		
		-65	70	200
Tensile Strength	psi x 10^3	31	30	18
Flexural Strength	psi x 10^3	80	62	42
Flexural Modulus	psi x 10^6	3.4	3.3	2.3

TABLE 56. MECHANICAL PROPERTIES OF EPOXY LAMINATES, (SCOTCHPLY NONWOVEN E GLASS) (ASTM LP-4066, SPECIMEN CONDITIONED 1/2 HR. AT TEST TEMPERATURES)(73, 74, 75)

Scotchply	Temp ° F.	Flexural Strength (K psi)			Modulus In Flexure (psi x 10^6)			Tensile Strength (K psi)			Compression Strength Edge (K psi)		
		U (1)	C (2)	I (3)	U	C	I	U	C	I	U	C	I
#1000	-67	154	112	85	4.8	3.5	---	105	85	63	80	62	67
	70	113	90	66	4.7	3.0	2.7	98	57	50	73	54	45
	160	90	75	55	3.6	2.6	2.3	89	52	37	58	43	30
#1002	-60	192	137	104	5.4	3.6	2.8	172	92	69	115	82	70
	70	165	120	80	5.3	3.5	3.0	160	75	57	90	75	60
	160	128	104	70	5.0	3.3	2.7	139	62	51	70	66	52
#1007 (4)	-60	179	129	---	6.7	3.4	---	---	--	--	85	78	--
	70 (5)	150	107	---	5.0	3.2	---	---	--	--	80	73	--
	70 (6)	140	87	---	4.9	3.1	---	---	--	--	67	63	--
	300	110	70	---	4.7	2.9	---	---	--	--	55	50	--

(1) U = Unidirectional; stress angle 0°
(2) C = Crossplied; stress angle 0° - 90°
(3) I = Isotropic; stress angle 0°, 60° or 120°
(4) High temperature epoxy/E glass laminate; cured as follows:

Thickness	Press Time at 400° F	Oven Post Cure at 400° F
1/8"	30 min.	16 hrs.
1/4"	35 min.	16 hrs.
1/2"	40 min.	16 hrs.
1"	50 min.	16 hrs.

(5) Dry
(6) 2-hr. boil

Table 57 shows the strain of epoxy resins in resin-copper laminates at various temperatures. From this it can readily be seen that the highest strains occur under low-temperature conditions rather than under the high temperature conditions. Therefore, to prevent cracking, the resin must possess sufficient elongation to withstand these strains. To determine the maximum strain allowable, the lowest and highest service temperatures desired and the differences in the coefficient of thermal expansion of the different materials must be known.

TABLE 57. STRAINS OF EPOXY RESINS IN RESIN-COPPER LAMINATES (76)

	at 125 ° C	at 25 ° C	at -25 ° C	at +225 ° C
Dimensional Changes of a Copper Bar, mil/inch	±0	-1.7	-2.5	+1.7
Dimensional Changes of an Epoxy Resin Bar, mil/inch	±0	-5.0	-7.5	+5.0
Strain of an Epoxy Resin in a Resin-Copper Laminate per thousand	±0	-3.3	-5.0	+3.3

Flexural tests were conducted on various epoxy compounds to determine the effect that the epoxy resins, flexibilizers and curing agents had on low temperature performance (76). Flexural instead of tensile tests were made on epoxy bars 1/2" x 1/2" x 4-1/2" to assure an accuracy of 0.5 percent elongation.

The results in Table 58 indicate that the flexible resin Epon 872 has excellent elasticity at room temperature and -40 ° C, but after heat aging at 200 ° C

TABLE 58. FLEXURAL PROPERTIES OF (BAKELITE ERL 2256, EPOTUF ED-1025 AND EPON 872) EPOXIDE RESINS (76)

Formulation	Heat Dist. Temperature (ASTM D-648) ° C	Weight Loss %	Flexural Properties of Test Bars					
			at Room Temperature			at -40°C		
			Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi	Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi
100 ERL 2256 112 HHPA (1) 0.4 BDMA (2)	102 After aging 150 hrs at 200 ° C	6.4	3.2 2.7	13 10	420 390	1.3 0.9	6.0 4.0	400 410
100 ERL 2256 34 MDA (3)	152 After aging 150 hrs. at 200 ° C	8.7	>3.5 1.4	>13 6.5	420 400	2.2 1.5	13.0 7.0	400 470
100 ERL 2256 17 MPDA (4)	157 After aging 150 hrs. at 200 ° C	10.1	>3.5 1.1	>15.5 5.5	490 490	2.1 0.7	11.0 3.6	540 510
100 ED-1025 75 HHPA 1 BDMA	82 After aging 165 hrs. at 200 ° C	2.2	>3.5 >3.5	>12.5 >12.3	370 380	3.0 >3.5	14.5 >13.5	430 400
100 Epon 872 21.5 HHPA 1 BDMA	44 After aging 80 hrs. at 200 ° C After aging 285 hrs. at 200 ° C	2.9	>3.5 3.6 3.0	>7.5 7.5 6.5	240 230 220	>3.5	>10.5	300
100 Epon 872 3.78 MPDA	35 After aging 80 hrs. at 200 ° C After aging 285 hrs. at 200 ° C	3.9	>3.5 2.2 1.7	>4.0 3.0 2.5	120 130 100	>3.5	>10.0	300

(1) Hexahydrophthalic anhydride
(2) Benzyldimethylamine
(3) Methylene dianiline
(4) m-phenylene diamine

this drops off rapidly. The very low viscosity resin, ERL-2256, is very fragile at -40 ° C and deteriorates on aging. Of the formulations tested, Epotuf ED-1025 cured with HHPA is superior. It remains elastic at -40 ° C even after extended heat aging.

Table 59 summarizes the effects of different epoxy resins on flexibility.

TABLE 59. EFFECT ON FLEXIBILITY OF CURED EPOXY RESINS WHEN THE BIS-PHENOL A DIEPOXIDE RESIN IS SUBSTITUTED BY OTHER EPOXIDES (76)

Flexible resins	Better at RT and -40 ° C, but thermally less stable
Very low viscosity resins	More brittle at -40 ° C. Thermally less stable
Novolac epoxide resins	Higher rigidity. Thermally more stable
Resorcinol epoxide resins	Better at RT and at -40 ° C. Thermally more stable
Cyclohexene epoxide resins and epoxidized polyolefins	Less flexible at RT and -40 ° C. Thermally less stable

The effect of flexibilizers on low-temperature flexibility is shown in Table 60. The flexibility of DP-437 and DP-125 test bars is limited to room temperature. At -40 ° C they are stiffer than conventional bis-phenol A epoxide resins. Modifiers X-2673.2 and X-2674 fail to increase elongation at -40 ° C. It was reported that initially the dimer or trimer acid-cured resins have satisfactory low-temperature properties. However, after prolonged aging, embrittlement takes place resulting in surface cracking when tested.

Table 61 compares the flexibility of epoxy compounds cured with polysebacic anhydride and aliphatic dicarboxylic acids. As can be seen, use of the polysebacic anhydride curing agent results in better low temperature flexibility and stability after heat aging. Good results are also obtained with the less expensive aliphatic dicarboxylic acids. As a result of its performance, availability and low melting point (108 ° C), azelaic acid is preferred. (76)

It was also found that good flexibility could be obtained when the amount of dicarboxylic acid was reduced and caprolactam was added. Caprolactam acts as an accelerator and lowers the freezing point and the viscosity of the resin mixture. Results are given in Table 62.

Stresses in casting resins were evaluated at various temperatures by means of strain gauges. (77) Three gauges were mounted in a stainless steel sensing element tube both circumferentially and axially. Two of the gauges were mounted around the circumference 180 ° apart in the center of the tube and the other gauge was positioned between these two in the direction of the tube axis. The tube was calibrated for temperature and pressure and placed in a specially prepared Teflon mold. The casting resins were poured in around the sensing element and up to the top level of the stainless steel tube. Two thermocouples were embedded in the

TABLE 60. FLEXURAL PROPERTIES OF RESIN COMPOUNDS CONTAINING "FLEXIBILIZING" ADDITIVES (76)

Formulation	Heat Dist. Temperature (ASTM D-648) °C	Weight Loss %	Flexural Properties of Test Bars					
			at Room Temperature			at -40°C		
			Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi	Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi
50 Epon 828 50 DP-437 13 DP-125	35	decomposed	>3.5	>2.8	100	2.1	10.0	500
					not recommended for high temperature applications			
25 Epon 828 75 DP-137 11 DP-125	<RT	decomposed	—	flexible	—	1.3	6.0	450
					not recommended for high temperature applications			
100 DEN 438 27.2 MDA	177 After aging 80 hrs. at 200° C	5.6	2.7 2.3	9.5 8.0	350 375	2.8 —	10.5 —	380 —
70 DEN 438 30 X-2673.2 23.4 MDA	108 After aging 80 hrs. at 200° C	12.3	2.5 1.3	7.5 5.0	350 390	1.5 —	6.0 —	410 —
100 DEN 438 86 MNA (1) 1.5 DMP-30	174 After aging 80 hrs. at 200° C	1.3	1.9 1.7	8.0 7.0	400 400	2.2 —	9.0 —	400 —
70 DEN 438 30 X-2674 1.5 DMP-30	120 After aging 80 hrs. at 200° C	2.7	2.3 3.5	9.0 12.0	380 370	1.7 —	7.0 —	400 —
100 Epon 828 100 Leecure A 44 MNA 0.7 DMP-30	33 After aging 80 hrs. at 200° C After aging for 420 hrs. at 200°C	4.6	>3.5 3.4 3.4	>2.0 8.0 8.5	70 260 260	>3.5 — —	>10.5 — —	300 — —
100 Epon 828 100 Empol 3162-D 25 MNA 1 BDMA	40 After aging for 80 hrs. at 200°C After aging for 420 hrs. at 200°C	3.9	>3.5 2.3 1.7	>6.5 4.5 3.5	190 190 200	>3.5 — —	>9.0 — —	260 — —
100 Unox 201 145 Empol 3162-D	38 After aging for 80 hrs. at 200°C After aging for 420 hrs. at 200°C	5.9	>3.5 >3.5 >3.5	>4.0 >1.0 > .8	150 50 30	>3.5 surface cracking	>7.5 —	220 — —
100 Oxiron 2000 100 Empol 1014	<RT After aging for 80 hrs. at 200°C	2.2	— >3.5	flexible > .5	– ~12	>3.5 2.0	>7.0 4.0	210 195

(1) Methylnadic anhydride

resin, one against the side of the sensing element and the other one inch from the outside of the sensing element. These thermocouples established the temperature gradient through the resin and indicated the temperature at which the strain readings were taken.

Figure 146 compares the stresses of rigid polyester (Selectron 5003 filled with silica) and two flexible casting resins at various temperatures. As can be seen, rigid materials have a fairly straight line relationship between stress and temperature. Flexible resins, however, do not exert stress until they become rigid at the lower temperatures.

TABLE 61. FLEXURAL PROPERTIES OF RESIN COMPOUNDS CURED WITH (HARCURE A) POLYSEBACIC ANHYDRIDE OR ALIPHATIC DICARBOXYLIC ACIDS (76)

Formulation	Heat Dist. Temperature (ASTM D-648) °C	Weight Loss %	Flexural Properties of Test Bars					
			at Room Temperature			at -40° C		
			Elon-gation %	Flexural Strength K psi	Modulus of Elasticity K psi	Elon-gation %	Flexural Strength K psi	Modulus of Elasticity K psi
100 ED-1025 161 Harcure A	<RT	9.5	—	flexible	—	>3.5	>8.0	255
	After aging for 150 hrs.*		>3.5	>1.2	40	3.5	>8.0	230
100 Epon 828 116 Harcure A	<RT	10.5	—	flexible	—	3.5	>7.5	225
	After aging for 80 hrs.		>3.5	>5.5	165	>3.5	7.0	210
	After aging for 420 hrs.		>3.5	>5.5	160	—	—	—
100 DEN 438 103 Harcure A	47	4.5	>3.5	>6.0	195	>3.5	>7.5	220
	After aging for 80 hrs.		>3.5	>5.5	190	>3.5	>7.0	200
	After aging for 420 hrs.		2.2	3.5	190	—	—	—
100 Unox 201 106 Harcure A	32	25.2	>3.5	>3.0	90	>3.5	>8.0	240
	After aging for 80 hrs.		>3.5	>6.5	185	>3.5	>8.0	220
	After aging for 420 hrs.		3.1	5.0	170	—	—	—
100 Unox 201 33.4 Adipic Acid (1:0.75)	71	9.1	1.3	5.0	380	2.3	9.0	395
	After aging for 80 hrs.		2.2	7.5	350	—	—	—
	After aging for 280 hrs.		2.2	7.5	330	—	—	—
100 Unox 201 34.5 Pimelic Acid (1:0.75)	80	10.5	>3.5	>12.5	380	3.1	12.0	385
	After aging for 80 hrs.		>3.5	>11.0	340	3.3	1.1	330
	After aging for 280 hrs.		2.8	7.5	270	—	—	—
100 Unox 201 39.2 Azelaic Acid (1:0.7)	66	15.0	>3.5	>11.5	330	3.5	12.0	350
	After aging for 80 hrs.		>3.5	>10.5	310	3.5	10.5	325
	After aging for 280 hrs.		2.8	8.0	290	—	—	—
100 Unox 201 55.5 Azelaic Acid (1:1)	46	15.8	>3.5	>10.3	>290	>3.5	>12.0	350
	After aging for 80 hrs.		>3.5	>9.5	280	3.5	10.0	280
	After aging for 280 hrs.		2.5	6.5	270	—	—	—
100 Epon 828 50 Azelaic Acid (1:1)	<RT	4.2	>3.5	>4.5	330 (plastic)	>3.5	>10.0	300
	After aging for 80 hrs.		>3.5	>8.0	245	>3.5	9.0	275
100 Unox 201 43.5 Sebacic Acid (1:0.7)	65	15.5	>3.5	>11.0	320	>3.5	>12.0	350
	After aging for 80 hrs.		>3.5	>10.0	300	2.6	8.5	320
	After aging for 280 hrs.		2.4	7.0	300	—	—	—

*all heat aging at 200°C

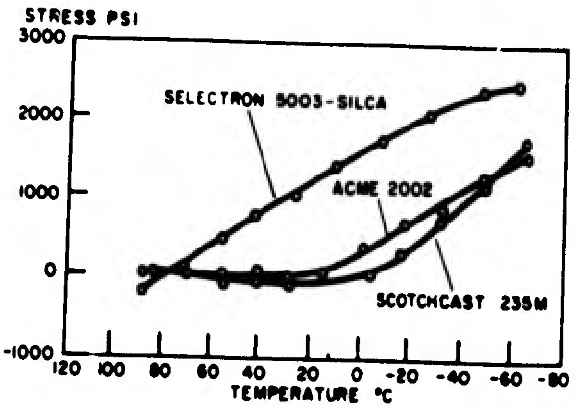


Figure 146. Comparison of flexible and rigid resins (77)

TABLE 62. FLEXURAL PROPERTIES OF RESIN COMPOUNDS CURED WITH ALIPHATIC DICARBOXYLIC ACIDS AND CAPROLACTAM (76)

				Flexural Properties of Test Bars					
				at Room Temperature			at -40° C		
Formulation	Heat Dist. Temperature (ASTM D 648) °C		Weight Loss %	Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi	Elongation %	Flexural Strength K psi	Modulus of Elasticity K psi
100 Epon 828 26 Harcure A 5 Caprolactam	<RT	Cured at 155°C	5.4	—	—	—	—	—	—
		Cured at 200°C		>3.5	>10.5	300	>3.5	>11.5	320
		Aged for 80 hrs.**		>3.5	>10.0	290	>3.5	>11.0	325
		Aged for 280 hrs.		3.4	9.0	290	—	—	—
100 Epon 828 13 Harcure A 10 Caprolactam	49	Cured at 155°C	5.2	—	—	—	—	—	—
		Cured at 200°C		>3.5	>10.5	305	>3.5	>12.0	350
		Aged for 80 hrs.		>3.5	>11.0	325	>3.5	>11.5	340
		Aged for 280 hrs.		2.8	10.0	350	—	—	—
100 Epon 828 32.6 Azelaic Acid 5 Caprolactam	51	Cured at 155°C	5.8	>3.5	>11.0	325	>3.5	>11.0	320
		Aged for 80 hrs.		>3.5	>10.5	315	>3.5	>11.0	325
		Aged for 280 hrs.		>3.7	~11.0	320	—	—	—
100 Epon 828 28 Sebacic Acid 2.5 Caprolactam	55	Cured at 155°C	4.6	>3.5	>10.5	325	>3.5	>10.5	300
		Aged for 80 hrs.		>3.5	>10.0	300	>3.5	>11.0	300
100 Epon 828 30 Dicarboxylic Acids*	<RT	Cured at 155°C	5.3	—	nearly flexible	—	1.7	.5	325
	41	Cured at 200°C		>3.5	>11.0	330	3.6	11.5	320
		Aged for 150 hrs.		2.5	7.0	280	—	—	—
100 Epon 828 35 Dicarboxylic Acids*	<RT	Cured at 155°C	4.3	>3.5	>2.5	85	2.9	9.0	325
	44	Cured at 200°C		>3.5	>11.0	330	3.6	11.5	320
		Aged for 150 hrs.		2.9	8.0	290	—	—	—
100 Epon 828 30 Dicarboxylic Acids* 5 Caprolactam	41	Cured at 155°C	5.5	>3.5	>10.0	310	>3.5	>12.0	340
	53	Cured at 200°C		>3.5	>11.0	320	—	—	—
		Aged for 150 hrs.		>3.5	>10.0	290	>3.5	>11.0	320

* Mixture of 2 pbw Harcure A, 2 pbw Azelaic Acid and 1 pbw Sebacic Acid.

** all heat aging at 200°C

Figure 147 shows the changes in stress at various temperatures when an epoxy is modified by styrene oxide and glycidyl phenyl ether. The styrene oxide modified epoxy is superior at the lower temperatures.

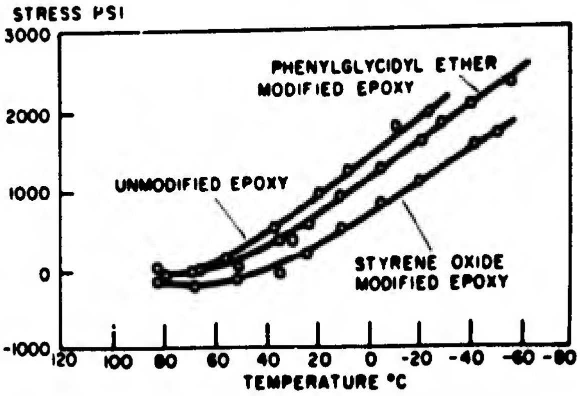


Figure 147. Effect of reactive diluents (77)

The effect of hardeners on stress of casting resin Epi-Reg 507 is shown in Figure 148. The importance of hardener selection is illustrated by the difference in the stress of the two curing agents. For example, the stress of curing agent "D" is half that of "Z" at 0° C.

Figure 149 illustrates the difference in stresses of an epoxy system cured for two hours at 65°C followed by two hours at 93°C with the same system cured for four hours at 65°C. As can be seen, the system cured at the lower temperature has the lowest stresses.

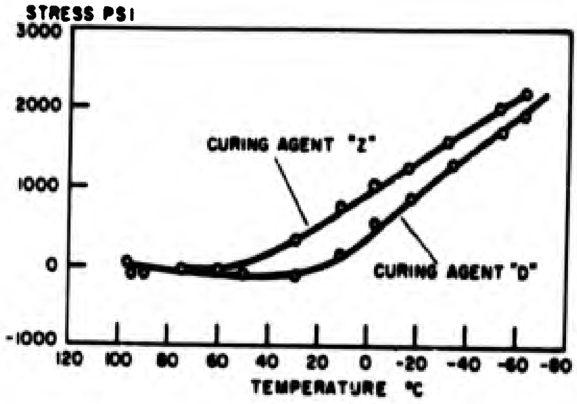


Figure 148. Effect of hardener (77)

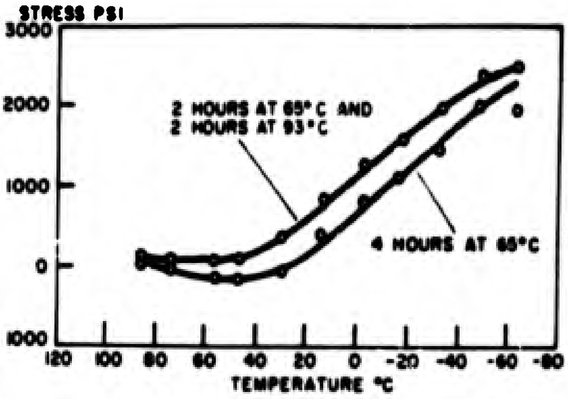


Figure 149. Effect of curing temperature (77)

Figures 150 and 151 show the effect of fillers and curing agents on the cracking of casting resins at low temperatures. These data again support the previous statement that the stresses from all factors (resin, fillers, flexibilizers, curing agent, cure cycle, etc.) are greater at the lower temperatures.

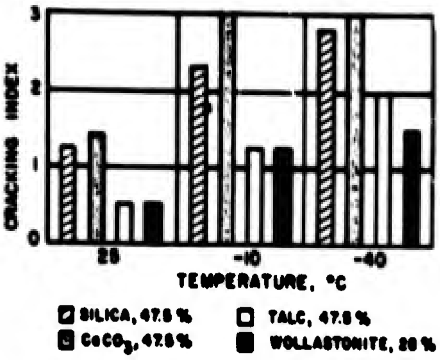


Figure 150. Effect of filler on cracking index of epoxy resin using the Black Test (hardener, 10.5 phr Shell D) (78)

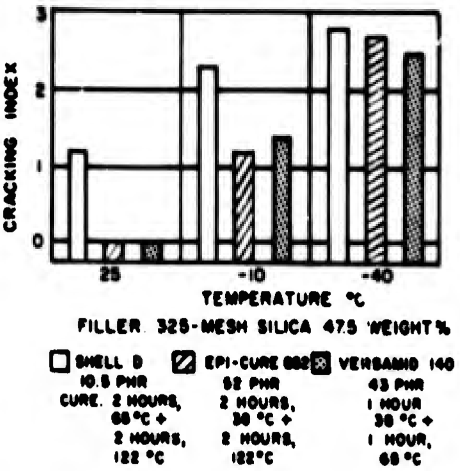


Figure 151. Effect of curing agent on cracking index of epoxy resins using the Black Test (78)

Low temperature creep properties of epoxy-glass laminates were determined by the adoption of a lever-load machine. (79) Table 63 summarizes the data obtained on four representative samples at -20° F.

TABLE 63. CREEP-RUPTURE DATA, R5 LAMINATE AT -20° F (79)

Specimen Number	Stress, Psi.	Rupture Life, Hours	Min. Creep Rate, % Hour
R5-6	41,000	2.9	0.02885
R5-8	40,000	13.1	0.00850
R5-13	43,000	0.7	0.138
R5-20	36,000	229.5	0.00095

Tables 64 through 67 present data on compressive, bearing, burst and shear strengths of epoxy filament wound composites as a function of temperature. The data (with the exception of compressive modulus) is consistent with the general observation that the mechanical properties increase with decreasing temperature.

TABLE 64. COMPRESSIVE PROPERTIES OF SPIRALLOY AS A FUNCTION OF TEMPERATURE (RINGS - 2.6 in. x 1.5 in. x 3/16 in.) (80)

Composition	°F	Compressive(3) Strength, Kpsi	Compressive(3) Modulus, psi x 10^6
E/114 Chrome-8 end + 828/A(1)	165	15.08	0.89
	76	21.86	1.52
	-60	28.50	1.20
E/114 Chrome-8 end + 828/CL(2)	165	21.37	1.05
	76	27.33	1.69
	-60	36.22	1.49
E/7.2 Garan-12 end + 828/A(1)	165	15.00	0.85
	76	22.93	1.66
	-60	27.85	1.52
E/7.2 Garan-12 end + 828/CL(2)	165	16.20	0.85
	76	21.74	1.67
	-60	26.40	1.71

(1) Cure schedule: 250F/1.5 hrs.
(2) Cure schedule: 400F/1.5 hrs.
(3) Data is average of 2 or more specimens.

TABLE 65. BEARING STRENGTH PROPERTIES OF SPIRALLOY AS A FUNCTION OF TEMPERATURE (RING - 2.6" x 1.5" x 3/16"; 80% LEVELS, 20% 15° HELICALS) (80)

Composition	Temp. °F	Bearing Strength (1) Ultimate, Kpsi	Cure Schedule °F/hr.
E/114 chrome - 8 end + 828/A	165	35.00	250/1.5
	76	33.40	280/1
	-60	41.50	280/1
E/114 chrome - 8 end + 828/CL	165	31.80	400/1
	76	39.76	400/1.25
	-60	53.45	400/1
E/7.2 Garan - 12 end + 828/A	165	22.75	280/1.5
	76	29.70	250/1.5
	-60	37.50	280/1.5
E/7.2 Garan - 12 end + 828/CL	165	29.10	400/1
	76	33.30	400/1
	-60	38.80	400/1
E/120 PX - 10 end + 828/A	165	21.60	280/1.5
	76	25.25	250/1.5
	-60	30.80	280/1.5
E/120 PX - 10 end + 828/CL	165	24.60	400/1
	76	27.70	300/1.5 + 400/1.5
	-60	32.70	300/1.5 + 400/1.5

(1) Constant load rate of 0.050 inch per minute on bearing area of 0.775 sq. in.

TABLE 66. BURSTING STRENGTH OF SPIRALLOY AS A FUNCTION OF TEMPERATURE (6" DIAMETER BOTTLES, 54 - 3/4° HELICALS) (80)

Composition	Temp °F	Ultimate Girth Stress (1) Kpsi	Burst Strength Kpsi
E/114 chrome - 8 end + 828/CL (No liner)	165	50.10	26.40
	-60	67.70	35.65
E/114 chrome - 8 end + 828/A	165	57.50	30.65
	-60	83.00	44.30
E/120PX - 10 end + 828/A (Nylon Liner)	-60	77.77	40.53

(1) Cylinders at -60F contained ethylene glycol.

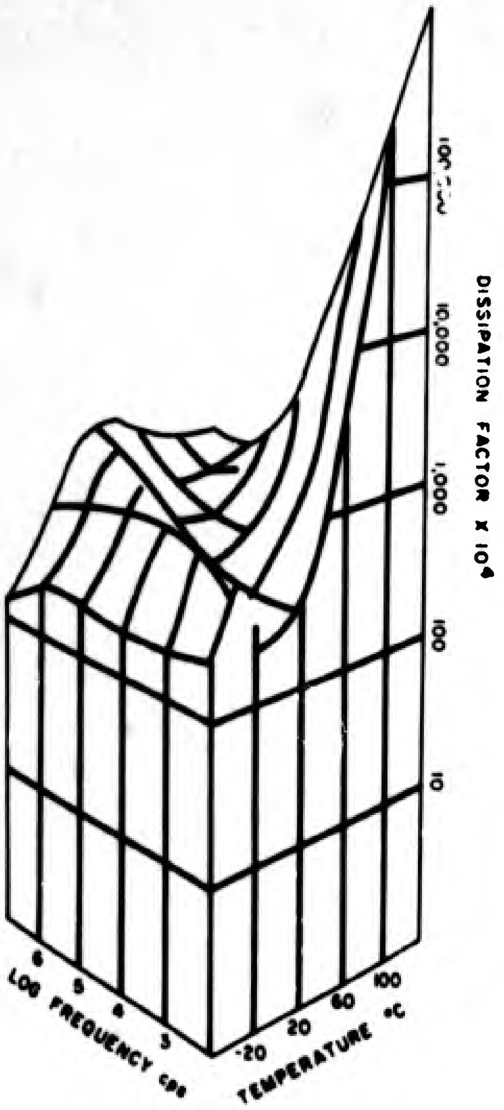


Figure 152. Dissipation factor of a modified epoxy resin (100 parts epoxy - 100 parts polysulfide rubber) as a function of frequency and temperature (61)

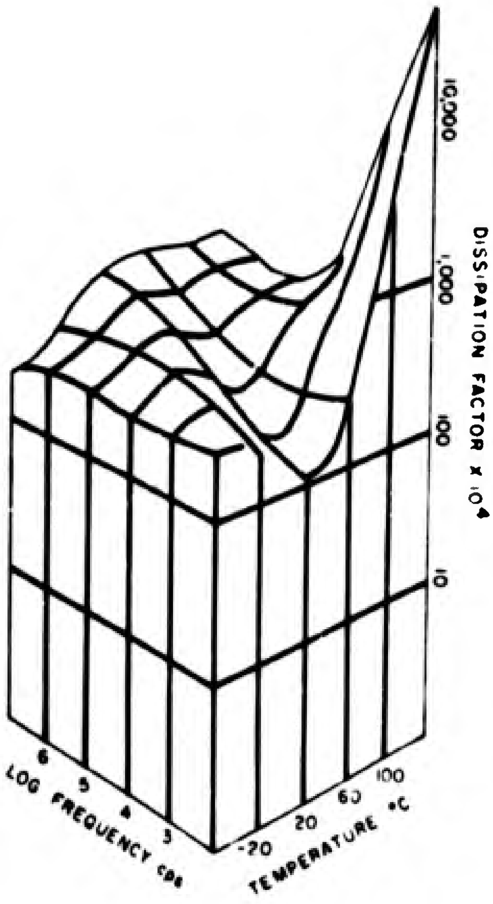


Figure 153. Dissipation factor of a modified epoxy resin (100 parts epoxy - 40 parts polysulfide rubber) as a function of frequency and temperature (61)

TABLE 67. SHEAR STRENGTH OF SPIRALLOY AS A FUNCTION OF TEMPERATURE (RINGS - 2.6" x 0.5" x 3/16") (80)

Composition	Temp °F	Interlaminar (1) Shear Strength (Kpsi)	Cure Schedule °F/hrs.
E/114 chrome - 8 ends + 828/A	165	4.01	280/2.5
	76	5.25	250/2 +280/1
	-60	6.24	280/1
E/114 chrome - 8 ends + 828/CL	165	5.02	300/1 + 400/0.5
	76	6.69	400/2.5
	-60	8.09	300/1 +400/1
E/7.2 Garan - 12 ends + 828/A	165	2.56	250/1.5
	76	3.98	200/1.5
	-60	5.79	250/1
E/7.2 Garan - 12 ends + 828/CL	76	3.87	400/0.5
	-60	7.08	300/1 + 400/1
E/120PX - 10 ends + 828/A	165	2.79	250/1.5
	76	3.93	250/1.5
	-60	5.36	250/1.5
E 120PX + 828/CL	165	2.83	400/0.5
	76	3.87	400/1.5
	-60	5.59	300/1 + 400/1

(1) Specimens were conditioned at respective temperatures, and wrapped in insulation during test.

It should be noted that most of the data in these tables are on resin and glass combinations which are not used today. For example, Epon 828/CL was used with E glass bearing the 801 finish or EPS-4, which was the forerunner of HTS. No data were given on S994 glass. Also, the data on interlaminar shear and compressive strengths were determined on 2.6" diameter rings. These specimens were obtained by the ring punch-out method which is considered inferior today. Therefore, there is some skepticism concerning the data because of poor reproducibility. (80)

Electrical Properties

Illustrations of three-dimensional plastic model field diagrams of the dielectric constant and dissipation factor of several epoxy casting compounds are given in Figures 152 through 161. (61) Data were measured by a modified Schering bridge at frequencies from 10^2 to 10^5 cps and a series resonance circuit at 10^6 and 10^7 cps. Samples were approximately 2.0 inches in diameter and 0.2 inches thick.

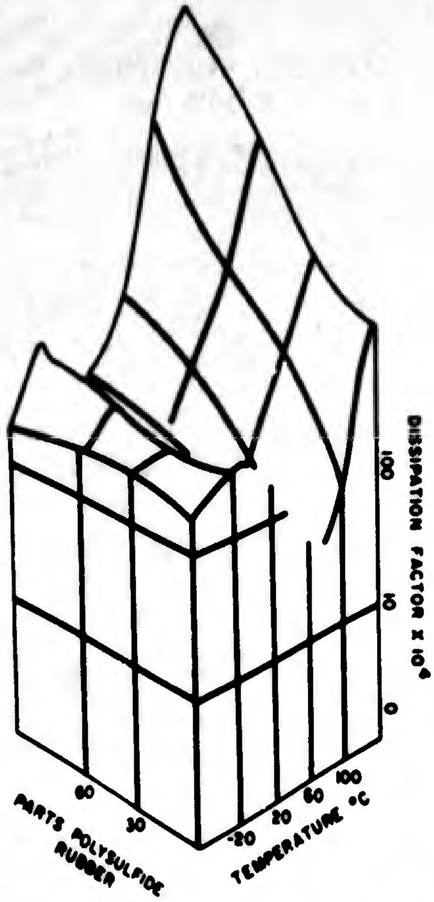


Figure 154. Dissipation factor of epoxy-polysulfide resins at 1000 cps as a function of temperature and resin composition (61)

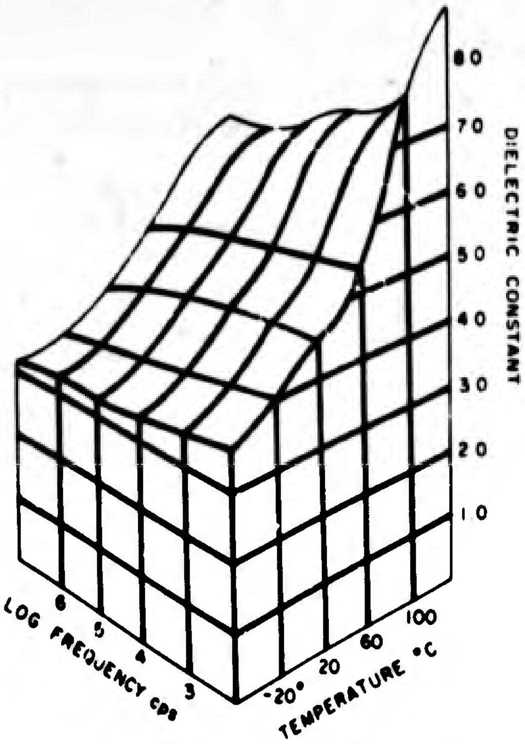


Figure 156. Dielectric constant of a modified epoxy resin (100 parts epoxy-40 parts polysulfide rubber) as a function of frequency and temperature (61)

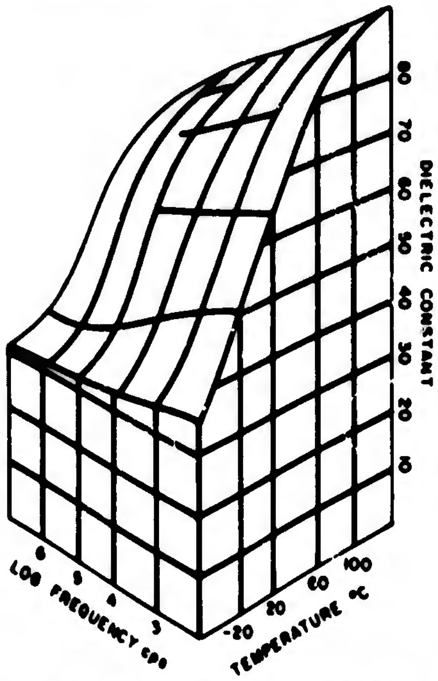


Figure 155. Dielectric constant of a modified epoxy resin (100 parts epoxy-100 parts polysulfide rubber) as a function of frequency and temperature (61)

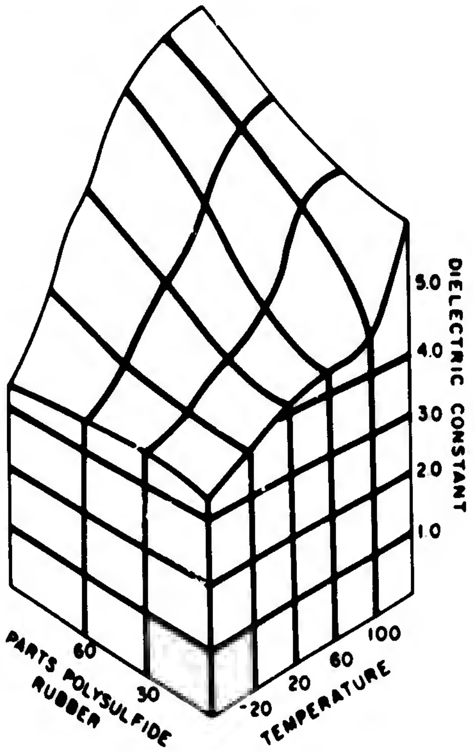


Figure 157. Dielectric constant of epoxy-polysulfide resins at 1000 cps as a function of temperature and resin composition (61)

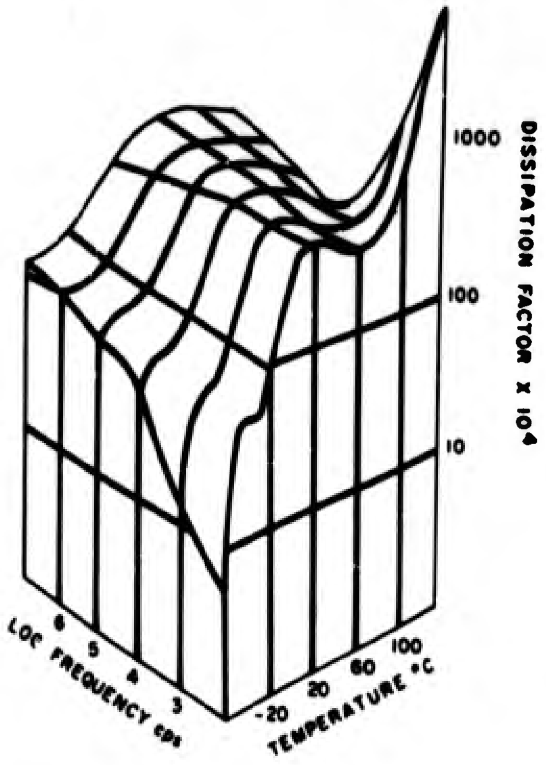


Figure 158. Dissipation factor of an epoxidized polybutadiene resin as a function of frequency and temperature (61)

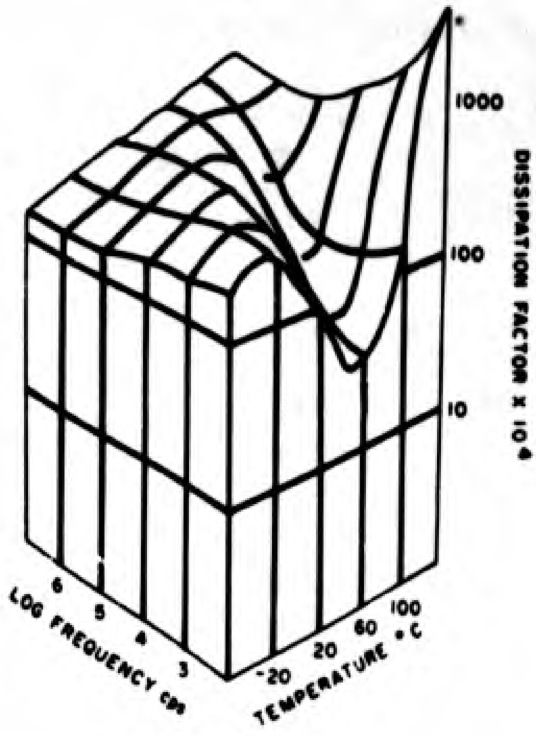


Figure 160. Dissipation factor of an epoxy resin (cured with diethylene triamine) as a function of frequency and temperature (61)

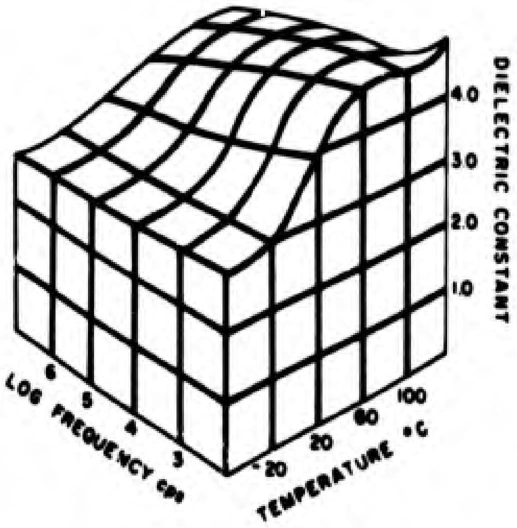


Figure 159. Dielectric constant of an epoxidized polybutadiene resin as a function of frequency and temperature (61)

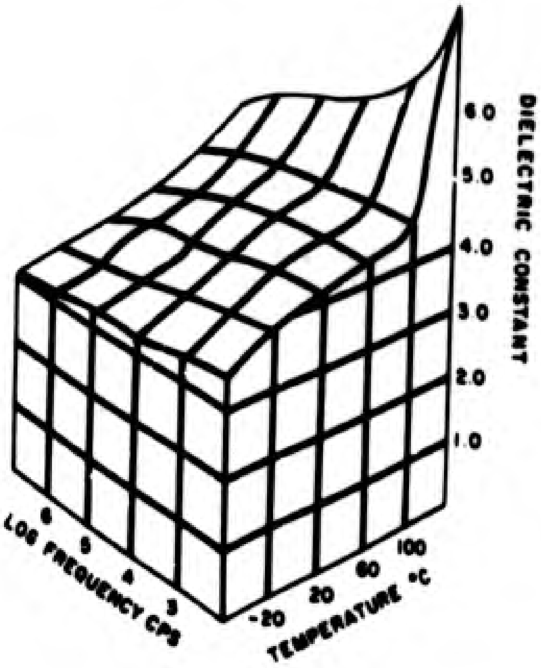


Figure 161. Dielectric constant of an epoxy resin (cured with diethylene triamine) as a function of frequency and temperature (61)

The dielectric constant and dissipation factor of an epoxy casting system at three frequencies is plotted in Figures 162 and 163.

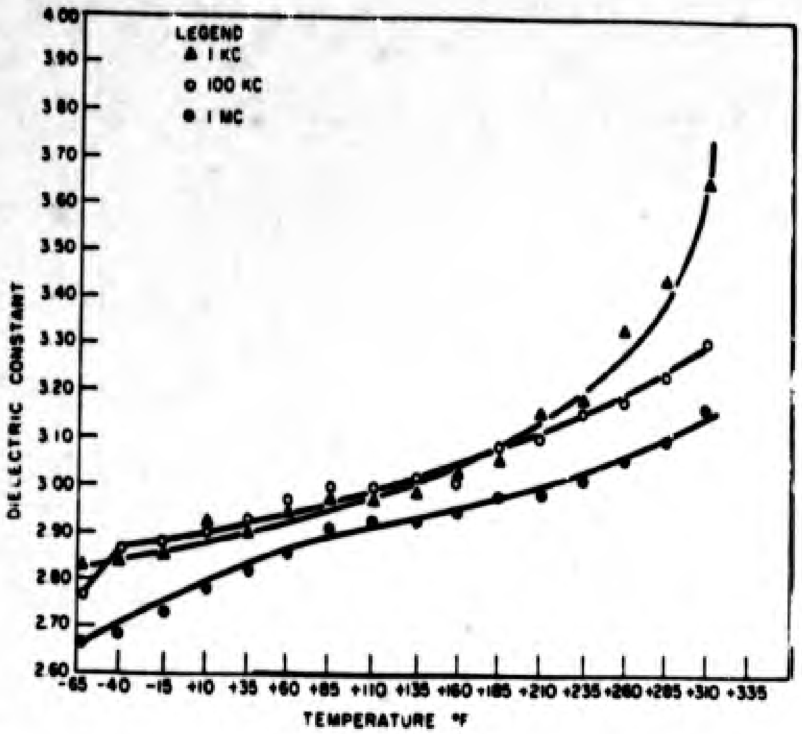


Figure 162. Dielectric constant for TOA-Epon 828 System (81)

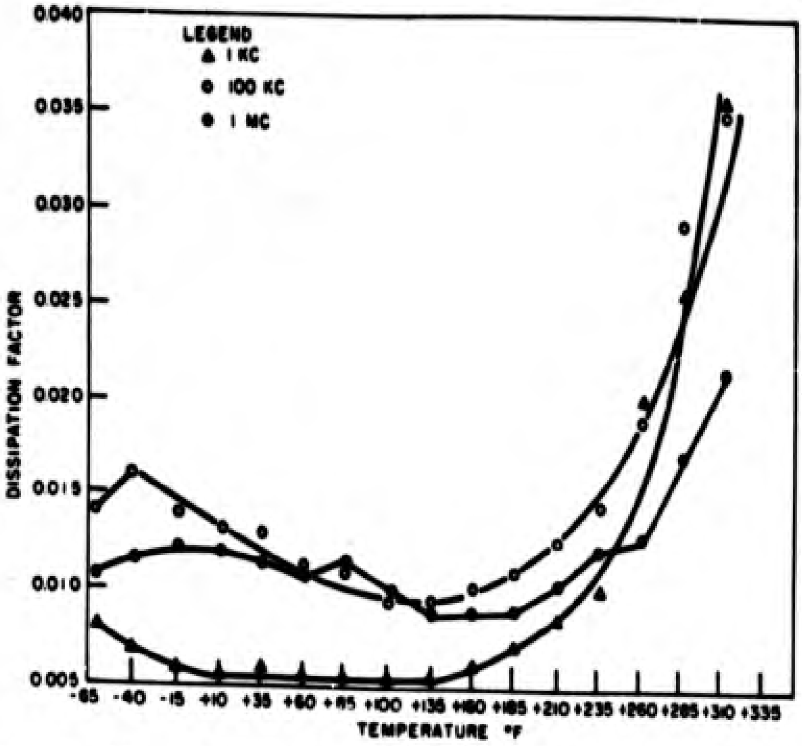


Figure 163. Dissipation factor for TOA-Epon 828 System (81)

Note: TOA = Tung oil trimaleate adduct

The volume resistivity of various epoxy encapsulants was determined. (82) Measurements were made at -65° F, 73.5° F and 160° F in accordance with ASTM D-257 and LP 406B, method 4041 using a Teraohmmeter. The results are given below:

TABLE 68. VOLUME RESISTIVITY OF ENCAPSULATING MATERIALS AT VARIOUS TEMPERATURES (82)

Material[1]	Volume Resistivity, ohm-cm[2]		
	at -65°F	at 73.5°F	at 160°F
Epoxy mixture Epon 828, epoxy, 100 Microballoons phenolic, 25 Piperidine hardener, 5 (5) HB 40, flexibilizer, 10 (7)	6×10^{15}	9×10^{13}	3×10^{12}
Stycast 2662, epoxy, 100 Hardener 14, 25 (6)	5×10^{15}	1×10^{15}	5×10^{13}
Stycast 2651, epoxy, 92 Hardener 11, 8 (6)	4×10^{15}	1×10^{15}	1×10^{14}
Epon 828, epoxy, 100 Hardener D, 10.5 (4)	8×10^{15}	6×10^{15}	2×10^{14}
Stycast 1095, foam-filled epoxy, 100 Hardener 17, 25 (6)	4×10^{14}	6×10^{13}	6×10^{12}
Stycast 2850 GT, highly filled epoxy, 96 Hardener 11, 4 (6)	4×10^{15}	3×10^{15}	1×10^{14}
Epoxy resin mixture: Epon 825, epoxy, 100 HB 40, flexibilizer, 10 (7) Piperidine, hardener, 5 (5)	4×10^{16}	7×10^{15}	9×10^{13} (3)

1. Parts by weight
2. Test data taken in accordance with ASTM D-257 on 1/8" thick specimen at 600 volts on all materials. Reading taken 1 minute after applied voltage
3. Test specimens undercured.
4. Shell Chem. Corp.
5. Olin Mathieson Chem. Co.
6. Emerson & Cuming, Inc.
7. Monstanto Chem Co.

Thermal Properties

The total expansion of plastic materials and composites must be known to design components and systems properly. Otherwise, as previously stated, failure will occur by cracking at joints, bonded surfaces, etc.

The average coefficient of linear thermal expansion from -65 to +160° F of various commercial epoxy encapsulating materials is given in Table 69. (82)

TABLE 69. COEFFICIENT OF LINEAR THERMAL EXPANSION OF ENCAPSULATING MATERIALS (82)

Material(1)	Coefficient of Linear Thermal Expansion (-65°F to 160°F) in./in./°F. x 10^{-5}
Epoxy resin mixture: Epon 828, epoxy, 100 Microballoons, phenolic, 25 Piperidine hardener, 5 HB 40, flexibilizer, 10	4.2
Stycast 2662, epoxy, 100 Hardener 14, 25	4.2
Stycast 2651, epoxy, 92 Hardener 11, 8	2.5
Epon 828, epoxy, 100 Hardener D, 10.5	4.8
Stycast 1095, foam-filled epoxy, 100 Hardener 17, 25	3.1
Stycast 2850 GT, highly filled epoxy, 96 Hardener 11, 4	1.6
Epoxy resin mixture: Epon 825, epoxy, 100 HB 40, flexibilizer, 10 Piperidine hardener, 5	4.5

(1) Parts by weight

Note: See table 68 for suppliers.

The total linear thermal expansion of five molded (filled) epoxy panels and two glass reinforced laminates was determined on a Leitz dilatometer. (30) The material description and codes are given in Table 70.

Generally, the total linear expansion of composites can be explained as follows: " In a laminate composed of alternate layers of reinforcement and resin, the expansion in the thickness direction approaches the condition obtained by stacking blocks of dissimilar materials. The expansion approaches the sum of the expansions of each layer, and results in a value between that of the two materials. In the directions normal to the thickness, the bonded layers try to expand at different rates

TABLE 70. MATERIAL DESCRIPTION OF MOLDED EPOXY-FILLED PANELS AND GLASS REINFORCED LAMINATES (30)

Identification		S1, S2	T-1	U-1	V-1	W-1	D-1	D-2	E-1	E-2
Manufacturer:	CTL (1)	X	X	X	X	X	--	--	--	--
	Raytheon	--	--	--	--	--	X	X	X	X
Resin source:	Shell Chemical	X	X	X	X	X	--	--	--	--
	Dow Chemical	--	--	--	--	--	X	X	X	X
Prepreg source:	Owens Corning Fiberglass	--	--	--	--	--	X	X	X	X
Resin Content, %		25	30	53-55	25	35	40	40	40	40
Resin, Epoxy:	EPON 828	X	X	X	X	X	--	--	--	--
	DER-332	--	--	--	--	--	X	X	X	X
Curing Agents:	DTA	X	X	X	X	X	--	--	--	--
	NMA (2) & DMP 30	--	--	--	--	--	X	X	X	X
Reinforcement:										
Perlite granules (LM-30)		X	--	--	--	--	--	--	--	--
Pot. titanate fibers (Tipersul)		--	X	--	--	--	--	--	--	--
Phenolic microballoons (BJ 00930)		--	--	X	--	--	--	--	--	--
Glass microballoons (Eccospheres R)		--	--	--	X	X	--	--	--	--
Glass roving (YM-31-A-Glass)		--	--	--	--	--	X	X	X	X
Lay-up:	Random	X	X	X	X	X	--	--	--	--
	Parallel	--	--	--	--	--	X	X	--	--
	Crossplied (57° and 303°)	--	--	--	--	--	--	--	X	X
Number of plies		--	--	--	--	--	80	20	80	20
Orientation:	Unidirectional	--	--	--	--	--	X	X	--	--
	Crossplied (90°)									
Cure Cycle:										
Press to stops		X	X	X	X	X	--	--	--	--
Press to 1/2" stops (200 psi)		--	--	--	--	--	X	--	X	--
Press to 1/8" stops (200 psi)		--	--	--	--	--	--	X	--	X
Temperature, °F		200	200	200	200	200	300	300	300	300
History: Raise to 200°F and hold 1 hr; cool		X	X	X	X	X	--	--	--	--
B-stage 30 min at 150°F; cure at 200°F for 2 hrs; 250°F for 2 hrs; 300°F for 2 hrs; cool to 150°F.		--	--	--	--	--	X	X	X	X
Post-Cure Cycle; 350°F for 12 hrs.		--	--	--	--	--	X	X	X	X

(1) Division of Studebaker Corp.
(2) Nadic methyl anhydride

because of the difference in expansion coefficients between the cloth and the resin. Since the resin and cloth are bonded, the higher modulus material will tend to control the overall expansion in the directions normal to the thickness. The overall expansion of the composite in this case will be a value closer to the expansion of the higher modulus material than to the lower modulus material. This only remains true if the resin-to-cloth bond can withstand the thermal stress." (30)

Figures 164 through 166 plot the linear expansion of epoxy laminates D and E. Material D which is reinforced with a unidirectional glass roving is similar (see insert) in both the thickness and perpendicular to roving direction. The expansion in the thickness direction is approximately four times that in the roving direction. The expansion of Material E, crossplied at 57° to one edge of the panel also supports the above statement. The expansion was greater in the thickness direction than in the other two directions.

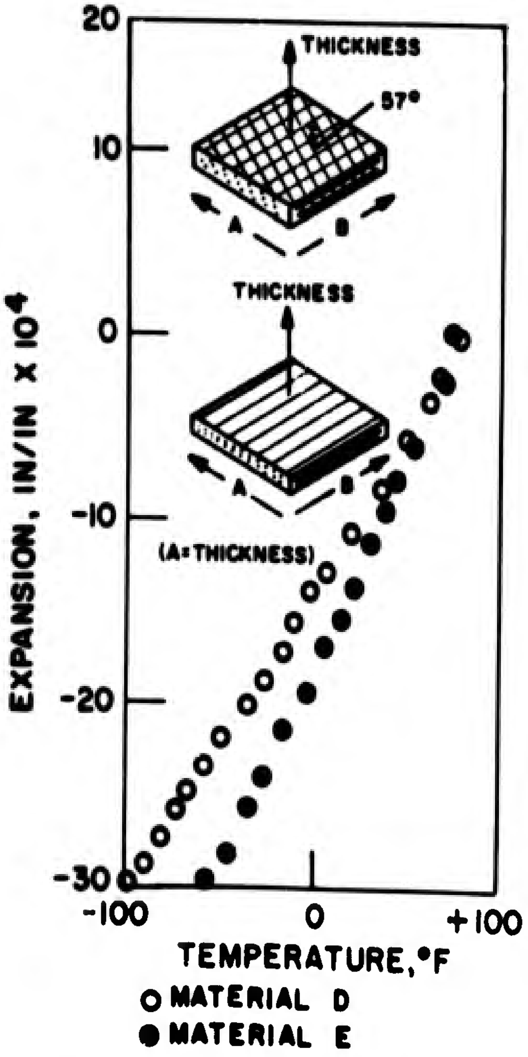


Figure 164. Total linear thermal expansion of material D and E (thickness) (30)

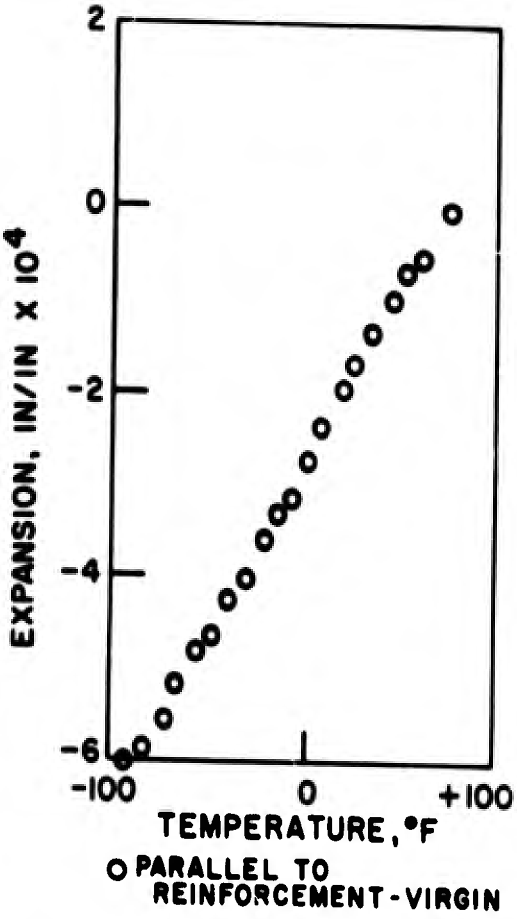


Figure 165. Total linear thermal expansion of material D (parallel to reinforcement (30)

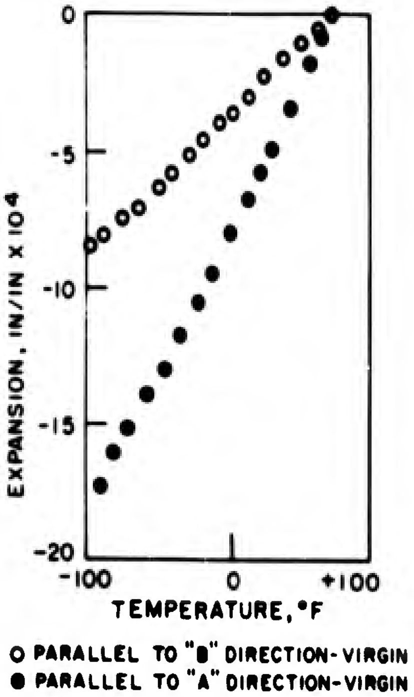
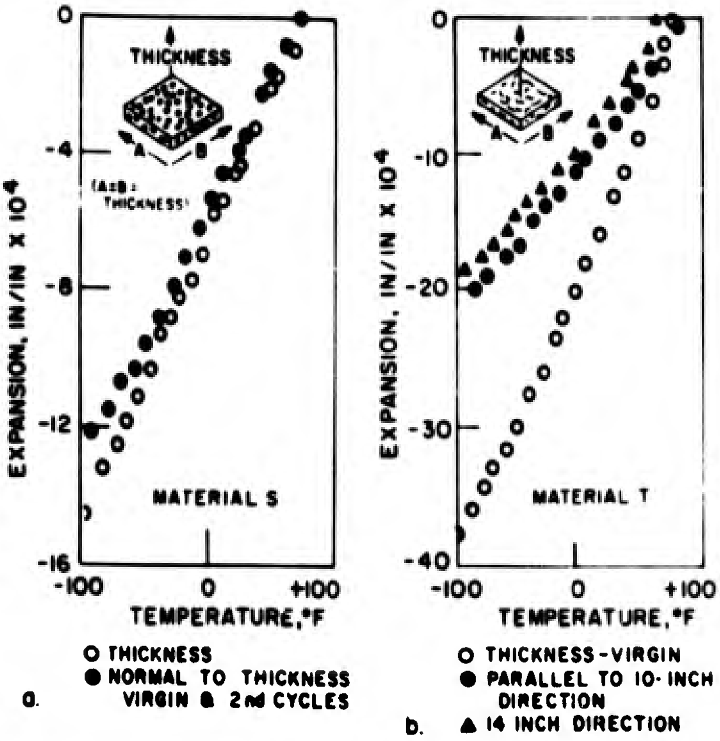
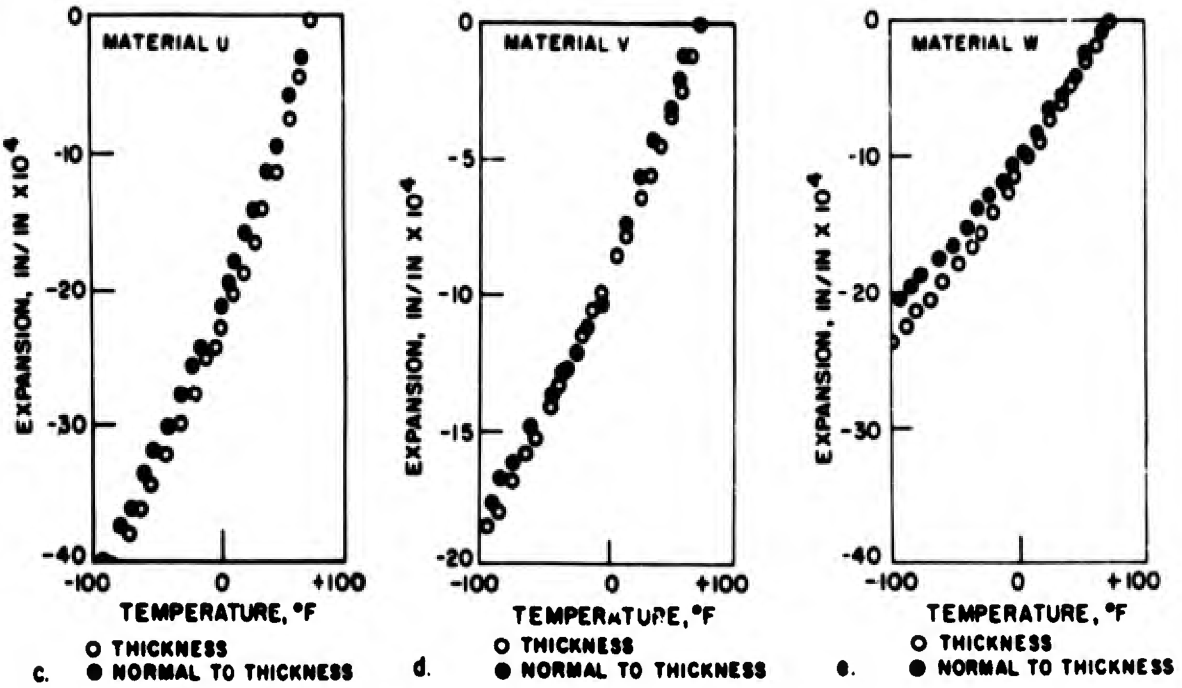


Figure 166. Total linear thermal expansion of material E (normal to thickness) (30)



a.

b.



c.

d.

e.

Figure 167. Total linear thermal expansion of epoxy molding panels (30)

Figure 167 plots the total linear expansion of epoxy molding compounds with random filler in two directions (normal to thickness and thickness). The little differences observed were attributed to variations in the resin contents. In fact, one material (V) had almost identical curves in both directions.

Measurements of sample T were made in three directions because it was felt that the potassium titanate filler would become orientated during molding. The data show the values in the thickness direction were approximately twice those in the other two directions. This indicates that there was more fiber orientation parallel to the panel surface. Also, the length of material T was assumed to be affected by the shrinking of the potassium titanate fibers when exposed to the dry helium atmosphere in the Leitz dilatomer.

Thermal conductivities are influenced by temperature, environment, type of reinforcement, reinforcement orientation, type of resin, density, void content and fabrication history.

Thermal conductivities were determined on glass reinforced epoxy panels and a casting compound. (83) Tests were run on a guarded hot plate slightly modified from standard ASTM C 177-45 to permit a range to 10 BTU/hr/ft^2/^{o}F/in. For determinations between -50^o F and 150^o F a gum rubber filler was used. Below room temperatures were obtained by pumping cold trichloroethylene through the cooling section. Accuracy was reported to be within +5 percent up to 10 BTU/ft^2/hr/^{o}F/in.

A description of the materials tested is given in Table 71.

Photomicrographs of the epoxy materials indicated excessive voids. Therefore, no well defined correlation of conductivity between resin percentage and material composition could be determined. However, some general conclusions were drawn from the data in Table 72, regarding the influence of direction on thermal conductivities.

- In the unidirectional parallel fiber materials, the conductivity was higher in the direction parallel to reinforcement than in the other two directions tested. The conductivities in the direction perpendicular to reinforcement and the thickness direction were almost identical.

- In the cross-plied panels, the directions parallel to and perpendicular to reinforcement had almost identical conductivities and were higher than the thickness direction.

- The thermal conductivities in the thickness direction of the two DER 332 epoxy YM 31 A glass fiber materials (c-1 and c-3 with unidirectional and cross-plied fibers, respectively) did not compare well with those for the parallel fibers (c-1). This was attributed to a variation in the fiber structure. The reinforcement fibers in the c-3 material were found to be grouped into separate bundles, whereas the fibers in the c-1 material were more evenly spaced, allowing for greater wettability by the resin and resulting in higher conductivity.

- The thermal conductivity of both glass flake materials were greater in the direction parallel to reinforcement than in the thickness direction.

A description of the materials tested is given below.

TABLE 71. DESCRIPTION OF EPOXY MATERIALS. (83)

Identification	C-1	C-2	C-3	C-4	C-5	D-1	D-2	F-3
Composite System:								
Density (specific gravity)	1.91	1.85	1.94	2.10	2.06	1.97	1.97	1.2
Resin content (before cure), %	40	30	40	20	20	30	23	100
Resin content (after complete fabrication) %	36.57	28.23	32.15	17.76	21.06	27.7	26.5	100
Void content (volume), %	5-10	5	5-10	5	5	Nil	Nil	Nil
Resin System:								
Trade name: DER 332	X	X	X	X	--	--	--	X
NRC 1174/3	--	--	--	--	--	X	X	--
DEN 438	--	--	--	--	X	--	--	--
Curing agents: NMA - 80 parts/100 gms resin	X	X	X	X	X	--	--	--
DMP, 2 parts/100 gms resin	X	--	X	X	X	--	--	X
DMP - 30, 30 parts/100 gms resin	X	--	--	--	--	--	--	--
Molded from epoxy powder at 350°F, 10 psi, 3 minutes	--	--	--	--	--	X	X	--
Reinforcement System:								
Trade name: High Modulus YM31A Glass (Fiberglass w beryllia, roving)	X	--	X	--	--	--	--	--
"E" Glass Roving (Fiberglass)	--	X	--	X	X	--	--	--
O-C Comp E Glass: Flake, (2 microns thick, 200-2000 microns diameter; 10-35 mesh)	--	--	--	--	--	X	X	--
Finish (HTS)	X	X	X	X	X	--	--	--
Lay-up: parallel to surface	X	X	X	X	X	--	--	--
Orientation; number of plies								
Unidirectional parallel	X	--	--	--	X	--	--	--
Parallel, 40 layers	--	X	--	--	--	--	--	--
Crossplied, 40 layers	--	--	--	X	--	--	--	--
Crossplied, parallel and 66° from horizontal axis, 40 layers	--	--	X	--	--	--	--	--
Random	--	--	--	--	--	X	X	--
Cure Cycle: Pressure, psi	200	200	200	200	200	800	1500	--
Temperature, °F	200-250	200-250	200-250	200-250	200-250	350	350	250
Time: 2 hrs at 200°F; 2 hrs at 250°F	X	X	X	X	X	--	--	--
120 minutes	--	--	--	--	--	X	X	--
1 hour	--	--	--	--	--	--	--	X
Post-Cure Cycle: Time Temperature:								
2 hrs at 300°F; 12 hrs at 350°F	X	--	X	--	--	--	--	--
2 hrs at 300°F; 12 hrs at 250°F	--	--	--	X	--	--	--	--
2 hrs at 300°F; 24 hrs at 350°F	--	X	--	--	X	--	--	--

TABLE 72. THERMAL CONDUCTIVITY OF EPOXY MATERIALS (83)

Thermal Conductivity of Epoxy Laminate with Unidirectional Parallel Glass Reinforcements												
Material	Parallel to Reinforcement				Perpendicular to Reinforcement				Thickness Direction			
Temperature °F	-50	0	+70	+150	-50	0	70	150	-50	0	70	150
C-5	3.1	3.3	3.4	4.0	2.8	2.8	2.8	3.1	2.8	2.9	3.0	3.2
C-2	3.8	4.0	4.2	4.7	2.9	3.2	3.3	3.1	2.8	3.0	3.1	3.2
C-1	4.8	5.0	5.4	5.6	3.5	3.7	3.8	3.9	2.7	2.9	3.1	3.2
	Thermal Conductivity of Epoxy Laminate with Cross Ply Glass Reinforcements											
C-4	3.1	3.4	3.8	4.0	3.2	3.4	3.7	3.9	2.7	2.9	3.2	3.3
C-3	2.2	2.8	3.4	3.5	2.8	3.0	3.3	3.4	1.7	1.8	1.8	1.9
	Thermal Conductivity of Epoxy Glass Flake Laminates											
D-1	3.1	3.4	3.7	4.1	-	-	-	-	2.3	2.4	2.5	2.6
D-2	3.9	4.0	4.0	4.1	-	-	-	-	2.5	2.6	2.7	2.8
	Thermal Conductivity of Epoxy Casting											
F-3	-	-	-	-	-	-	-	-	.83	.9	1.0	1.18

- The thermal conductivity of the casting material was very low compared to the reinforced materials.

Table 73 lists the different coefficients of linear expansion for various fiber orientations of epoxy-E glass laminates.

TABLE 73. COEFFICIENT OF LINEAR EXPANSION OF EPOXY-E GLASS NONWOVEN LAMINATES (73, 74)

Method - ASTM D696-42T

Tested over Temperature Range of -30°F to + 200°F

Fiber Orientation	Direction of Measurement	Coefficient of Linear Expansion Per °F	
		Scotchply 1000	Scotchply 1002
Isotropic	Parallel to one array of filaments	8.8×10^{-6}	8.4×10^{-6}
Crossplied	Parallel to lengthwise filaments	12.8×10^{-6}	7.1×10^{-6}
Crossplied	Parallel to crosswise filaments	12.8×10^{-6}	7.1×10^{-6}
Unidirectional	Parallel to all filaments	8.4×10^{-6}	4.8×10^{-6}
Unidirectional	Perpendicular to all filaments	21.9×10^{-6}	12.3×10^{-6}

Figure 168 also shows the importance of reinforcement orientation on thermal expansion. As can be seen, expansion on the same panel increased from a minimum at 0° to the major axis of the fiber, parallel to the reinforcement, to a maximum of 90° to the major axis of the fiber. The expansion of an isotropic laminate was measured for comparison and found to be closest to the 0° to the major axis expansion.

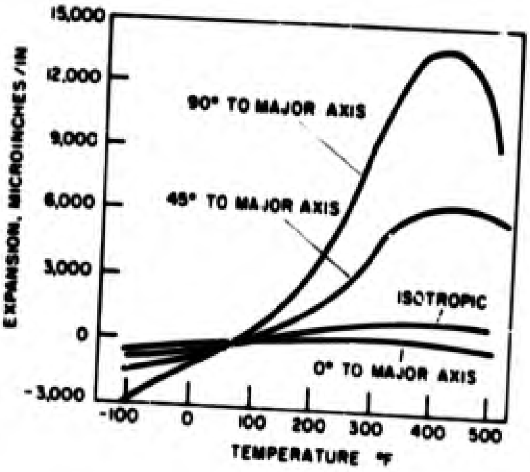


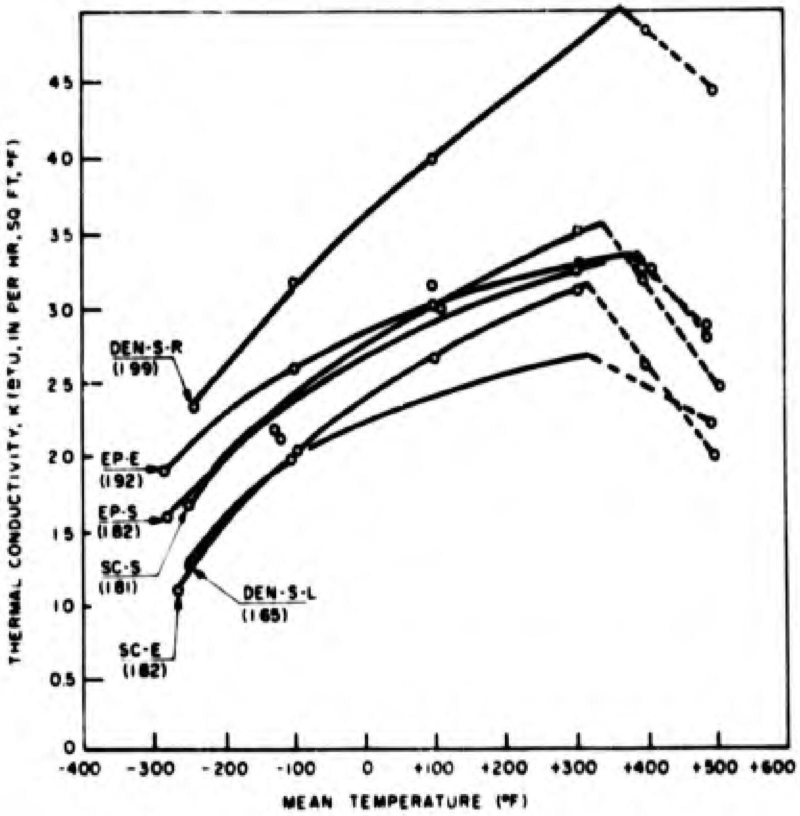
Figure 168. Influence of reinforcement orientation on expansion of 60N roving epoxy laminates (84)

Note

Type	Reinforcement	Resin	No. of plies	Thickness in.	Amount of Resin %	Finish	Catalyst	Fabrication and Curing
Unidirectional laminate	60N roving	Epoxy 3MXP 175	30	0.125	30 ±1.5	Garan	3MXP-175	Press temp 275-300°F Total time in press 60 min Pressure on panel 80-95 psi Postcure temp 325°F Time in postcure 10 hr
Isotropic laminate	60N roving	Epoxy 3MXP 175	21	0.122	30 ±1.5	Garan	3MXP-175	Press temp 300-370°F Total time in press 60 min Pressure on panel 80 psi Postcure temp [illegible]°F Time in postcure 10 hr

The authors point out that this data was on a roving reinforcement material and probably had greater difference in expansion at different orientations than would have been found for a woven reinforcement.

Forest Products Laboratory measured the thermal conductivity of six epoxy laminates by means of a heat flow meter in conformance with ASTM C 518-63 T. Values were obtained at six mean temperatures and the data plotted. Data in this report's temperature range must be "picked off" the smooth curves drawn through the plotted points -100° to 200° F. From Figure 169 the following is demonstrated:



Note:

DEN-S-L	Resin, Dow DEN 438; reinforcement, S-glass 181 fabric, parallel laminated, low density
DEN-S-R	Resin, Dow DEN 438; reinforcement, S-glass 181 fabric, parallel laminated, regular density
SC-E	Resin, Scotchply 1002; epoxy reinforcement, unwoven E-glass fiber with alternate plies oriented at ±5° to the principal axis
SC-S	Resin, Scotchply 1002; epoxy reinforcement, unwoven S-glass fibers with alternate plies oriented at ±5° to the principal axis
EP-E	Resin, epoxy ERSB-0111; reinforcement, E-glass 181 fabric, parallel laminated
EP-S	Resin, epoxy ERSB-0111; reinforcement, S-glass 181 fabric, parallel laminated

Figure 169. Thermal conductivity - mean temperature curves for aircraft plastics. (Values in parentheses are specific gravities) (85)

- Conductivity is a function of density and formulation as well as temperature. Conductivities increase with increased temperature and density. (Den S-L versus Den S-R.)

- Use of higher conductivity reinforcements will result in higher conductivity laminate. SC-E and SC-S were essentially the same material except for the difference in reinforcement (E-glass and S-glass respectively). The conductivity curves of both laminates were almost parallel at all temperatures. However, the SC-E laminate (with the lower conductivity reinforcement factor) gave the lower curve.

SECTION XVI. MELAMINE

Mechanical Properties

Table 74 presents tensile and impact data for two melamine laminates and two molded melamines at -65°, -40°, +10° and +77° F. (9)

Tensile determinations were obtained on the Tinius-Olsen Plastiversal Testing Machine at a constant loading rate of 2500 psi per minute except for the glass-fabric laminate which was tested at 8000 psi per minute. The temperature was controlled to within +2° F. All specimens were conditioned for two hours before testing. Impact tests were run on a Baldwin-Southmark pendulum type

TABLE 74. MECHANICAL PROPERTIES OF FILLED AND LAMINATED MELAMINE AT VARYING TEMPERATURES (9)

Property (5)	Temp °F	Melamine (1) Glass	Melamine/(2) Asbestus	Material Melamine Formaldehyde (3) Electric	Melamine Formaldehyde (4) Alpha
Tensile Strength, Kpsi	77	32.5	13.5	5.4	7.8
	10	32.9	14.5	6.7	6.9
	-40	38.1	15.4	5.7	6.9
	-65	37.2	14.1	5.6	6.7
Modulus of Elasticity, psi x 10^6	77	2.130	2.146	1.060	1.270
	10	2.290	3.040	1.610	1.640
	-40	1.430	3.390	1.540	1.730
	-65	1.580	3.060	1.390	1.880
Elongation at Break, %	77	2.15	0.82	0.54	0.62
	10	2.17	0.64	0.50	0.44
	-40	2.36	0.60	0.40	0.39
	-65	2.75	0.55	0.38	0.37
Work to Product Failure, ft lb in.^{3}	77	31.0	-	1.32	2.08
	10	36.7	-	1.40	1.33
	-40	44.3	-	1.02	0.86
	-65	52.2	-	0.97	1.13
Proportional Limit, Kpsi	77	15.8	14.5	33.2	25.9
	10	7.1	9.5	10.5	9.5
	-40	5.4	6.7	5.7	5.6
	-63	7.8	6.9	6.9	6.7
Izod Impact Strength, ft lb in notch. (ASTM D256-41T)	77	11.12	0.98	0.33	0.31
	10	12.64	0.90	0.37	0.28
	-40	13.43	0.96	0.32	0.28
	-65	14.68	0.93	0.28	0.29

(1) Laminated melamine glass fabric base.
(2) Laminated melamine asbestos paper base.
(3) Melamine-formaldehyde, cellulose filler, electrical grade.
(4) Melamine-formaldehyde, alpha cellulose filler.
(5) Average values.

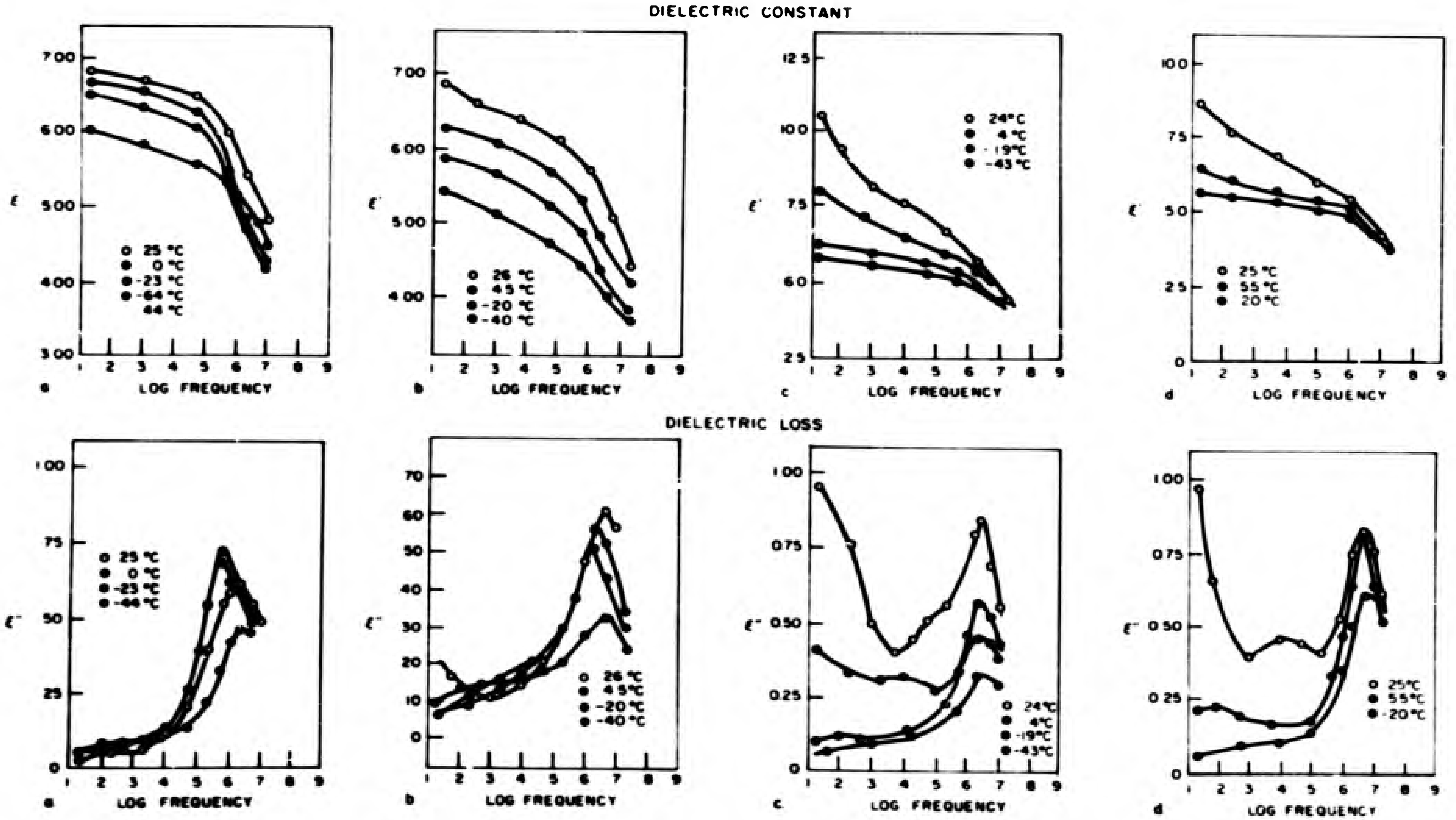


Figure 170. Dielectric constant and loss of melamines as a function of frequency and temperature (86)

Note:

a. 100% Melamine-formaldehyde resin

b. 35% Pentaerythritolsebacate + 1% zinc stearate

c. 10% Polyesteramide + 1% zinc stearate

d. 10% Nylon FM-21 + 5% mono o-cresylglycerylether + 1% zinc stearate impact machine conforming to ASTM D 256-41 T.

Generally, tensile strength, modulus of elasticity and proportional limit increased with decreasing temperature. With the exception of the glass-laminate, elongation at break, work-to-produce failure and impact strength decreased with descending temperature. The glass reinforcement had a decided effect on elongation and impact. These increased with decreasing temperature. The increase in elongation was small but the influence of the reinforcement on impact strength was significant. The increase in impact strength with decreasing temperature is an outstanding property of the glass reinforced materials.

Impact strength of molded melamine was low and both these and the paper laminate were virtually unaffected by decreasing temperatures.

Electrical Properties

The effects of frequency and temperature on the dielectric constant and dielectric loss of plasticized melamine moldings were determined. (86) Measurements were made on a Schering bridge at four temperatures ranging from approximately room temperature to -40° C. The temperature was controlled by circulating cooled or heated toluene through the double-walled jacket of a thermostat surrounding the sample cell. To prevent condensation on the cell leads at low temperatures, a current of dry nitrogen was passed through the space about the cell. The data are plotted in Figure 170.

SECTION XVII. PHENOLICS

Mechanical Properties

Tables 75 through 80 and Figures 171 through 173 present data on the effect of temperature on phenolic materials.

TABLE 75. EFFECT OF TEMPERATURE ON GLASS-FILLED PHENOLICS*(87)

Property	Temp °F	Value
Tensile Strength, psi	160	5,600
	72	7,000
	-50	9,750
Flexural Strength, psi	160	19,000
	72	20,000
	-50	30,000
Compressive Strength, psi	160	13,500
	72	16,500
	-50	18,500
Impact Strength Ft. lbs inch notch; Izod 1/4" bar	72	15.0
	-50	17.0

*Std ASTM methods, Specimens conditioned 2 hrs at test temp.

TABLE 76. EFFECT OF TEMPERATURE ON STRENGTH PROPERTIES OF PHENOLIC MATERIALS (87)

	100°F	0°F	-100°F
Tensile Strength, psi - ASTM D638			
General-Purpose Phenolic Wood-Fiber Filled	5,450	6,050	6,250
Shock-Resistant Phenolic Cotton-Fiber Filled	5,650	6,200	6,400
Heat-Resistant Phenolic Asbestos-Fiber Filled	5,400	6,000	6,900
Glass Fiber-Filled Phenolic	6,500	6,700	7,500
Flexural Strength, psi			
General-Purpose Phenolic	9,800	11,900	13,700
Shock-Resistant Phenolic	13,200	15,300	16,000
Heat-Resistant Phenolic	9,800	11,200	14,000

TABLE 77. MECHANICAL PROPERTIES OF (DUREZ)*GLASS-FILLED PHENOLIC MOLDING MATERIALS (88)

Property	Temp°F	16771-1"	16771-1/2"	16221-1"	21288-1/2"
Tensile Strength psi, D638	160	5,600	-	8,600	5,100[1]
	72	7,000	5,600	11,000	5,260
	-50	9,750	5,000	12,750	6,200[2]
Tensile Modulus psi, 10^6	200	-	-	-	1.7
	72	-	-	-	2.0
	-40	-	-	-	2.47
Flexural Strength psi, D790	160	19,000	-	24,000	14,600[1]
	72	20,000	13,200	25,000	17,000
	-50	30,000	20,000	37,000	21,500[2]
Flexural Modulus psi, 10^6	200	-	-	-	1.6
	72	-	-	-	1.9
	-40	-	-	-	2.2
Compressive Strength psi D695	160	13,500	-	12,000	-
	72	16,500	-	25,000	-
	-50	18,500	-	26,000	-
Impact Strength D256 ft. lb/inch	160	-	-	-	-
	72	15.0	7.2	-	-
	-50	17.0	11.2	-	-

*glass-filled, one stage compounds, 1" and 1/2" rovings
Specimens conditioned 2 hrs at test temperature

[1] - 200°F

[2] - -40°F

TABLE 78. PROPERTIES OF (FIBERITE)PHENOLIC MOLDING MATERIALS AT LOW TEMPERATURE (89)

Material*	Conditioned and Tested At	Tensile Strength P.S.I.	Flexural Strength P.S.I.	Impact Strength Ft. Lbs./Inch
FM-1303	+170°F	6540	10200	0.57
	+ 70°F	9220	12380	0.51
	- 60°F	8460	12980	0.48
FM-3510	+ 70°F	6750	13830	2.08
	- 20°F	7590	13930	1.50
	- 40°F	7200	13680	1.45
FM-4030	+ 70°F	8770	20350	20.4
	- 20°F	10000	22560	22.5
	- 40°F	9040	22390	26.3
FM-4035	+ 70°F	8650	28700	27.5
	- 20°F	9670	28780	30.5
	- 40°F	9060	29570	31.7

* Fiberite FM-1303 - a nylon reinforced phenolic compound of improved impact strength
Fiberite FM-3510 - fabric reinforced phenolic. MIL-M-14F, Type CFI-20.
Fiberite FM-4030 - fiber glass reinforced 2 - stage phenolic. MIL-M-14F, type GPI-100.
Fiberite FM-4035 - similar to FM-4030 except one - stage resin.

TABLE 79. EFFECT OF TEMPERATURE AND POST-BAKE OPERATION ON FLEXURAL PROPERTIES OF GLASS-FILLED PHENOLICS (90)

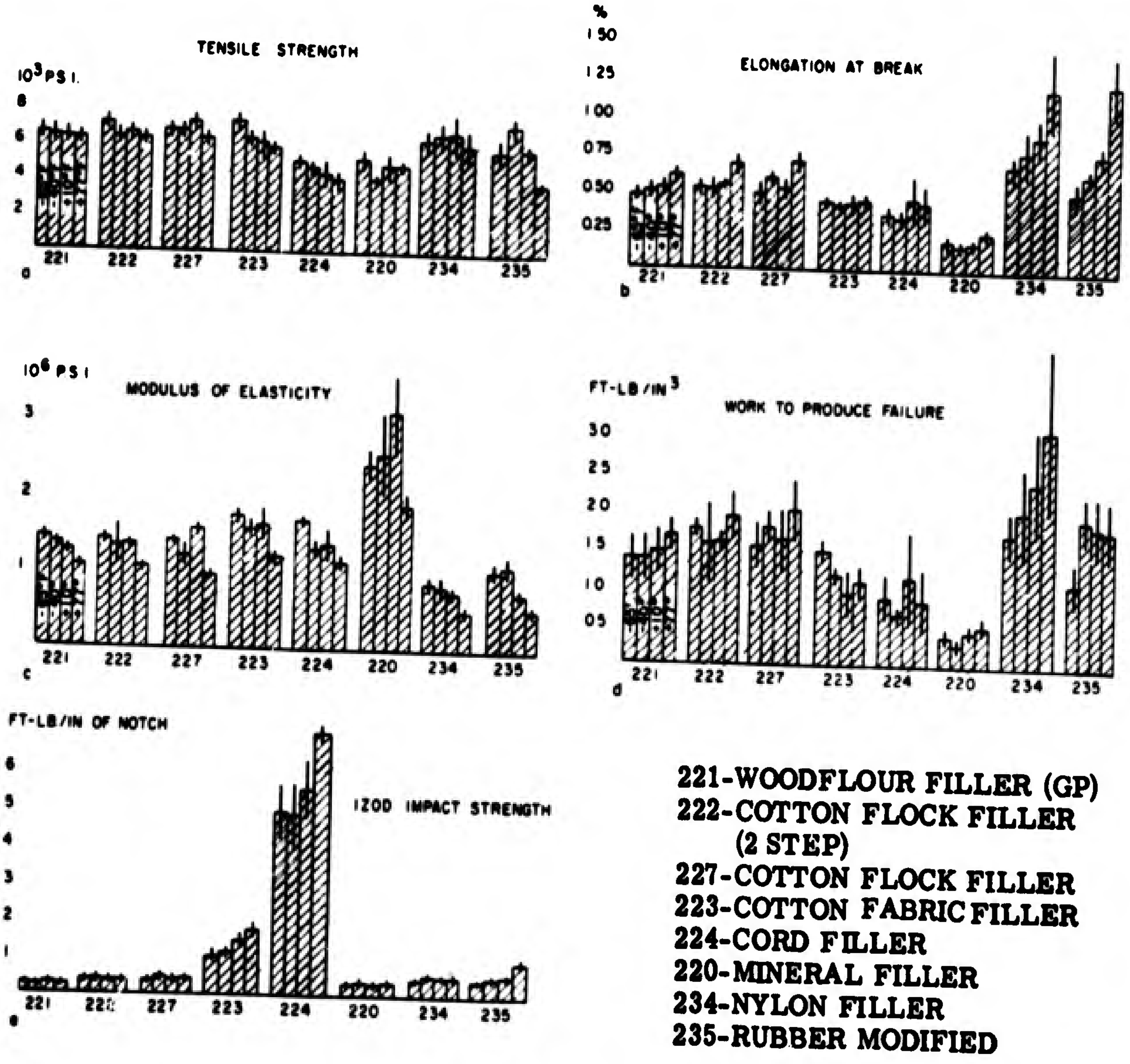
Phenolic Glass-Filled	Flexural Strength	Flexure in Modulus
As Molded		
-40°F	21,500	2.2×10^6
72°F	17,000	1.9×10^6
200°F	14,500	1.6×10^6
Post-Baked 24 Hrs. at 390°F in Tempering Oil		
-40°F	15,000	2.1×10^6
72°F	14,400	2.0×10^6
200°F	14,100	2.0×10^6

Note: 2 inch rovings

TABLE 80. TENSILE PROPERTIES OF COMPRESSION AND TRANSFER PHENOLIC MOLDINGS* VS TEMPERATURE (90)

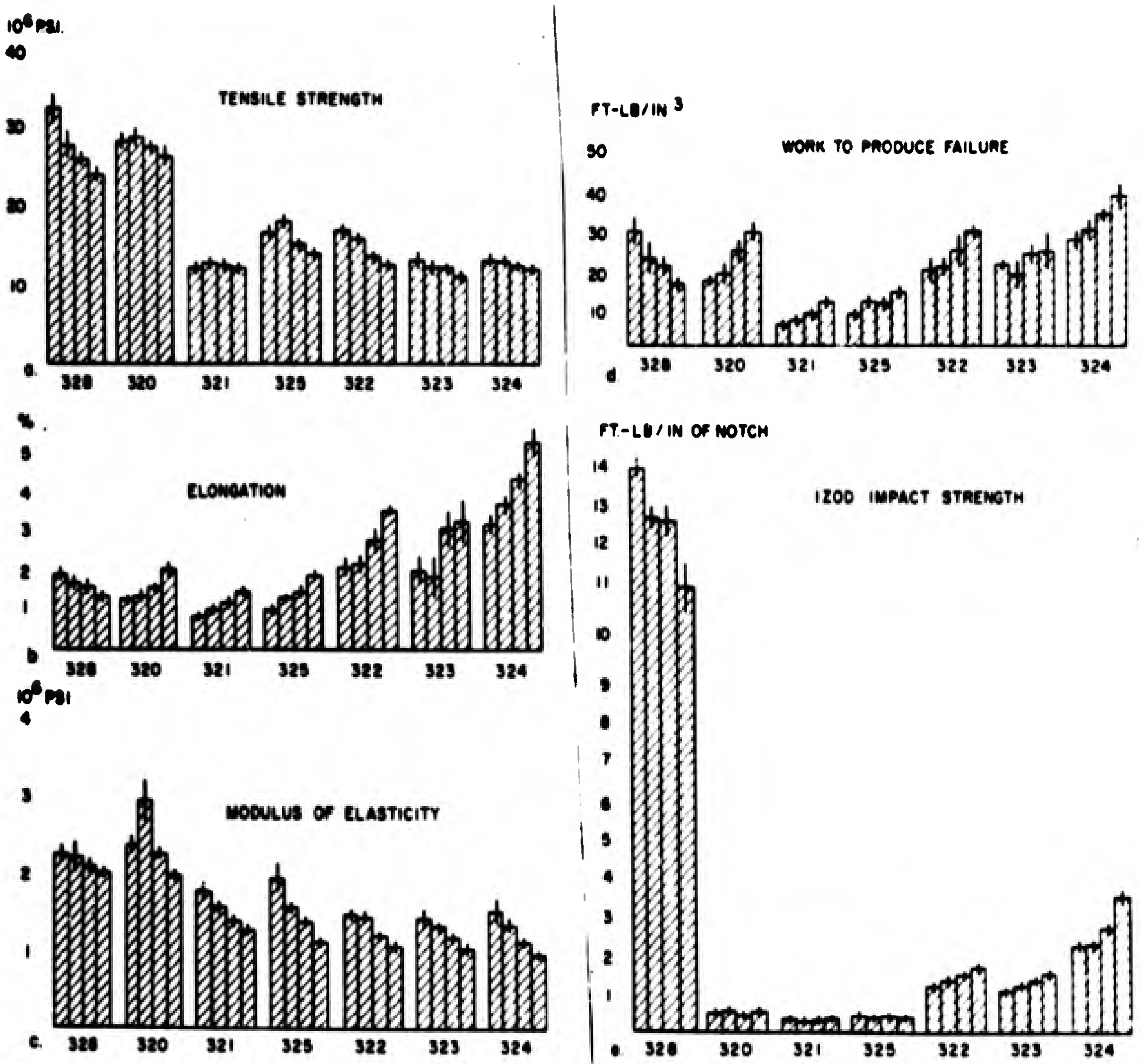
Temperature	Compression		Transfer	
	Tensile, p.s.i.	Modulus x 10^6	Tensile, p.s.i.	Modulus x 10^6
-35°F	6,200	2.47	6,300	2.49
25°F	5,600	2.09	6,000	2.08
75°F	5,300	2.11	5,000	1.75
350°F	3,800	1.81	4,500	1.53

*glass-filled phenolic; 2" rovings; data on "as molded" specimens



221-WOODFLOUR FILLER (GP)
222-COTTON FLOCK FILLER (2 STEP)
227-COTTON FLOCK FILLER
223-COTTON FABRIC FILLER
224-CORD FILLER
220-MINERAL FILLER
234-NYLON FILLER
235-RUBBER MODIFIED

Figure 171. Mechanical properties of phenolic molding materials (18)



328-Phenolic-Glass
320-Phenolic-Paper
321-Phenolic-Paper
325-Phenolic-Paper
322-Phenolic-Paper
323-Phenolic-Fabric (L)
324-Phenolic-Fabric (C)

Figure 172. Mechanical properties of phenolic laminate materials (18)

DESCRIPTION OF PLASTIC MATERIALS TESTED

Code No.	Material	Classification MIL-P-15035A	Description
		a. Glass Fabric Base	
328	Laminated phenolic, glass fabric base	NEMA G-1	Representative, medium glass fabric laminate, heat resistant
		b. Paper Base	
320	Laminated phenolic, paper base	PBG	Representative paper base laminated sheet; 40% phenolic resin
321	Laminated phenolic, alpha-cellulose paper base	PBE	Alpha-cellulose paper base laminate; maximum moisture resistance, minimum cold flow, 60% resin
325	Laminated phenolic, alpha-cellulose paper base	PBG-P	Alpha-cellulose paper base laminate; treated with penetrating and laminating resin; punching stock
		c. Cotton Fabric Base	
322	Laminated phenolic, fabric base	FBI NEMA L	Representative fabric base laminate; 4-oz. fabric base, 48 to 54% resin
323	Laminated phenolic, fabric base	FBE NEMA LE	Representative electric grade; 4-oz. fabric base, high resin content; fabric thoroughly dried; high moisture resistance
324	Laminated phenolic, fabric base	FBM NEMA C	Representative high impact strength laminate; 12-oz. fabric base, 40% resin

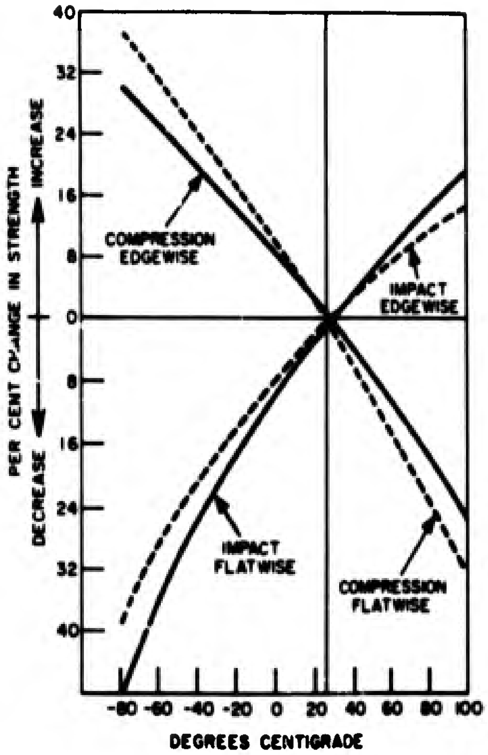


Figure 173. Effect of temperature and direction on compression and impact properties of a phenolic (Micarta 262) laminate (NEMA grade-C-reinforced with coarse cotton weave fabric; MIL-P-15035 FBM) (91)

The above data indicate that the mechanical properties of filled molded phenolics behave as do other plastics with decreasing temperature. The influence of the reinforcement on impact strength, however, is noteworthy. As previously stated, glass-filled materials have the highest impact strength and the strength increases with decreasing temperature. The impact strengths of other filled materials are low (with the exception of cord and cotton fabric fillers) and are little influenced by temperature. Those with the cotton fabric and cord filler perform as expected, i.e., the strength decreases with decreasing temperature.

The laminate results are as expected. Tensile strength and modulus of elasticity increase with decreasing temperature; laminates with glass reinforcement had the highest strengths; and work-to-produce failure, elongation and impact strengths increased with glass reinforced laminates at lower temperatures but decreased for all others. Again this substantial increase in impact strength at low temperatures is one of the outstanding properties of glass reinforced laminates.

The effect of load direction and temperature on laminates is illustrated in Figure 173. Impact strength below room temperature is greater parallel to the laminations (edgewise) than perpendicular to the laminations. The reverse is true at temperatures above 23°C. Compression strength at low temperatures is higher perpendicular to the laminations (flatwise) than parallel to the laminations. At above room temperatures the compression strength is greater in the edgewise direction.

Electrical Properties

Figure 174 plots the electric strength of a mica-filled phenolic molding versus temperature and frequency.

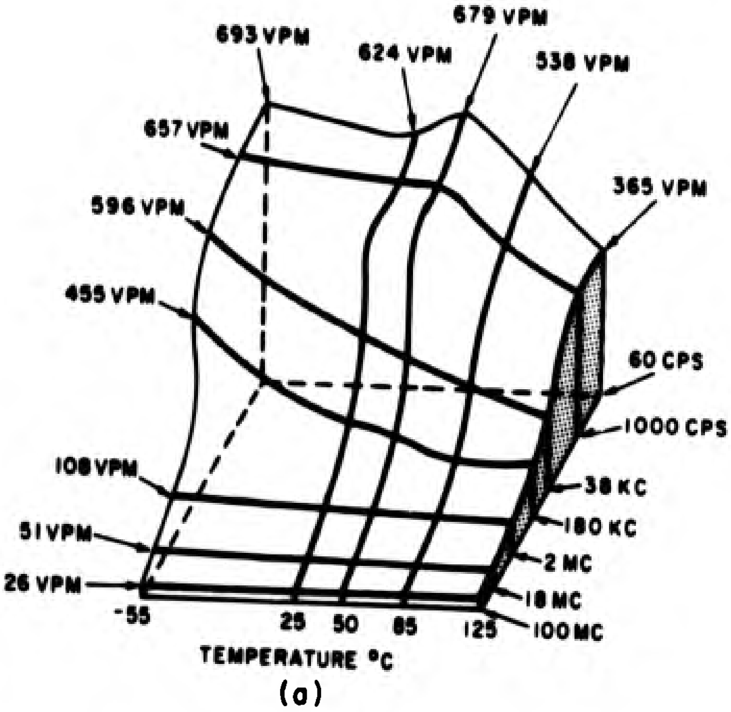


Figure 174. Electrical strength of mica-filled phenolic molding (29)

Thermal Properties

Thermal expansion and conductivity are influenced by many factors in addition to temperature. At the same temperature these will vary depending on resin, cures, post cures, reinforcements, orientations, etc. The complete history will determine its thermal properties.

The total linear thermal expansion of various reinforced phenolics (Table 81) are given in Figure 175. The total expansion was measured in two directions, normal-to-thickness and thickness by a Leitz dilatometer. The results reflect the effect of orientation as previously discussed in the Epoxy Section. The expansion in the thickness direction approaches the sum of expansions of each layer and is usually a value between the two materials. In the normal-to-thickness direction, the higher modulus material will tend to control the overall expansion. This is demonstrated in specimen B where microscopic examination showed excellent bonding. The expansion was almost identical to that of a straight phenolic resin (the higher modulus component of the material). This phenomena can be altered of course by the presence of voids, etc. as was the case in specimen A.

TABLE 81. MATERIALS DESCRIPTION OF REINFORCED PHENOLICS (30)

Identification		A-1	B-1	C-1	N-1	O-1	P-1
Composite System:							
Manufacturer:	Raytheon Company	X	X	X	--	--	--
	Fiberite Corp	--	--	--	X	X	X
Resin source:	Cincinnati T & R Labs	X	X	--	--	--	--
	Monsanto Chem Co.	--	--	X	--	--	--
Prepreg source:	U.S. Polymeric Co.	X	X	--	--	--	--
	National Carbon Co.	--	--	X	--	--	--
Resin content, %		25-27	42-44	30	--	--	--
Resin System:							
Trade name:	CTL-91-LD	X	X	--	--	--	--
	SC-1008	--	--	X	--	--	--
Reinforcement System:							
Material:	Glass cloth ("E" glass)	X	--	--	--	--	--
	Nylon cloth (SN-19)	--	X	--	--	--	--
	Graphite cloth	--	--	X	--	--	--
	Asbestos fibers	--	--	--	X	X	X
Fabric style:		181	YN-25	WCB	--	--	--
Form:	Fabric	X	--	X	--	--	--
	Chopped fabric (1/2" x 1/2")	--	X	--	--	--	--
	Fibers filled with phenolic microballoons	--	--	--	X	X	X
Finish:	Volan "A"	X	--	--	--	--	--
	Heat set and scoured	--	X	--	--	--	--
Lay-up:	Parallel laminate	X	--	X	--	--	--
	Molded	--	X	--	--	--	--
Number of plies:		54	--	52	78	18	--
Orientation:	Random	--	X	--	--	--	X
	Warp in 10" dia.	X	--	X	--	--	--
	Machine direction corresponds to 10" dia.	--	--	--	X	X	--
Cure Cycle:							
Pressure:	2000 psi to 1/2" stops	X	X	X	--	--	--
	25 psi	--	--	--	X	--	--
	200 psi	--	--	--	--	X	--
Temperature, °F		325	325	325	300	300	--
Temperature/time history:							
Preheat to 200°F; raise to 325°F in 1 hr: cure for 1 hr; cool to 300°F		X	--	--	--	--	--
Preheat to 200°F; raise to 300°F in 1 hr: increase to 325°F and cure for 2 hrs; cool to 200°F.		--	X	--	--	--	--
B-stage at 220°F for 15 min; preheat to 200°F; raise temp to 325°F in 1 hr; cure for 1 hr; cool to 200°F.		--	--	X	--	--	--
Preheat 300°F; hold for 30 min; cool to 80°F.		--	--	--	X	X	--
Post-Cure Cycle:							
Time/Temperature:	24 hrs at 350°F in hot air oven	X	X	X	--	--	--

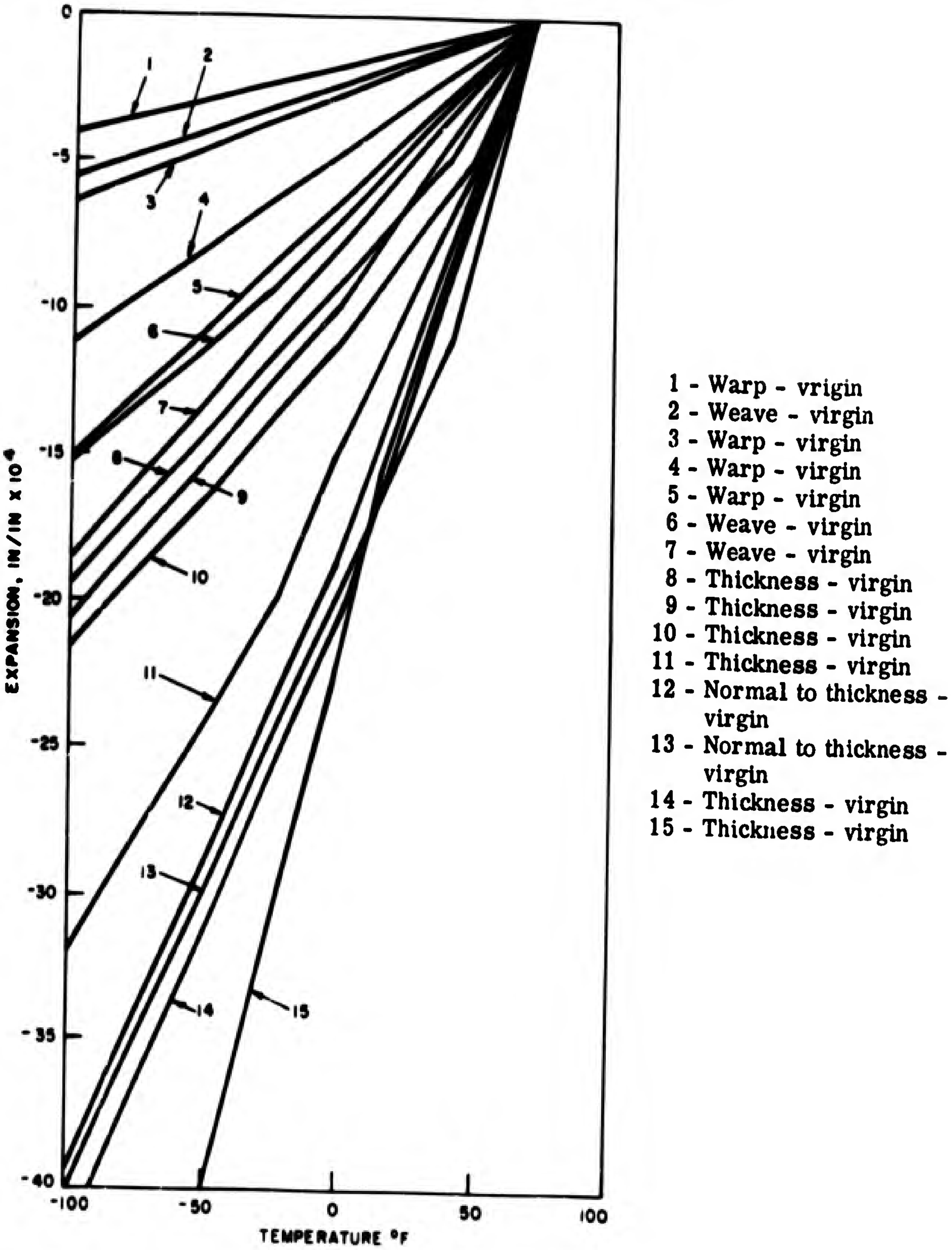
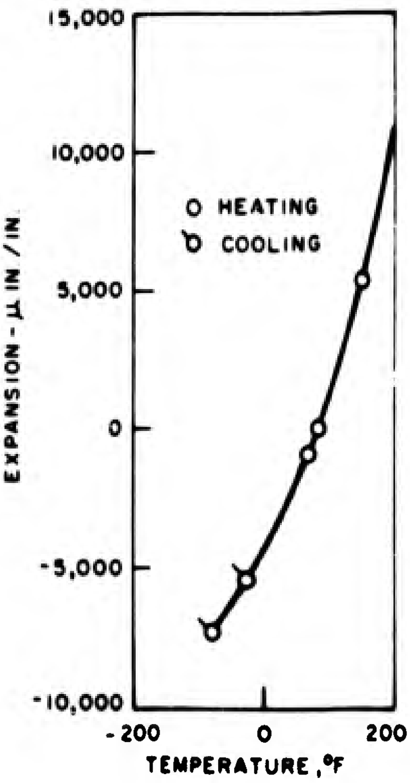


Figure 175. Total linear thermal expansion of reinforced phenolics in the thickness and normal-to-thickness directions.

Cloth reinforcements usually have more threads per inch in the warp than in the weave direction. Therefore, the expansion should be greatest in the weave direction (see specimen C). However, this was not always the case. In fact, differences of 10 to 15 percent were observed between specimens cut in the same direction from the same material. As a result, it is understandable why the small difference due to cloth construction was not always apparent. The specimens are small, and the distance between threads in the reinforcement is very small. For example, there are 57 threads per inch in the warp direction and 54 threads per inch in the fill direction in 181 style glass cloth. Therefore, the difference between expansion in the warp and weave directions was attributed to the cutting of the threads on the edges of the specimen during the machining operation.

Specimen B-1 is reinforced with 1/2" x 1/2" chopped nylon laid parallel to the surface with random orientation in the other direction. Therefore, the expansion should be equal in all directions normal to the thickness.

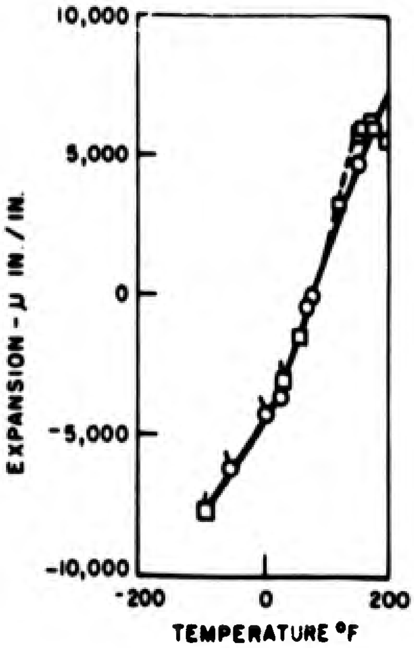
The thermal expansion of a phenolic casting material was determined by a quartz tube dilatometer of Bureau of Standards design (92). The total expansion in the thickness and longitudinal directions are plotted in Figures 176 and 177. In our interested temperature range the total expansion agreed fairly well.



Note:

Density	1.21 Specific Gravity
Resin Percent[1]	100% Monsanto SC1008
Void Content	Nil
Cure Cycle	
Pressure	2000 psi
Temp.	325°F ± 5°F
Time	60 ± 10 Minutes
Post-Cure Cycle	
Time-Temp.	140°F - 48 Hours 200°F - 72 Hours

Figure 176. Thermal expansion in the thickness and longitudinal directions of material f-1 (SC1008 phenolic casting) (92)



Specimen No. 1

○ Heating

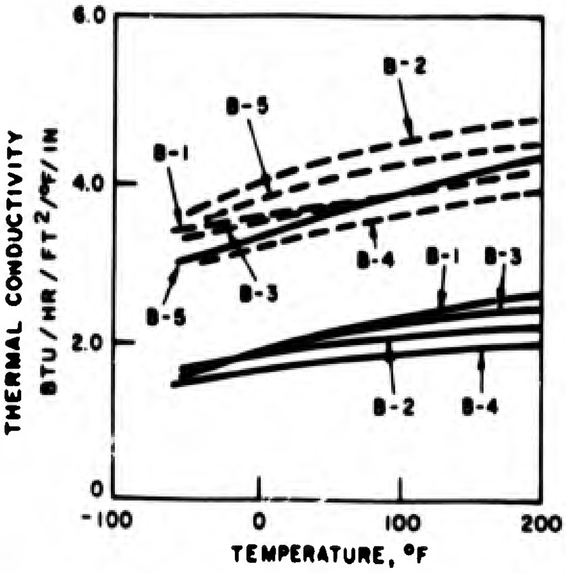
○̀ Cooling

Specimen No. 2

□ Heating

□̍ Cooling

Figure 177. Thermal expansion in the longitudinal direction of material f-1 (SC1008 phenolic casting) (92)



Thickness --------c Direction
Parallel --------a Direction
--------b Direction

Figure 178. Thermal conductivity in the thickness and parallel to the reinforcement direction of "B" materials (phenolic - silica fabric) (83)

Figure 178 plots the conductivity of phenolic-silica reinforced laminates (see Table 82). Average values in the thickness direction ranged from 1.5 at -50°F to 2.3 at 150°F. The values parallel to the reinforcement and 45° layup were twice as high (approximately 3.4 at -50°F to 4.2 at 150°F) as the thickness direction.

TABLE 82. MATERIAL DESCRIPTION OF PHENOLIC/SILICA LAMINATES (83)

Identification		B-1	B-2	B-3	B-4	B-5
Composite System:						
Density (sp gr)		1.80	1.41	1.73	1.41	1.70
Resin content (before cure), %		30	20.6	29.9	19.0	30
Resin content (after complete fabrication), %		27	19.2	26.0	19.6	22.9
Void content, %		17(1)	28(1)	17	25	12(1)
Resin System:						
Trade Name:	Monsanto SC 1008	X	X	--	--	X
	CTL-91LD	--	--	X	X	--
Processing:	Molding temperature, °F	275	275	275	275	--
	Lay-up temperature, °F	--	--	--	--	200
Reinforcement System (Fabric):						
Trade Name:	General Electric Silica (quartz)	X	X	X	X	X
Fabric designation:	U.S. Polymeric - 581-A1100 Quartz SC 1008	X	X	--	--	--
Finish:	A1100	X	X	X	X	X
Lay-up:	Parallel	X	X	X	X	--
	45° to the surface	--	--	--	--	X
Number of plies and orientation:	26 plies, 181 style	X	X	X	X	--
	181 style	--	--	--	--	X
Cure Cycle:						
Pressure, psi		2000	2000	2000	2000	200
Temperature, °F		325	325	325	325	325
Time, hour		1/2	1	1	1	1
Post-Cure Cycle:						
Time/temperature: 24 hrs at 350°F		X	X	X	X	X

(1) By volume.

It should be noted that specimen B1 and B3 had higher conductivities in the thickness direction, opposite to what would be expected. This is attributed to a higher void content than specimen B-2 and B-4 as determined by photomicrographs. Because of the voids, poor bonding was suspected. Even with the strong influence of the void structure, the CTL-91 LD phenolic laminates had a slightly lower conductivity than the SC 1008 phenolics. Although the conductivity should theoretically increase with decreasing resin, it was the opinion of the authors that there is a minimum below which the resin does not provide a continuous matrix between the reinforcements and conductivity decreases with further decreases in resin. This was believed to be at about 25 percent. However, it was explained that poor bonding and voids would have the same effect as resin contents.

Figure 179 illustrates the effect of fabric lay-up on the conductivity of 50 percent phenolic-graphite laminates. The conductivity increased from across the reinforcement (thickness direction), to 45° to the reinforcement, to parallel or with the heat flow. The difference between the conductivities of A-7 and A-3 parallel to the reinforcement was attributed to the greater void content of A-7.

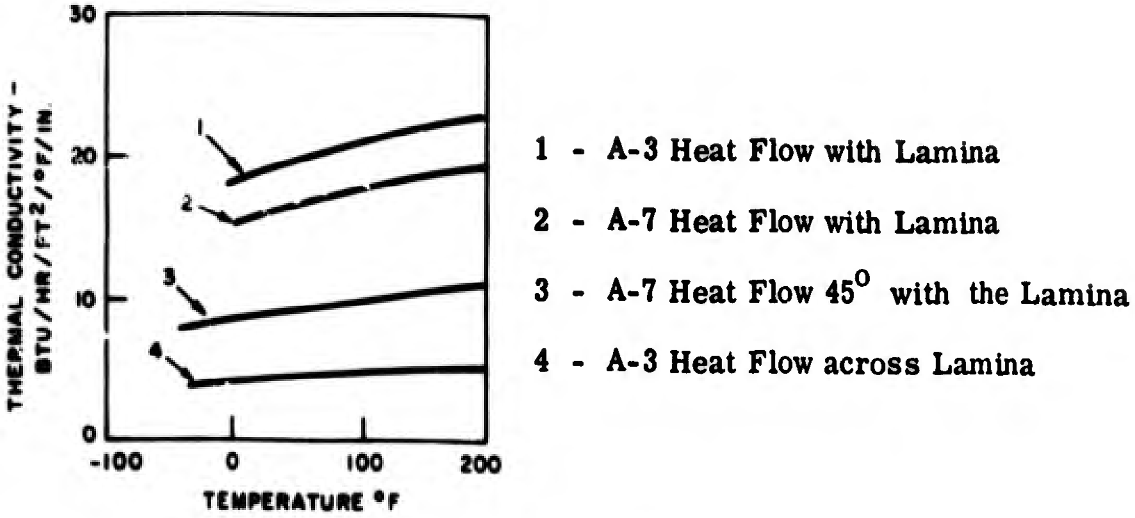


Figure 179. Effect of fabric lay-up on thermal conductivity for a 50 percent SC1008 phenolic-WCB graphite fabric (92)

Figure 180 also illustrates the influence of fabric orientation on the thermal conductivity of phenolic laminates. The conductivity of the laminates, with regular layup, increased as the orientation of the heat flow direction to the fabric was varied from normal to the panel, to 45° to normal, to 90° to normal to the panel (edgewise).

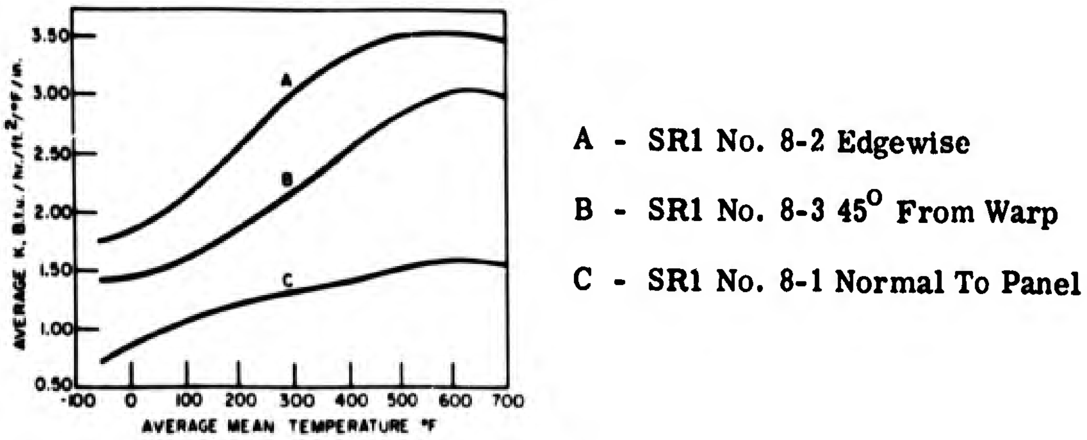


Figure 180. Influence of fabric orientation on conductivity (CTL 37-9X phenolic-glass) (84)

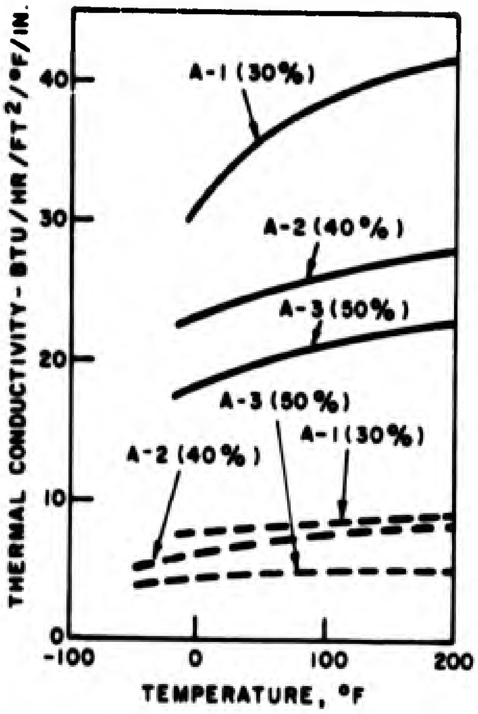
The thermal conductivities of phenolic laminates were obtained on a guarded hot plate with a slightly modified ASTM C177-45 method (See Epoxy Section). Figures 181 and 182 compare the thermal conductivities of phenolic laminates reinforced with continuous graphite fabric and graphite squares. (See Table 83.) Data was obtained in both the thickness direction and parallel-to-reinforcement direction.

TABLE 83. MATERIAL DESCRIPTION OF PHENOLIC/GRAPHITE PANELS (83)

Identification		A-1	A-2	A-3	A-4	A-5	A-6
Composite System:							
Density (sp gr)		1.44	1.43	1.38	1.44	1.42	1.39
Resin content (before cure), %		30	40	50	30	40	50
Resin content (after complete fabrication) %		29	38.2	52	29.1	38.1	48.1
Void content		Nil	Nil	Nil	Nil	Nil	Nil
Resin System (Phenolic):							
Trade Name:	Monsanto SC 1008	X	X	X	X	X	X
Reinforcement System (Graphite):							
Trade Name:	National Carbon Co., WCB Graphite	X	X	X	X	X	X
Fabric designation:		WCB	WCB	WCB	WCB	WCB	--
Form:	Fabric	X	X	X	--	--	--
	Fabric squares (1/2" x 1/2")	--	--	--	X	X	X
Lay-up:	Parallel to surface	X	X	X	--	--	--
	Random	--	--	--	X	X	X
Number of plies and orientation:							
9 plies, 90° to molding pressure		X	X	--	--	--	--
8 plies, 90° to molding pressure		--	--	X	--	--	--
Random		--	--	--	X	X	X
Cure Cycle:							
Pressure, psi		1700	1950	1700	2000	1700	800
Temperature, °F		300	300	300	300	310	300
Time hour		1/2	1/2	1/2	1/2	1/2	1/2
Post-Cure Cycle:		None	None	None	None	None	None

The greatest increase in thermal conductivity was between -50°F and room temperature. Comparing the conductivities of the chopped fabric reinforced laminate with the continuous fabric, the chopped fabric panels had higher conductivities across the ply than the other laminates with the same amount of resin. This was attributed to a better conductive heat path as a result of the overlapping fabric. Photomicrographs of the continous fabric laminate showed good resin-to-graphite bonding, virtually void free. Conductivity, therefore, increased with percentage of graphite fabric.

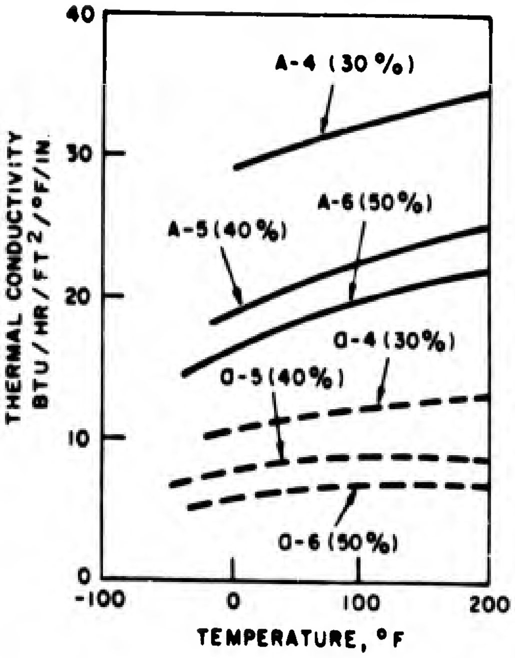
Figure 183 charts the conductivities of uncoated and coated carbonized phenolic-graphite composites. The coated carbonized laminate was higher in both directions. These higher values resulted from the dispersion of the impregnation and silicon-silicon carbide coating in the material. (92)



—— Parallel to Reinforcement Direction

---- Thickness or Across Lamina Direction

Figure 181. Thermal conductivities in both the thickness and parallel to the reinforcement directions of materials A-1 through A-3 (SC1008 phenolic-WCB continuous graphite fabric) (92)

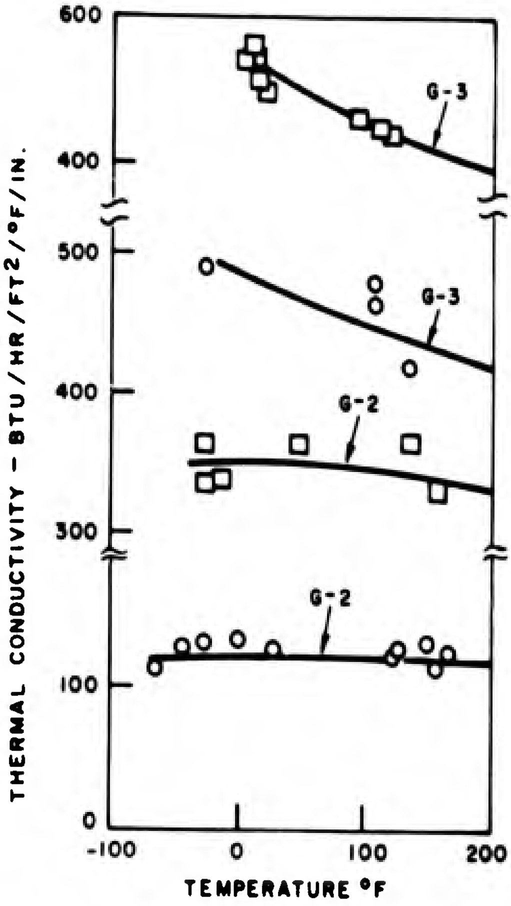


—— Parallel to Reinforcement Direction

---- Thickness or Across Lamina Direction

Figure 182. Thermal conductivities in both the thickness and parallel to the reinforcement directions of materials A-4 through A-6 (SC1008 phenolic-WCB graphite fabric squares 1/2" x 1/2") (92)

Thermal conductivities of carbon and graphite-phenolic laminates parallel to the reinforcement are compared in Figure 184. (See Table 84.) Specimen I-1 compared well to A-2, a similar material previously discussed. The values for I-2 and I-3 were considerably lower than I-1 due to the differences in thermal conductivity between carbon and graphite.



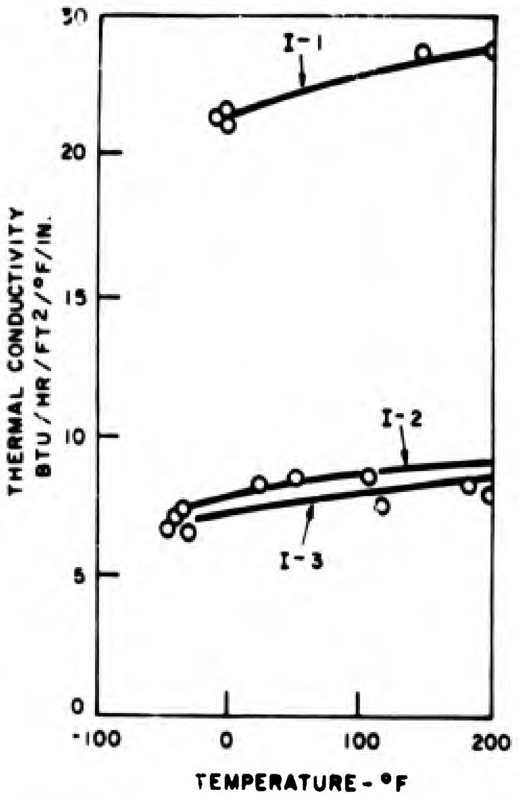
○ = Parallel to Reinforcement Direction (with Lamina)

□ = Thickness Direction (across Lamina)

NOTE: Material G-2 is a composite of phenolic (R-120) and graphite cloth (National Carbon - WCA), square weave fabric, in lay-up parallel to surface, 66 plies, crossplied. Cure was 2 hours under 400 psi, 325°F, post-cured at 500° F during 40 hours interval. Density is nominal - 74 lb/cu ft avg; edge-grain - 75 lb/cu ft avg. Resin content is 56%, void content is 27% by volume.

Material G-3 is a composite identical to G-2, but Si-SiC coated. Density of the uncoated laminate was: nominal - 75.5 lb cu ft avg; edge-grain - 68 lb/cu ft avg; Density of the coated laminate is: nominal - 122 lb/cu ft avg; edge-grain - 123 lb/cu ft avg. Resin content is 56%. Void content is not applicable.

Figure 183. Thermal conductivity in the thickness and parallel-to-reinforcement directions of material G-2 (uncoated carbonized phenolic composite) G-3 (Si-S-C coated carbonized phenolic composite) (92)



Material I-1 (40% SC1008 Phenolic-HITCO G Graphite Fabric)

Material I-2 (30% SC1008 Phenolic-CCA-1 Carbon Fabric).

Material I-3 (30% SC1008 Phenolic-Pluton B-1 Carbon Fabric).

Comparative Rod Apparatus
Pyroceram Reference
O Specimen 1

Figure 184. Thermal conductivity in the parallel to the reinforcement direction of material I-1, I-2 and I-3 (92)

TABLE 84. MATERIAL DESCRIPTION OF CARBON AND GRAPHITE/PHENOLIC LAMINATES

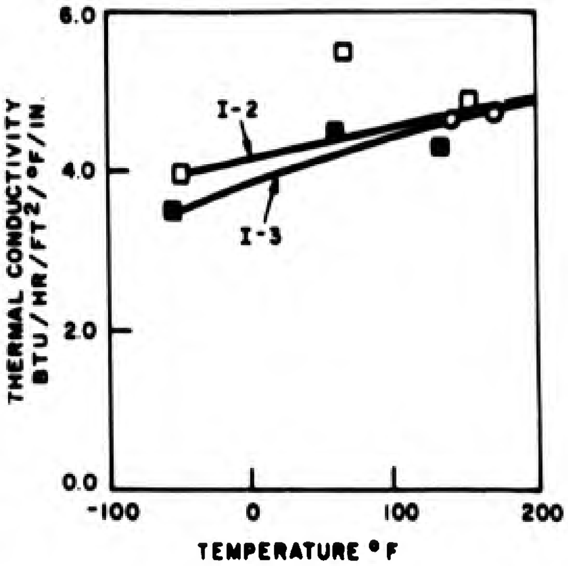
Identification		I-1	I-2	I-3
Composite System:				
Density:	lb/cu ft	84.9	88.2	--
	Specific gravity	--	--	1.437
Resin content (before cure), %		40	30-35	38
Resin content (after fabrication), %		43.5	---(1)	30
Void content, %		Nil	Nil	Nil
Resin System (Phenolic):				
Trade Name:	Monsanto SC 1008	X	X	X
Reinforcement System:				
Trade Name:	HIT Company-G-Graphite	X	--	--
	CCA-1 Carbon	--	X	--
	3M Company - Pluton B-1 (Carbon)	--	--	X
Fabric designation		None	None	B-1
Lay-up:	Parallel to surface	X	X	X
Number of plies, and orientation:				
30 plies, warp aligned		X	--	--
18 plies, warp aligned		--	X	--
110 plies/inch, warp aligned		--	--	X
Cure Cycle:				
Pressure, psi (2)		None	None	1000
Time at pressure, minutes (2)		240	240	---
Pressure, psi (3)		500	500	---
Time at pressure, minutes (3)		50	50	--
Temperature, °F		325	325	325
Time at temperature, minutes		290	290	45
Post-Cure Cycle:		None	None	None

(1) Not determinable

(2) and (3) Consecutive operations

Two carbon reinforced laminates, measured in the thickness direction, are compared in Figure 185. The conductivities were very similar.

The thermal conductivities of reinforced phenolics (see Table 81) using the guarded hot-plate method at one atmosphere nitrogen and helium gas are plotted in Figures 186 through 189. Figure 190 shows the difference in conductivities between air and helium for a phenolic-glass laminate.



Material I-2 (30% SC 1008 Phenolic-
CCA-1 Carbon Fabric)

Material I-3 (30% SC 1008 Phenolic-
Pluton B-1 Carbon Fabric)

3-Inch Apparatus

☐ Gum Rubber Filler

○ Fiberfrax Filler

Figure 185. Thermal conductivity in the thickness direction of materials I-2 and I-3 (83)

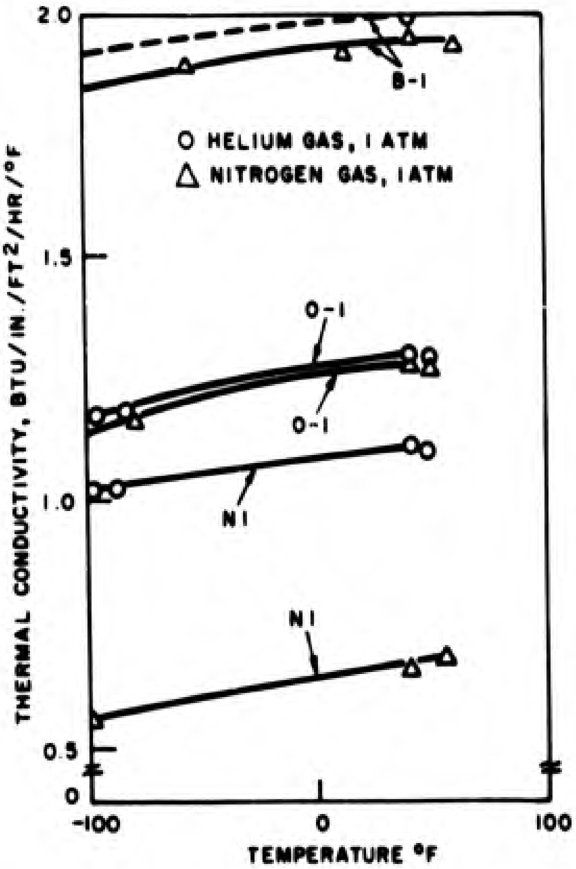


Figure 186. Thermal conductivity of phenolic panels, thickness direction (30)

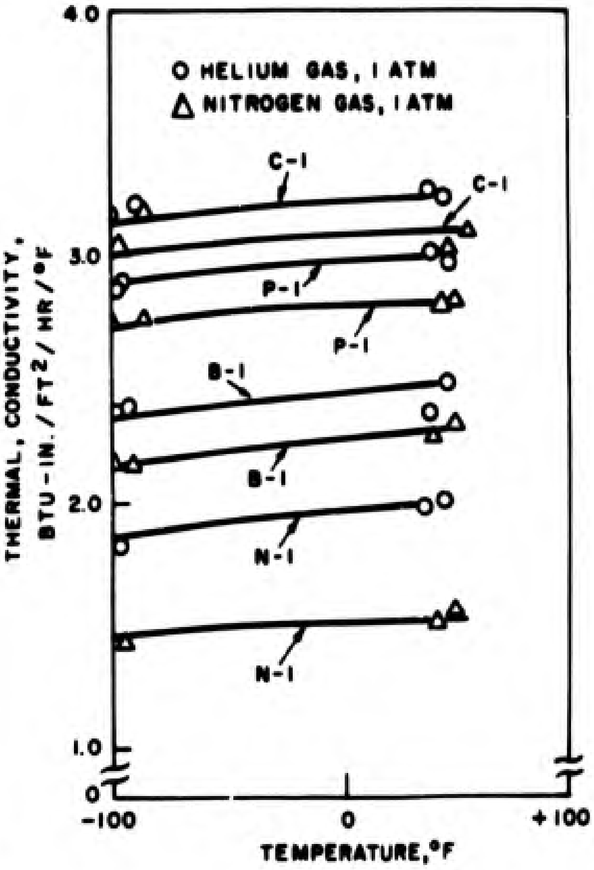


Figure 187. Thermal conductivity of phenolic panels, perpendicular to thickness direction (30)

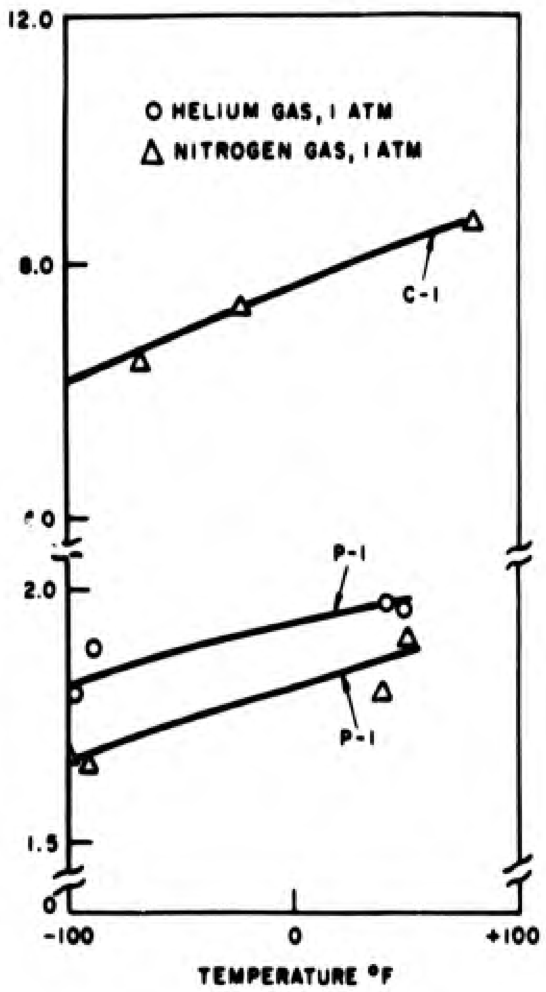


Figure 188. Thermal conductivity of phenolic laminates (C-1 and P-1 materials)in thickness direction (30)

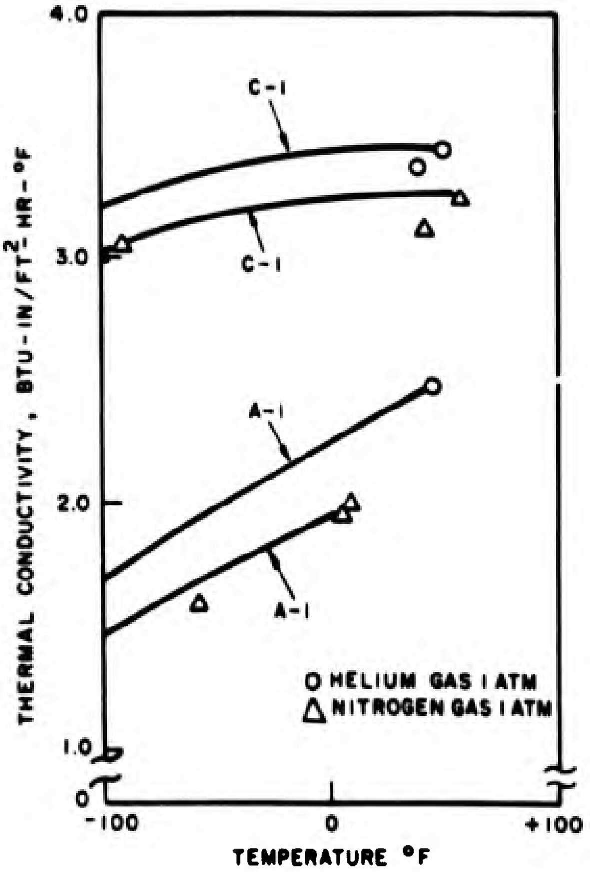


Figure 189. Thermal conductivity of phenolic laminates (A-1 and C-1 materials) in warp direction (30)

The influence of environment on thermal conductivity is well demonstrated. The difference in the K factor between the nitrogen and helium atmospheres is the result of two factors: (1) the reduction of contact resistance with helium and (2) the presence of the relatively high conductivity helium gas within the voids of the material.

It was pointed out that there is presently no analytical model available which can approximate the relative magnitude of these two factors. Also, separating these factors is complicated since contact resistance varies with each set-up.

The thermal conductivities varied normally with direction. The high conductivity of C-1 is the result of the higher K factor of the reinforcement. Graphite fiber has a conductivity 100 times greater than "E" glass. (30)

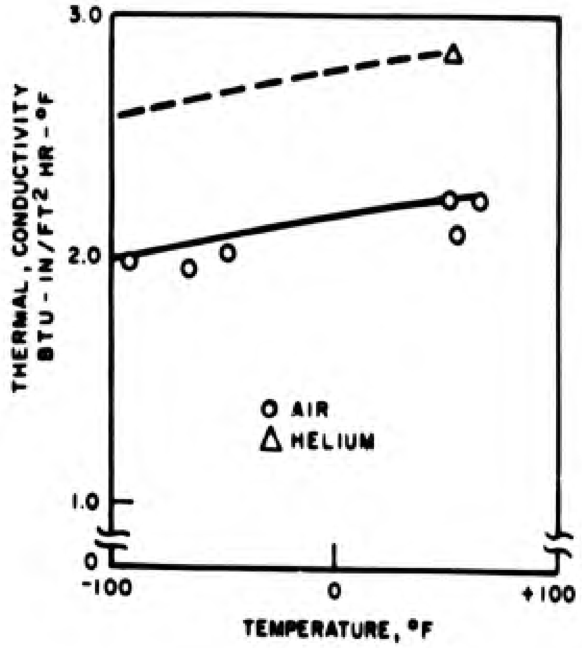


Figure 190. Thermal conductivity of phenolic laminate (A-1 material) in thickness direction (30)

The N and O materials are similar with the exception of fabrication pressure. The higher pressure material (O) resulted in higher density and consequently higher conductivity. The lower density material (N) had greater variations in conductivities between the helium and nitrogen. This was expected since helium would have a greater influence in a more porous material.

As a means of comparing thermal conductivities Figure 191 presents the thermal conductivity of a phenolic casting material. Values ranged from 1.35 Btu/hr/ft²/°F/in at -50°F to about 1.6 at 130°F.

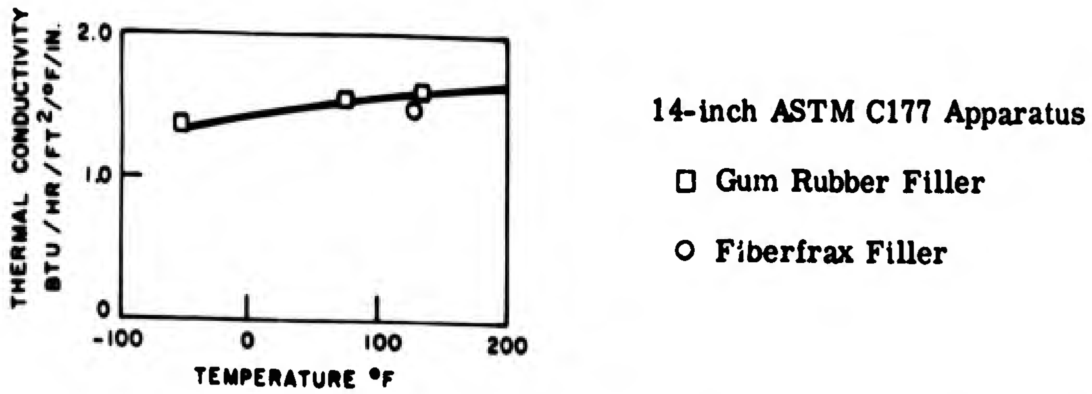


Figure 191. Thermal conductivity in the thickness direction of phenolic casting (Material F-1, SC1008) (92)

SECTION XVIII. POLYBENZIMIDAZOLES

Polybenzimidazoles are a new family of resins and are now commercially available for laminates and adhesives. They are also known for their high temperature attributes which make data additionally scarce for this report.

Mechanical Properties

The tensile, compressive, flexural and interlaminar shear properties of polybenzimidazole laminates reinforced with 1581-994S glass fabric were studied. (93) Tests were run in accordance with ASTM procedures with the following exceptions: the size of the tensile specimen was 6-1/2" x 1/2" with a 1/4" test width and the interlaminar parallel shear specimens were of a Sandia Corp. design. The data in Table 85 are the average of five test specimens after 1/2 hr. temperature conditioning.

TABLE 85. MECHANICAL PROPERTIES OF POLYBENZIMIDAZOLE 1581/994S GLASS LAMINATES (93)*

Property	Temperature		
	-65°F	75°F	250°F
Tension			
Yield, 10^3 psi	53.9	53.5	44.9
Ultimate, K psi	100.8	87.0	77.1
Retention, %	115	100	88
Modulus, 10^6 psi	5.08	4.74	4.64
Modulus retention, %	107	100	92
Compression			
Ultimate, 10^3 psi	70.9	70.1	68.9
Retention, %	101	100	97
Modulus, 10^6 psi	5.40	5.40	4.88
Modulus retention, %	100	100	90
Flexure			
Ultimate, 10^3 psi	121.0	111.8	103.2
Retention, %	108	100	93
Modulus, 10^6 psi	4.66	4.82	4.60
Modulus retention, %	97	100	96
Interlaminar shear			
Ultimate, psi	6810	6275	6290

*approximately 80% glass content

Table 86 gives the compressive and flexural properties of 181-994 glass cloth laminate.

TABLE 86. MECHANICAL PROPERTIES OF POLYBENZIMIDAZOLE 181 - 994 GLASS CLOTH LAMINATES* (122)

(Short-Time Exposure at Test Temperatures)

Test Condition, °F	Compression Strength psi	Compression Modulus, psi x 10^6
-65	71,000	5.4
Room Temperature (77)	70,000	5.4
250	69,000	4.9

(Long-Time Aging Strength at Test Temperatures)

Test Condition, °F	Compression Strength, psi	Compression Modulus, psi x 10^6
-65	78,000	6.1
Room Temperature (77)	77,000	6.0
RT after 2 hr water boil	50,000	5.5

(Short-Time Exposure at Test Temperatures)

Test Condition, °F	Flexural Strength, psi	Flexural Modulus, psi x 10^6
-65	140,000	6.0
77	135,000	5.5
77 after 2-hr water boil	90,000	4.0
250	112,000	4.8

*Prepreg Formulation:

Resin System	AF-R-100
Reinforcement	181-style AF-994 glass Cloth with HTS finish
Impregnation Procedure	Hot-melt coated
Cure Cycle	3-minute contact @ 700°F, then 30 hr @ 700°F under 200 psi pressure in air, cooled under pressure to approximately 150°F
Post Cure Cycle	24 hr each @ 600°F, 650°F and 700°F, then 6 hr each @ 750°F, 800°F and 850°F, all in nitrogen
Laminate Size	14 plies, 12 inches by 12 inches
Specific Gravity	Approximately 1.80
Resin Content	Approximately 22 ± 2%

From Tables 85 and 86 it can be seen that mechanical properties follow those of other plastics, i.e., strength and modulus increase with decreasing temperature.

Thermal Properties

The coefficient of thermal expansion on a polybenzimidazole 1581-994S laminate was determined to be $2.38^{10^{-6}}$ in/in/°F from -100 to +200 (heating) and $2.45^{10^{-6}}$ in/in/°F from +200 to -100 (cooling) in the warp direction. Tests were made on a quartz dilatometer in accordance with ASTM D696-44. (93)

SECTION XIX. POLYESTERS

Mechanical Properties

The flexural strength of a polyester (modified Selectron 400) molding material was determined in accordance with method 1031 of Federal Test Method Standard No. 406, modified to four-point loading. (94) Tests were conducted on 1/4" thick notched and unnotched specimens. The notched specimens were prepared with a nominal 0.005-inch-deep scratch normal to the specimen length, in the center of the span, and to within 0.050 inch of either edge. The point of the scribe had a radius of less than 0.002 inch. The specimens were loaded so that the notch was in tension. The average strength is plotted in Figure 192.

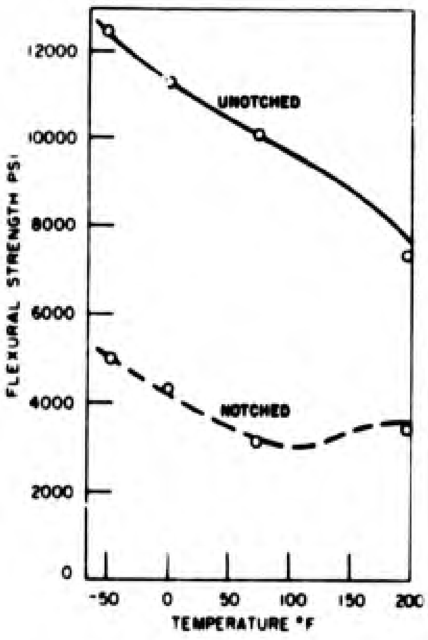


Figure 192. Flexural Strength of Modified Selectron 400 (94)

Table 87 gives low temperature tensile and flexural properties of various polyester laminates. Specimens were prepared in accordance with Federal Specification L-P-406a and conditioned as follows: 24 hours in dry-ice box at -65°F; immersed in distilled water for 14 days, then 24 hours in dry-ice box at -65°F. All were tested within 30 seconds from conditioning.

TABLE 87. EFFECT OF LOW TEMPERATURE ON STRUCTURAL POLYESTER LAMINATES (95)

Material	Moisture Absorption	Tensile, Ultimate, K psi				Flexure, Ultimate, K psi			
		Room Temp		-65°F Frozen		Room Temp		-65°F Frozen	
		Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
A	.25%	52.9	48.7	64.7	62.8	64.8	47.7	72.9	69.4
B	-	34.0	25.4	52.7	39.8	31.3	22.1	39.6	46.0
C	.11%	50.0	46.1	67.2	59.6	65.2	55.8	62.4	59.8
D	.35%	56.5	46.6	65.3	59.2	75.9	45.9	86.0	69.0
G	.35%	52.3	50.2	65.0	59.2	58.6	45.7	65.5	64.4
K	.28%	13.0	11.8	16.2	13.3	31.1	23.1	31.9	29.4
L	2.6 %	5.5	6.0	5.9	7.2	13.2	-	12.8	-

MATERIAL

	Polyester	Reinforcement	Ply
A	Selectron 5041	Glass, 181-114	11
B	Selectron 5041	Glass, 181, untreated	11
C	Vibrin 140	Glass, 181-114	11
D	Selectron 5003	Glass, 181-114	11
G	Laminac 4143	Glass, 181-114	11
K	Selectron 5041	Glass, T36 Mat, (60% resin)	-
L	Selectron 5041	Cotton Duck, 8 oz	3

The tensile properties of a 181 glass-polyester laminate were measured over a temperature range of -60°C to 80°C at the conventional static rate (approximately 6 x 10^4 milliseconds to failure) and at a higher rate (10 milliseconds to failure). The results are presented in Table 88. (96) It is pointed out that the results reported are averages for equal numbers of samples tested in both the parallel and perpendicular direction since the differences in direction were not appreciable.

TABLE 88. EFFECT OF TEMPERATURE ON TENSILE PROPERTIES OF 181 GLASS CLOTH REINFORCED POLYESTER (96)

Property (1)	Temperature, °C						
	-60	-40	-20	0	23	50	80
Modulus of Elasticity, primary							
High rate, K psi	4,770	4,480	4,540	4,460	5,545	3,500(2)	3,360(2)
Static, K psi	3,760	2,910	2,690	2,820	2,825	2,500(2)	2,240(2)
Increase, %	27	54	69	58	96	40	50
Modulus of Elasticity, secondary							
High rate, K psi	1,930	2,090	1,870	1,990	2,352	------	------
Static, K psi	1,840	1,890	1,610	1,890	2,086	------	------
Increase, %	5	11	16	5	13	------	------
Tensile Strength							
High rate, K psi	73.6	72.5	68.8	66.47	62.34	58.00	54.2
Static, K psi	51.58	44.84	41.4	39.0	36.74	34.13	29.4
Increase, %	43	62	66	70	70	70	84
Elongation							
High rate, %	3.20	3.24	2.91	2.78	2.41	2.42	2.10
Static, %	2.46	2.21	2.01	1.83	1.66	1.65	1.48
Increase, %	30	47	45	52	45	47	42
Work to Produce Failure							
High rate, ft-lb/in.^{3}	111.5	111.2	97.2	90.1	74.6	70.4	52.4
Static, ft-lb/in.^{3}	60.7	45.3	38.6	32.9	27.4	25.4	19.6
Increase, %	84	145	152	174	172	177	167
Time to Failure							
High rate, milliseconds	10.7	10.7	10.1	10.1	9.9	9.8	9.0
Static, milliseconds, x 10^4	7.8	6.8	6.5	6.2	5.9	5.8	5.8

(1) ASTM D638, type I, specimen; 1/8" thick hand lay-up panels formulated with Stypol 25 resin, vacuum pressure >12 psi; gelled under heat lamps; post cured 3 hrs @ 250°F.

(2) Value at 1/2% strain.

The increase in tensile strength with decreasing temperature (and increasing rate) is as expected. However, the increase in elongation with increased rate and decreased temperature is not generally characteristic of all plastic materials, but has been obtained with other glass-reinforced laminates. The increase in work-to-produce failure is the result of increases in tensile strength and elongation. The primary modulus values at both rates vary considerably and do not show any definite trend.

It should be noted that at the beginning of any test, the surrounding temperature may be accurately controlled, and the specimens may be conditioned long enough to assure temperature equilibrium. But, as the test progresses, the internal temperature of the specimen is raised, to unknown temperature gradients. The magnitude of these gradients is dependent on the heat generated locally, the thermal conductivity of the material and the surface-to-volume ratio of the specimen. Also, there is considerable internal stress which is due to the high shrinkage of the resin during cure and to large difference in the coefficient of expansion of the glass reinforcement and the polyester resin. (96)

It was considered probable that in this study, the erratic fluctuation of the modulus was the result of one or more of these factors.

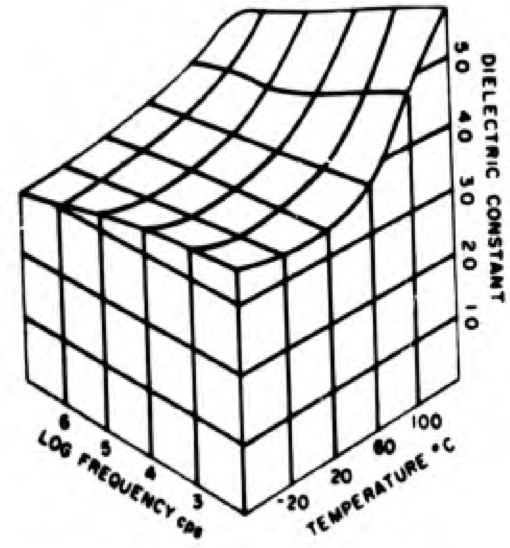
At room temperature and below, the primary modulus values are over 50 percent higher under high-rate loading than under static conditions except at -60° C where the increase is only 27 percent. Also, the tensile strength increase of more than 60 percent (high loading rate over static) at these temperatures drops to 43 percent at -60°C. From this it appears that at low temperatures the static and high loading rate values tend to approach each other.

The secondary modulus occurring at 23°C and below was affected less by temperature or rate than the other properties studied.

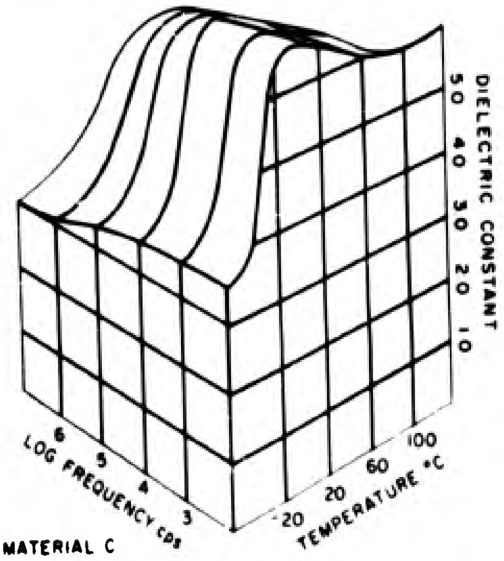
Electrical Properties

The dielectric constant and dissipation factor of commercial modified polyester casting materials were measured on samples 2 inches in diameter and .2 inches thick using a modified Schering bridge at frequencies from 10^2 to 10^5 cps and a series resonance circuit at 10^6 and 10^7 cps. (61) Figures 193 and 194 are field diagrams of the dissipation factor and dielectric constant as a function of frequency and temperature.

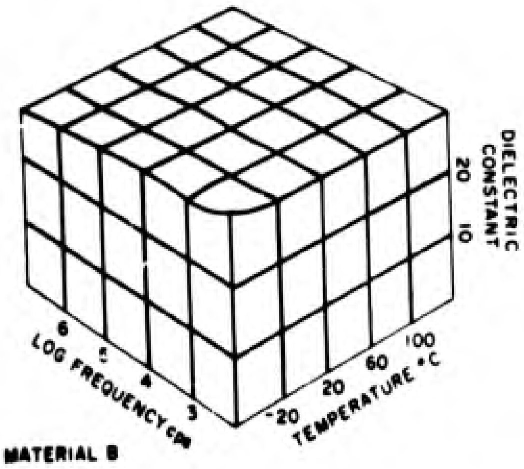
Figures 193 and 194 show the dielectric constant and dissipation factor vary considerably for these formulations. The more highly crosslinked system is less temperature dependent and less affected by frequency.



MATERIAL A



MATERIAL C



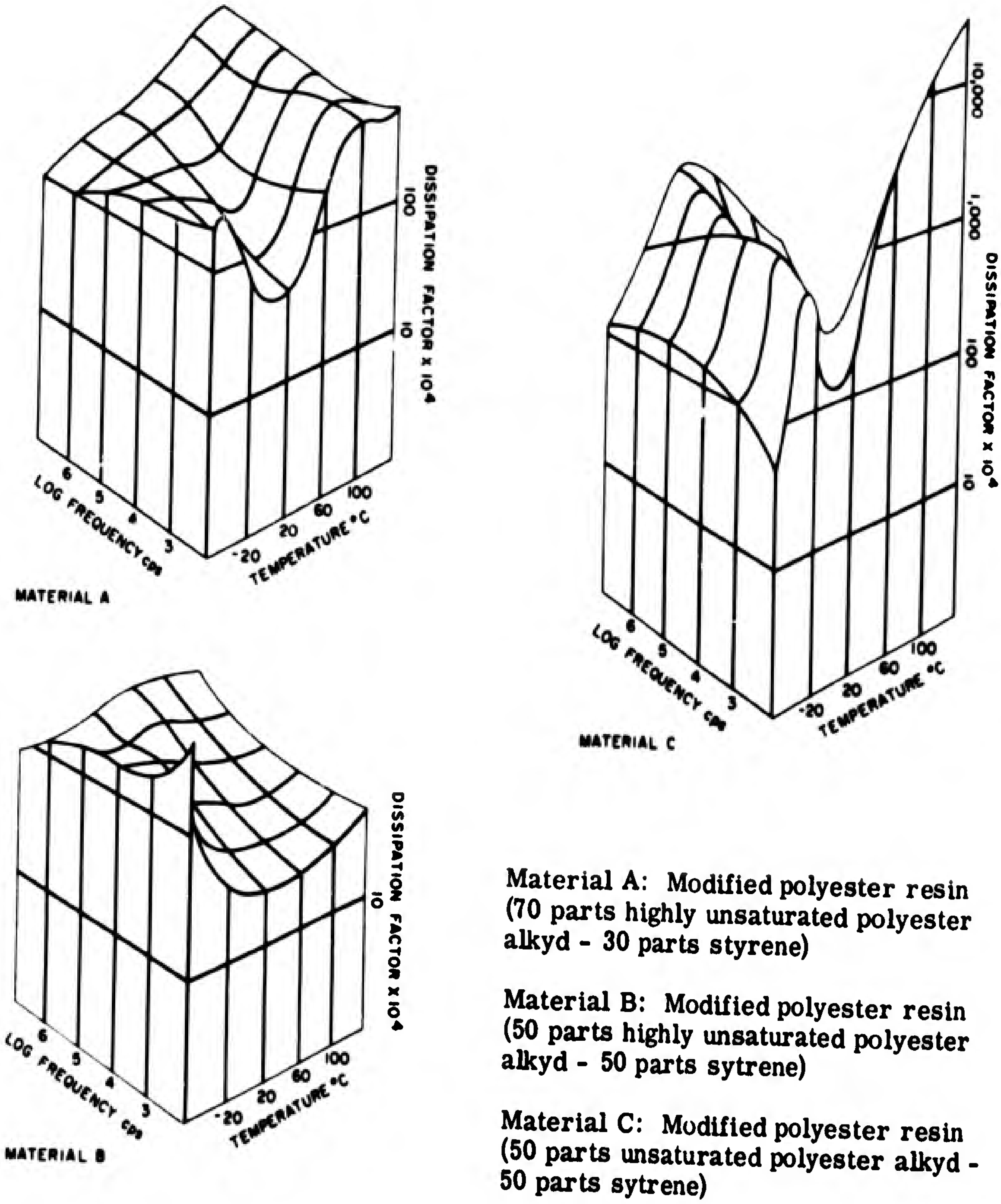
MATERIAL B

Material A: Modified polyester resin (70 parts highly unsaturated polyester alkyd - 30 parts styrene)

Material B: Modified polyester resin (50 parts highly unsaturated polyester alkyd - 50 parts styrene)

Material C: Modified polyester resin (50 parts unsaturated polyester alkyd - 50 parts styrene)

Figure 193. Dielectric constant of modified polyester resin as a function of frequency and temperature (61)



Material A: Modified polyester resin (70 parts highly unsaturated polyester alkyd - 30 parts styrene)

Material B: Modified polyester resin (50 parts highly unsaturated polyester alkyd - 50 parts sytrene)

Material C: Modified polyester resin (50 parts unsaturated polyester alkyd - 50 parts sytrene)

Figure 194. Dissipation factor of modified polyester resin as a function of frequency and temperature (61)

Thermal Properties

The thermal expansion of polyester modified molding material (Selectron 400) was measured by method 2032, Federal Test Method Standard 406. The results on 1/8", 1/4" and 1/2" thick sheet material is shown below in Table 89.

TABLE 89. COEFFICIENT OF THERMAL EXPANSION OF POLYESTER (MODIFIED SELECTRON 400) MOLDING MATERIAL (94)

Temperature °F	Thermal Expansion (in./in. x 10^4) (1)		
	1/8" Material	1/4" Material	1/2" Material
-55	-38	-36	-41
32	-16	-16	-12
72	0	0	0
85	0.63	1.3	0.3
105	5.6	5.0	5.5
120	10.0	10.6	13
140	20	18	23
175	39	35	40

(1) Average of values for 5 samples

Figures 195 and 196 contain the total thermal expansion of a polyester laminate and a film. (30) Data was obtained from a modified Leitz dilatomater. Measurements in the thickness direction of the film were made by cutting 40 disks, 0.168" in diameter and stacking them together to form a 1/2 inch long specimen.

The data for the laminate is as expected. (See Section on phenolics.) The expansion in the thickness direction is significantly greater than in the parallel to reinforcement direction.

Although the authors do not account for the difference in expansion in the two directions of the film, one factor was undoubtedly the influence of film orientation.

The thermal conductivity in one atmosphere of nitrogen and helium gas of a polyester laminate (see Figure 195 for description) is shown in Figure 197 (30). Again, the results follow the pattern previously discussed for glass-reinforced laminates (see Phenolics).

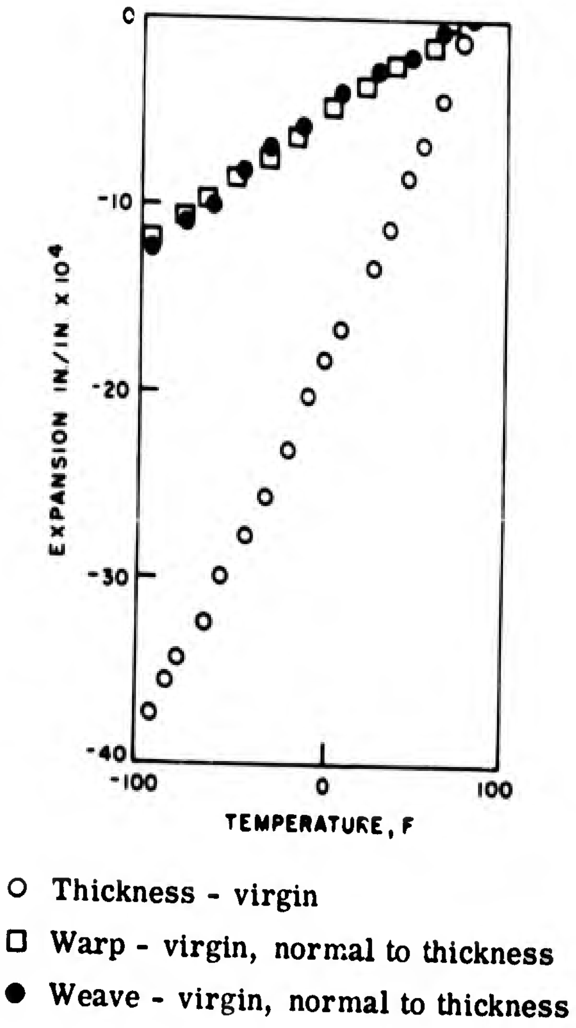
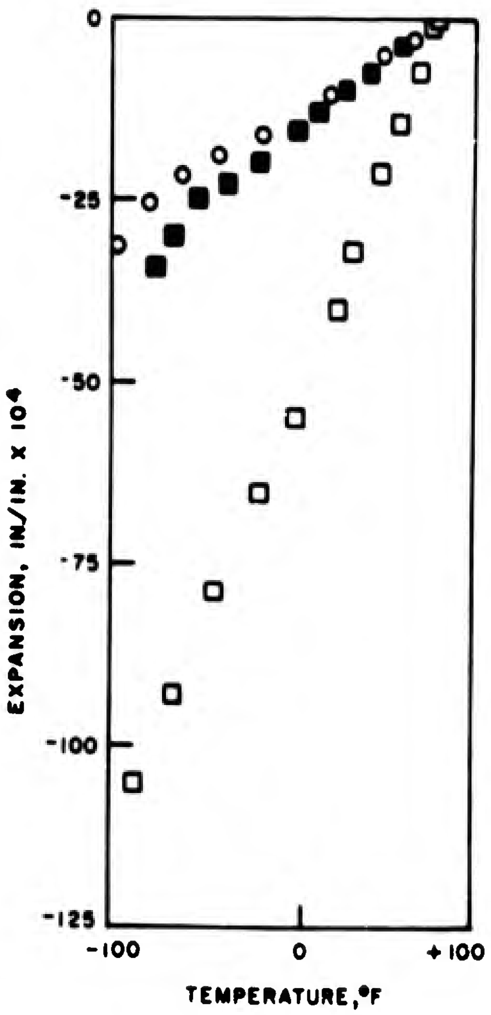


Figure 195. Total linear thermal expansion of material H, thickness and normal to thickness directions (30)

Composite System	
Manufacturer:	Raytheon Co.
Resin source:	Rohm & Haas Co.
Pre-Preg source:	Hess Goldsmith United Merchants
Resin content:	38%
Resin System	
Trade name:	Paraflex P-43
Chemical name:	Polyester
Curing agent:	Benzoyl
Additives:	Peroxide, styrene
Reinforcement System	
Trade name:	E glass
Generic name:	glass cloth
Fabric designation:	Style 181
Finish:	Volan "A"
Lay-up	Parallel laminate
No. of plies:	48

Orientation:	Warp in 10" dimension
Cure Cycle	
Pressure:	1000 psi to 1/2" stops
Temperature:	250°F
Temperature - time history:	Impregnate cloth and put in pre-heated mold (200°F, apply pressure. Heat to 250°F in 15 min. Cure 2 hrs. Cool to 180°F.
Post-Cure Cycle	
Time-temperature:	Cure for 4 hrs at 280°F in hot air oven

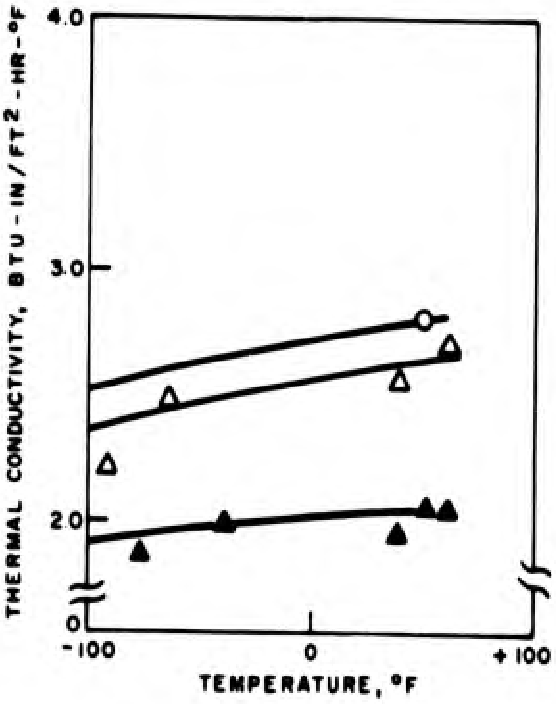


○ Normal to thickness, traverse

■ Normal to thickness, roll

□ Thickness

Figure 196. Total linear thermal expansion of polyester (Mylar) film in thickness and normal to thickness directions (30)



○ Helium Gas, 1 ATM, Warp Direction

△ Nitrogen Gas, 1 ATM, Warp Direction

▲ Nitrogen Gas, 1 ATM, Thickness Direction

Figure 197. Thermal conductivity of H-1 material in thickness and warp directions (30)

SECTION XX. POLYIMIDES

Mechanical Properties

This family of materials is relatively new and was tested primarily for high temperature and cryogenic applications. Therefore, little data was found in the low temperature region.

Figure 198 plots the tensile strength of a filled and unfilled Polymer SP and Table 90 contains data on the tensile properties of the film material.

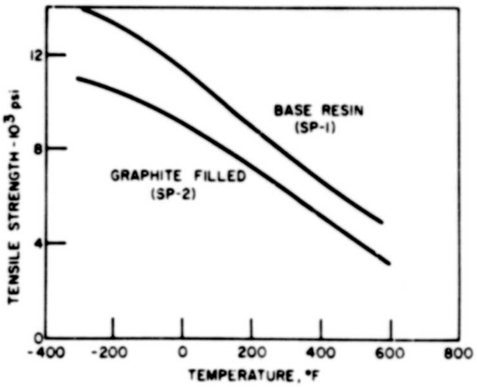


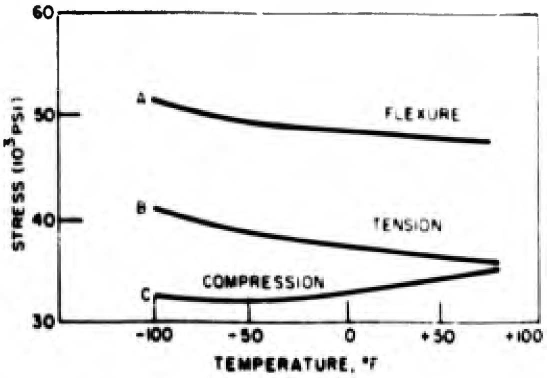
Figure 198. Tensile strength vs. temperature of polyimides (Vespel) (97)

TABLE 90. TENSILE PROPERTIES OF POLYIMIDE FILM (1 MIL THICK, AT 100% PER MINUTE ELONGATION) (98)

Property	Temperature	Value
	°C	
Tensile strength, Kpsi (ASTM D 882)	100	22.0
	20	25.0
	-20	32.0
Yield point stress, Kpsi	100	12.0
	20	14.0
	-20	16.0
Tensile modulus, Kpsi (ASTM D 882)	100	320.0
	20	430.0
	-20	480.0
Elongation, % (ASTM D 882)	100	87.0
	25	70.0
	-20	65.0

The mechanical properties of polyimide-glass reinforced laminates are shown in Figures 199, 200 and Table 91.

Mechanical properties are as expected except for the dry compressive laminate strength (Fig. 199 which decreases slightly from room temperatures to -100°F.)

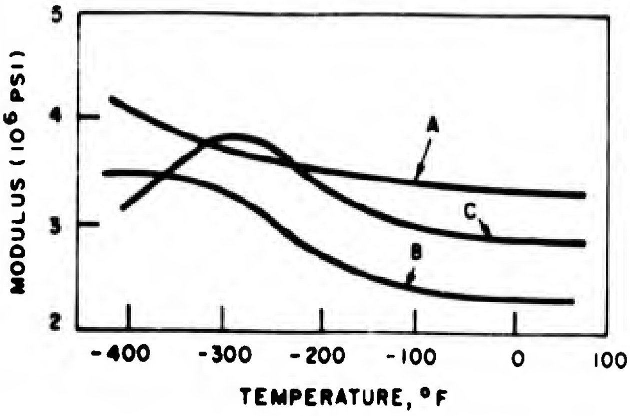


A - Westinghouse 1-8 resin, E - 181/A1100 reinforcement, 35% resin content, 0.125 - in.nominal panel thickness.

B - Westinghouse -1-8 resin, E-181/A-1100 reinforcement, 35% resin content, 0.125 in.nominal panel thickness.

C - Westinghouse 1-8 resin, E-181/A1100 reinforcement, 34.6% resin content, 0.500-in.nominal panel thickness.

Figure 199. Tensile, flexural and compressive strength of polyimide-fiberglass laminate as a function of temperature (98)



A - Westinghouse 1-8 resin, E-181/A-1100 reinforcement, 34.6% resin content, 0.500-in.nominal panel thickness (Compressive)

B - Westinghouse 1-8 resin, E-181/A-1100 reinforcement, 35% resin content, 0.125-in. nominal panel thickness (Flexural)

C - Westinghouse 1-8 resin, E-181/A1100 reinforcement, 35% resin content, 0.125-in.nominal panel thickness (Tensile)

Figure 200. Modulus of elasticity, flexural and compressive modulus of polyimide-fiberglass laminate (98)

TABLE 91. MECHANICAL PROPERTIES OF POLYIMIDE 181 A-1100 GLASS CLOTH LAMINATES (BRUNSWICK) (98)

Test Temperature	Exposure Conditions	Tensile Strength	Compressive Strength	Flexural Strength
		psi	psi	psi
77°F	1/2 hr. soak @ 77°F	39,000	38,000	52,500
-65°F	1/2 hr. soak @ -65°F	58,000	45,000	64,000

Electrical Properties

The dissipation factor versus temperature and frequencies for 1 mil H film is given in Figures 201 to 203. The loss peaks occur at the lower temperatures and the effects of frequency becomes more significant.

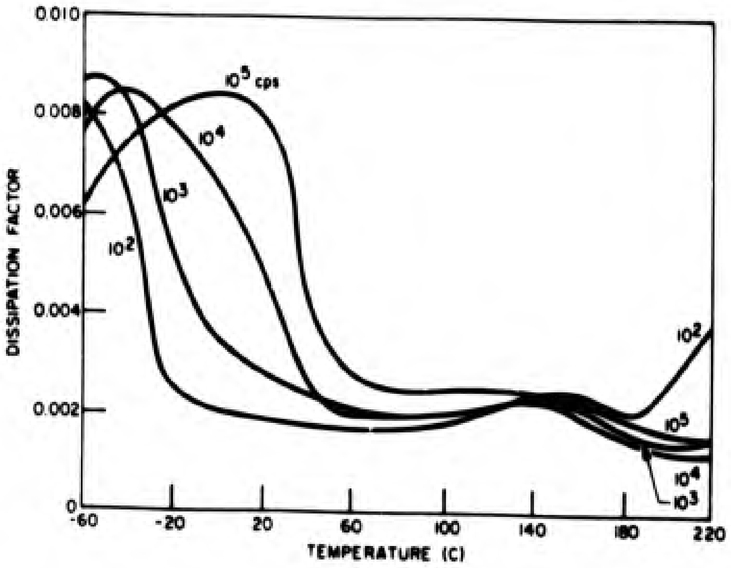


Figure 201. Dissipation factor of H film (1-mil thick) vs. temperature (38)

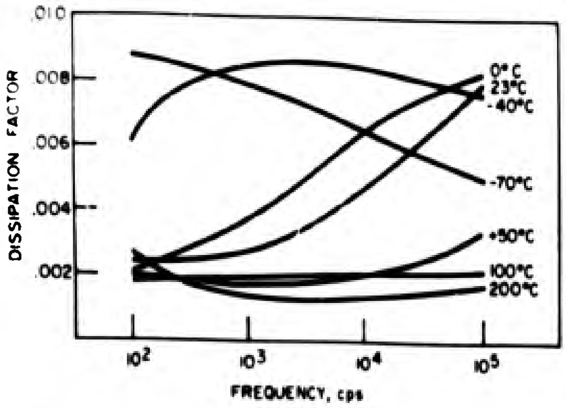


Figure 202. Dissipation factors vs frequency of H film (1 mil) (99)

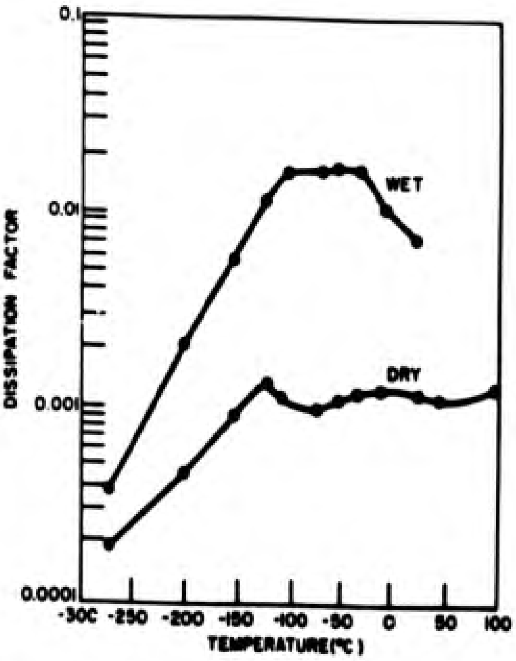


Figure 203. Dissipation factor at 1 Kc as a function of temperature. "Wet" H-Film (as above plus 2 days in water at 80°C) "Dry" H-Film (heated 2 days at 200°C) (98)

Polyimide film has a moderately high dielectric constant at low temperatures but is relatively unaffected by frequency as indicated in Figures 204 to 206.

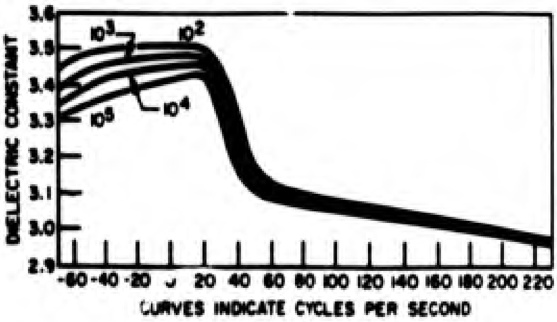


Figure 204. Dielectric Constant vs. temperature of H film (1 mil) (99)

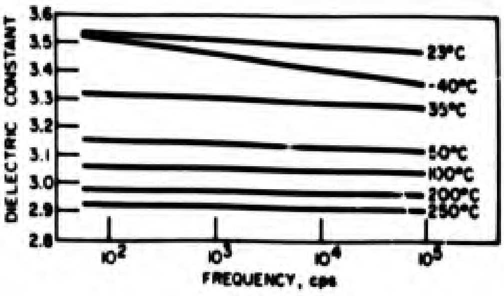


Figure 205. Dielectric constant vs. frequency of H film (1 mil) (99)

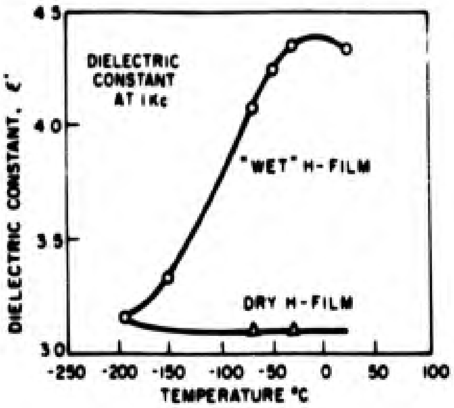


Figure 206. Dielectric constant as a function of temperatures for a dry (heated 2 days at 200°C) and wet H-film (heated 2 days at 200°C plus 2 days in water at 80°C) (98)

The dissipation factor and dielectric constant for "Polymer SP" at 1 Kc is given in Figures 207 and 208. These are essentially unaffected by change in temperature from +20 to -60°C.

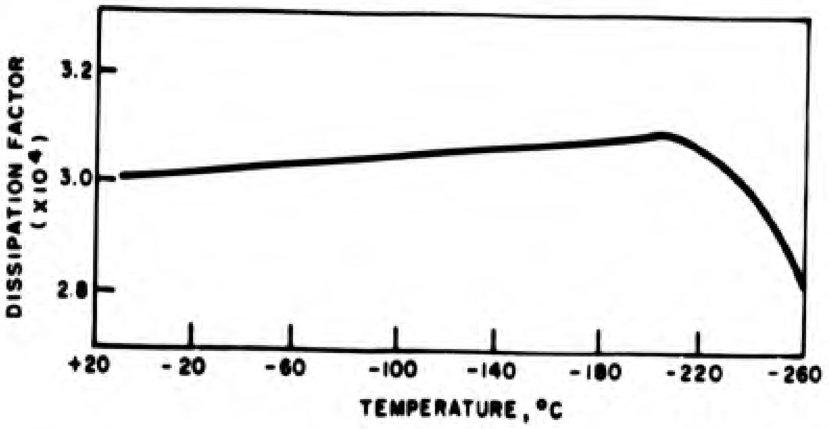


Figure 207. Variation of dissipation factor with temperature for SP* at 1 Kc (98)

*A moldable polyimide product.

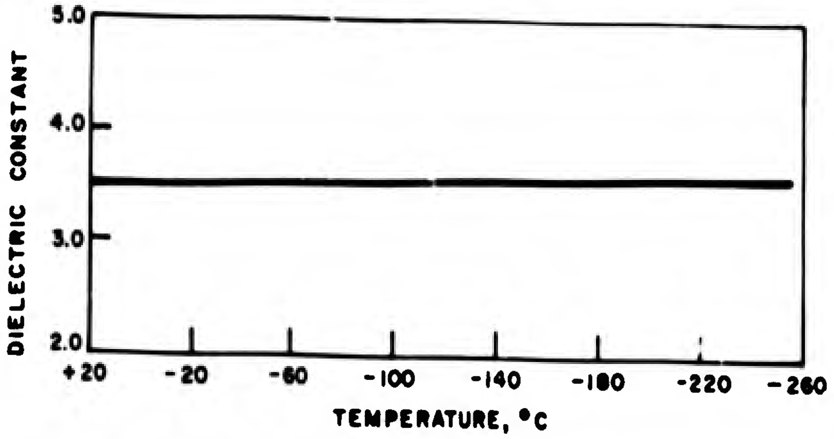


Figure 208. Variation of a dielectric constant with temperature for SP at 1 Kc (98)

Thermal Properties

The average coefficient of linear expansion for 1 mil film is 2.0×10^{-5} in/in/°C from -14°C to 38°C by ASTM D-696-44. (100)

Polyimide SP coefficient of expansion is reported at 28.4×10^{-6} in/in/°F in the -300 to +500°F range. (98)

SECTION XXI. SILICONES

Mechanical Properties

Table 92 gives the average tensile and impact properties of a glass-reinforced silicone laminate. Tensile determinations were made using a constant rate of loading of 8000 psi per minute on a Tinius-Olsen Plastiversal Testing Machine. Test specimens conformed to Type 1, Federal Specification L-P 406a - 1011. Load-elongation graphs were recorded to the point of specimen failure using Baldwin Model PS 6-PS-7 and PS8 Microformer Type Plastics Extensometers, coupled to a Baldwin Microformer type recorder.

TABLE 92. TENSILE AND IMPACT PROPERTIES OF A SILICONE LAMINATE (9)

Modulus of Elasticity (psi)

	-65°F	-40°F	+10°F	+77°F
Laminated Silicone Glass Fabric Base	2,130,000	2,060,000	2,102,000	2,360,000

Tensile Strength (in psi)

Laminated Silicone Glass Fabric Base	34300	33500	29600	29200

Work to Product Failure (Ft.-Lbs per Cubic Inch)

Laminated Silicone Glass Fabric Base	21.6	23.0	17.7	16.4

Elongation-at-Break (Percent)

Laminated Silicone Glass Fabric Base	1.63	1.60	1.41	1.32

Zod Impact Strength (Ft-Lbs Per Inch of Notch)

Laminated Silicone Glass Fabric Base	11.42	10.74	9.94	8.20

Impact tests were conducted on a Baldwin-Southmark pendulum impact machine (Federal Specifications L-P-406 and ASTM D 256-41T). All samples were conditioned two hours before testing.

The results are as expected for glass reinforced laminates: tensile strength, work-to-product failure, elongation at break and impact strength increased with decreasing temperature. Modulus of elasticity varied and no trend was evident (see Polyester).

Electrical Properties

Figures 209 and 210 show the power factor and dielectric constant of a silicone elastomer at 100 cps as a function of temperature.

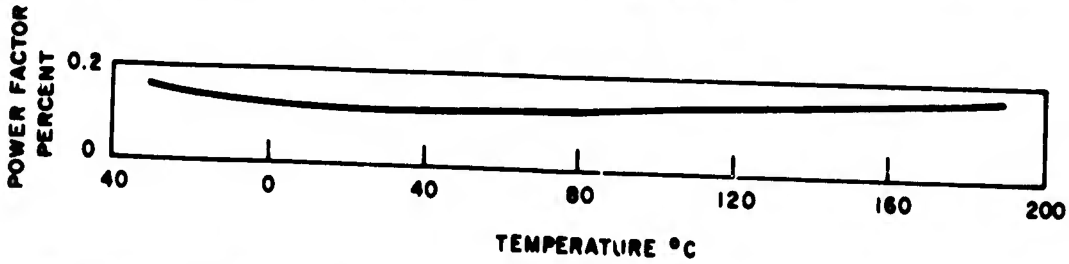


Figure 209. Effect of temperature on the power factor (at 100 cps) of (Silastic 250) silicone rubber. (101)

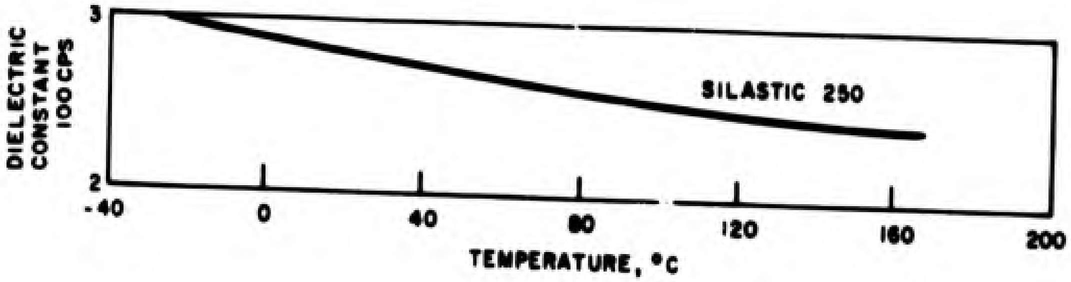


Figure 210. Delectric constant of (Silastic 250) silicone rubber as a function of temperature at 100 cps. (101)

The electrical strength of a glass-reinforced silicone laminate versus temperature and frequency is charted in Figure 211.

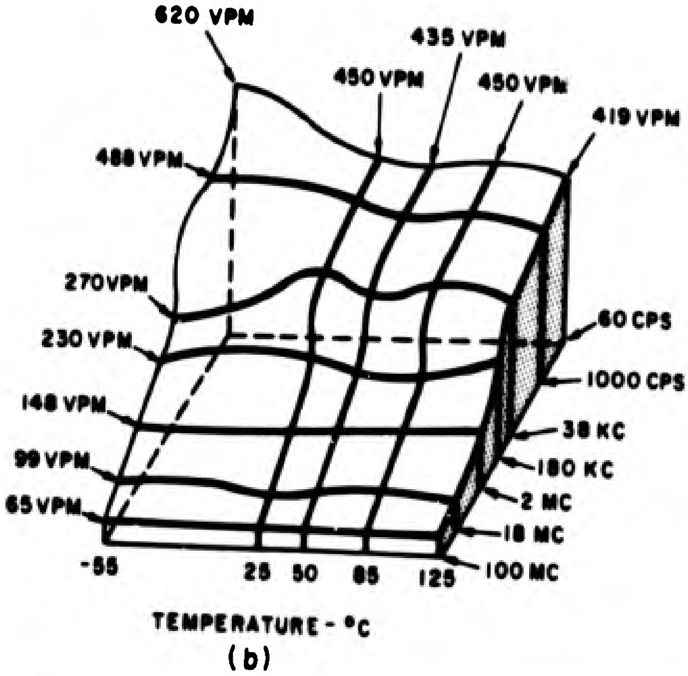


Figure 211. Electric strength vs. temperature and frequency of silicone laminate (29)

Thermal Properties

Figure 212 shows the total thermal expansion of a silicone casting material. The determinations were made on a quartz tube dilatometer. (92) The thermal expansion was the highest of all casting materials tested. This was attributed to the fabrication history. The casting was not fully cured during its fabrication due to cracking problems.

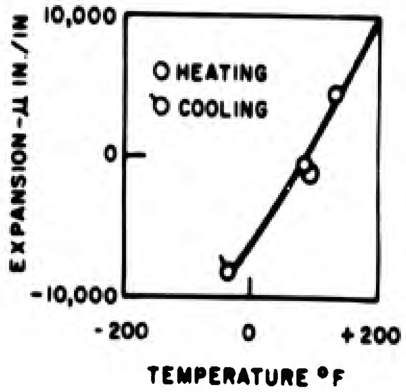


Figure 212. Thermal expansion in the longitudinal direction of a DC2106 silicone molding resin with Dow Corning Catalyst 15 (92)

The total linear expansion of a glass-reinforced silicone laminate measured in the thickness and normal to thickness direction is shown in Figure 213. Again the effect of orientation is evident as discussed in previous sections.

The thermal conductivity in two directions for the same material in a helium and nitrogen environment is given in Figure 214. This also agrees with data for other glass-laminates.

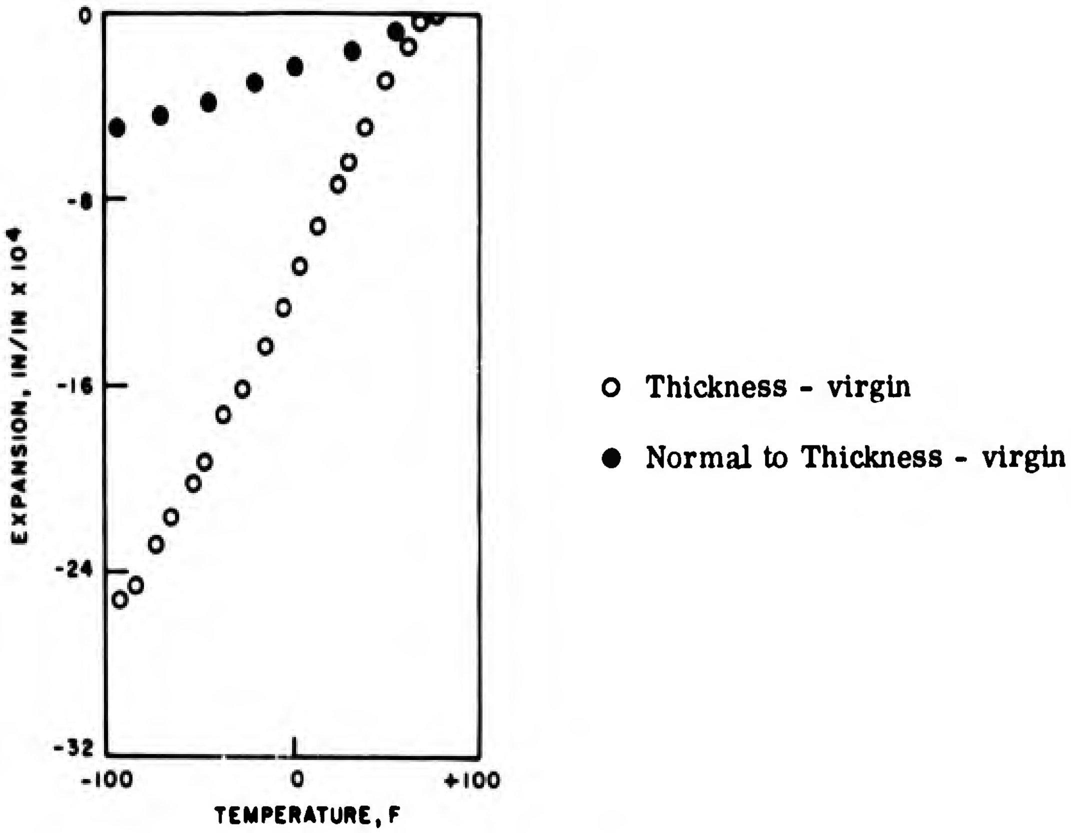


Figure 213. Total linear thermal expansion of silicone molding material thickness and normal to thickness directions (30)

Composite System
Manufacturer: Raytheon Company
Resin Source: Dow Corning
Pre-preg Source: Owens-Corning Fiberglas Co.
Resin Content: 34%

Resin System
Tradename: DC-2106
Chemical Type: Silicone
Catalyst: XY-15
Curing Agents, Additives, Etc.: -

Reinforcement System
Tradename: "E" Glass
Generic Name: Glass roving
Fabric Designation: 20 end roving
Form of Reinforcement: Roving
Finish: 140 HTS

Lay-up: Alternate layers cross-plied at 90°
Number of plies: 80
Reinforcement Orientation: Cross-plied at 90°

Cure Cycle
Pressure: 300 psi to 1/2" stops
Temperature: 350°F
Temperature-Time History: Preheat platens to 250°F. Cure at 250°F for 2 hrs. Raise temp to 350°F and hold for 3 hrs., lower temp. to 150°F & remove.

Post-Cure Cycle
Time-Temperature: -

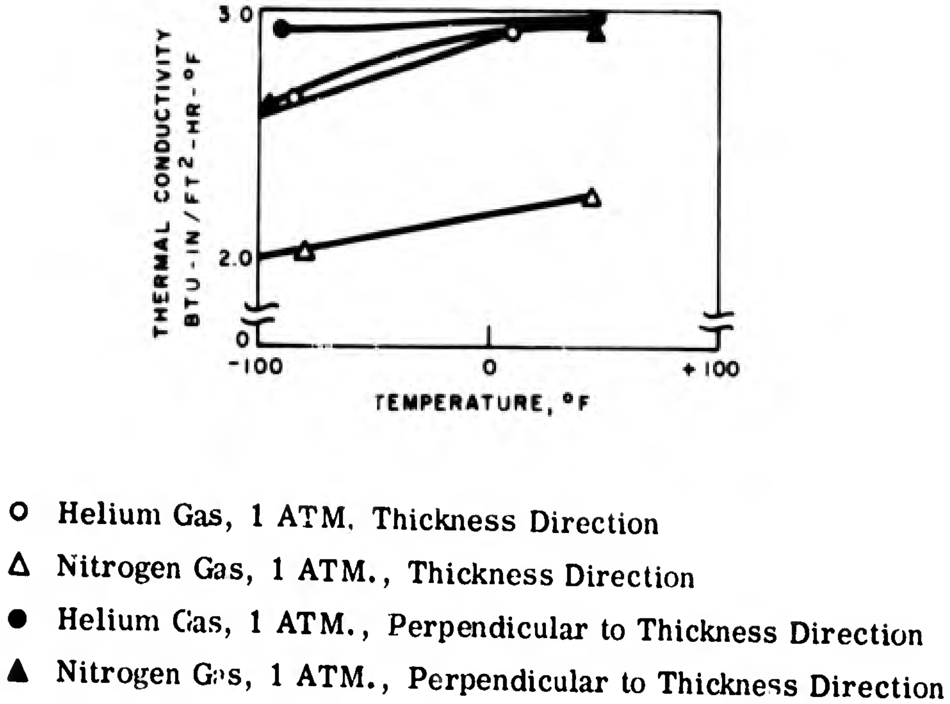


Figure 214. Thermal conductivity of silicone casting material in the thickness and perpendicular to thickness direction (30)

SECTION XXII. URETHANES

Mechanical Properties

Various polyether urethane elastome formulations were investigated for low temperature flexibility and oil resistance (102). Of the isocyanates evaluated in Table 93, the 100 percent 2, 4-toluene diisocyanate had slightly better low temperature properties. Although the DMMDI elastomer had a slightly higher low temperature modulus, it had higher tensile strength and lower oil absorption.

TABLE 93. EFFECT OF VARIOUS DIISOCYANATES ON PROPERTIES OF A ONE-SHOT CASTING ELASTOMER (102)

Polyol Blend

7000 M.W. Diol:	1 mole
Trimethylolpropane:	2 moles
Ave. Equiv. wt. of Polyols:	900
Diamine:	DCB
NH2 OH Ratio:	0.62
Excess NCO, Equiv. / 10,000 g:	1.4

Catalyst

Stannous Octoate 0.025

Cure

2 Hrs. @ 100°C
22 Hrs. @ 100°C Postcure

Code	1668-B	1668-C	1668-D	1668-E	1668-F	1668-G	1668-H
Diisocyanate	80 20 TDI (1)	2, 4 TDI	65/35, TDI (2)	MDI	(3)	(4)	(5)
Young's Modulus, E							
-60°C.	31,600	31,600	90,200	69,400	137,000	31,000	11,500
-55°C.	14,100	13,800	16,100	30,600	84,800	18,500	3,200
-50°C.	7,900	7,300	13,400	20,600	50,100	11,000	1,300
-45°C.	5,950	4,300	3,860	16,900	21,200	8,400	-
Tensile Strength, psi	1220	1010	1270	1400	1860	1460	380

(1) 80 20 ratio 2, 4 and 2, 6 isomers of Toluene diisocyanate
(2) 65 35 ratio
(3) Polyarylene polyisocyanate (crude)
(4) 3, 3' - Dimethyl Diphenyl Methane-4, 4' - Diisocyanate
(5) Hexamethylene Diisocyanate

The effect of various diamines as a substitute for dichlorobenzidine (DCB) are shown in Table 94. The blends of N, N'-dicyclohexyl-p-phenylenediamine had the best low temperature flexibility. They also have tensile properties comparable to the DCB elastomers but are softer and are lower in modulus; oil resistance was poorer than the DCB elastomer. No improvements were gained by using 4, 4'-sulfonyl dianiline or lauroguanamine.

The low temperature flexibility of DCB samples compared with 2,6-dichloro-p-phenylene diamine are shown in Table 95. The phenylene diamine blends were more flexible at low temperatures; however, they had reduced oil resistance.

The effect of polyol chain extender type was studied from elastomers prepared from a 7000 M.W. experimental polyether diol blended with various lower equivalent weight polyols (Table 96).

In an attempt to obtain improved oil resistance various low molecular weight diols containing chlorine or an aromatic ring were blended with a 7,000 M.W. diol and DCB to the same average equivalent weight and NH_2/OH ratio. Although they had slightly improved oil resistance, Table 97, indicates they had reduced flexibility at -55°C.

TABLE 94. EFFECT OF VARIOUS DIAMINES ON PROPERTIES OF POLYETHER-DIAMINE-TDI ELASTOMERS (102)

Polyol Blend

7000 M.W. Diol: 1 mole
Trimethylolpropane: 2 moles
Ave. Equiv. wt. of Polyols: 900
NH_2 OH Ratio: 0.62

Excess NCO. Equiv./10,000 g.: 1.4

Code	1661-C	1667-B	1667-C	1671-B	1672-B
Diamine	DCB	(1)	(1)	(2)	(3)
Catalyst PPH	0.025	0.025	0.05	0.025	0.025
Young's Modulus, E					
-60°C.	-	14,900	7,000	63,300	17,400
-55°C.	7,900	5,400	2,100	12,300	6,300
-50°C.	3,500	3,200	1,400	6,600	3,900
-45°C.	3,200	1,800	-	3,700	4,200
Tensile Strength, psi	890	890	880	660	670

(1) N, N' - dicyclohexyl-p-phenylenediamine
(2) 4', 4'-sulfonyl dianiline
(3) Lauorguanamine

TABLE 95. COMPARISON OF PROPERTIES OF ELASTOMERS PREPARED WITH DCB OR 2, 6-DICHLORO-P-PHENYLENE DIAMINE (102)

14,000 M.W. Triol, moles: 1
2,900 M.W. Polytetramethylene glycol, moles: 3
Excess NCO, Equiv./10,000 g.: 1.43

Code	1684-C	1684-O	1684-U	1684-Q	1684-R	1684-S	1684-T
Polyol Blend							
Trimethylolpropane, moles	1	1	1	1	1	2	3
Ave. Equiv. wt. of Polyols	1905	1905	1905	1905	1905	1520	1280
Diamine	DCB	(1)	(1)	(1)	(1)	(1)	(1)
NH_2/OH Ratio	1.0	1.0	1.33	1.5	2.0	1.0	1.0
Young's Modulus, psi							
-55°C.	6100	2550	2500	2900	4700	3500	4000
-50°C.	5000	1780	1800	1900	4650	1500	2200
-45°C.	3100	<1700	<1800	1400	3200	1500	2000
Tensile Strength, psi	1130	530	1040	1100	1400	550	880

(1) 2, 6-dichloro-p-phenylene diamine

TABLE 96. EFFECT OF VARIOUS POLYOLS ON PROPERTIES OF POLYETHER-TDI-DCB ELASTOMERS (102)

Code	Diol Blends		Diol-Triol Blends					Diol-Triol Blends		Diol-Tetrol Blend	Triols Alone		Diol-Pentol Blend
	1661-AA	1661-E	1661-C	1661-G	1661-Q	1661-S	1661-I	1661-K	1661-M	1661-W	1661-O	1661-Y	1674-F
Polyol 7,000 M. W. Diol blended with	Ethylene Glycol	Propylene Glycol	Trimethylol Propane	Glycerol	Triethanol Amine	Triisopropanol Amine	TP-340	TP-740	TP-2540	Tetrol*	TP-2540 alone	GP-3030 alone	Pentol**
Ave. Equiv. wt. of of Polyols	900	900	900	900	900	900	900	930	1100	900	870	1000	900
NH_2/OH Ratio	0.62	0.62	0.62	0.62	0.63	0.63	0.63	0.65	0.76	0.63	0.61	0.69	0.62
Excess NCO, Equiv./ 10,000 g.	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Molar ratio 7,000 Diol to other Polyol	1:3	1:3	1:2	1:2	1:2	1:2	1:2	1:2.4	1:7.2	1:1.4	-	-	1:1.2
Young's Modulus, E													
-60°C.	13,900	-	-	-	-	-	-	-	-	-	388,000	321,000	210,000
-55°C.	5,700	14,200	7,940	5,800	4,100	9,200	12,900	49,800	145,000	5,500	259,000	219,000	13,200
-50°C.	4,900	9,800	3,500	3,200	3,100	6,000	7,750	20,500	71,600	4,900	155,000	28,800	5,650
-45°C.	3,900	7,200	3,250	2,850	2,900	5,050	6,600	13,900	19,800	3,300	-	21,300	1,200
Tensile Strength, psi	1160	630	890	720	730	1010	1300	740	580	1100	750	650	880

* N, N, N', N'-tetrakis (2-hydroxypropyl) ethylene diamine
** Anhydroenneaheptitol

TABLE 97. A COMPARISON OF ELASTOMERS PREPARED FROM BLENDS OF A 7,000 M.W. DIOL WITH VARIOUS SHORT CHAIN GLYCOLS (102)

Diisocyanate: TDI; Diamine: DCB; NH_2/OH Ratio (% Diamine) 0.63 (7.0);
Ave. Equiv. Wt. of Polyols: 900
Excess NCO, Equiv./10,000 g.: 1.4

Code	1661-E	1661-AA	1661-AB	1661-AC	1661-AD	1661-AE
Polyol Blend						
7,000 M.W. Diol, moles	1	1	1	1	1	1
Other Diol, moles	3.0 (1)	3.0 (2)	3.1 (3)	3.46 (4)	3.2 (5)	3.1 (6)
% Cl in Elastomer	0	0	3.5	7.1	4.7	0
Young's Modulus, E						
-60°C.	- -	13,900	-	-	-	-
-55°C.	14,200	5,700	127,000	101,000	51,900	13,800
-50°C.	9,800	4,900	25,500	327,000	17,400	11,000
-45°C.	7,200	3,900	12,400	20,800	8,100	6,300

(1) Propylene Glycol
(2) Ethylene Glycol
(3) Trichloropropylene Glycol
(4) HET Diol
(5) 2,2,3,3-Tetrachlorobutane Diol
(6) p-Xylylene Diol

Table 98. compares the use of ethylene glycol, 1,4-butanediol, and trimethylolpropane in Triol 14,000-Polytetramethylene glycol 2,900-DCB elastomers. Those containing ethylene glycol and butanediol (on the average) had slightly better tensile strength, oil resistance and low temperature flexibility.

TABLE 98. PROPERTIES OF ELASTOMERS PREPARED FROM TRIOL 14,000 BLENDED WITH LOW MOLECULAR WEIGHT POLYOLS, TDI AND DCB (102)

Excess NCO, Equiv./10,000 g.: 1.4

Code	1665-AD	1665-AF	1665-AG	1665-W	1679-B	1654-O	1654-AH
Polyol Blend							
14,000 M.W. Triol, moles	1	1	1	1	1	1	1
Trimethylolpropane, moles	2	1	1	1	-	2	2
Ethylene glycol, moles	-	1.5	-	-	1.5	-	1.5
1,4-Butanediol, moles	-	-	1.5	-	[illegible]	-	-
2,900 M.W. Polytetramethylene glycol, moles	3	3	3	4	4	-	-
Ave. E.W. of polyols	1530	1530	1530	1840	1840	1600	1240
NH_2/OH Ratio	0.8	0.8	0.8	1.3	1.3	1.3	1.0
Tensile Strength, psi	1090	1020	1330	1970	2420	1070	1410
Young's Modulus, E							
-60°C.	-	-	-	10,500	-	10,500	9,900
-55°C.	5,900	5,600	4,200	8,600	5,300	7,500	7,800
-50°C.	3,300	4,500	3,300	6,900	7,100	4,400	6,600
-45°C.	2,600	3,600	3,800	6,600	7,000	-	-

Tables 99 amd 100 list the moduli of elastomers prepared from a 7,000 M.W. polyether diol and a 14,000 M.W. triol, each blended with varying amounts of trimethylolpropane and dichlorobenzidine and cured with toluene diisocyanate. By increasing the percent of diamine, the tensile strength and oil resistance improved, but the low temperature moduli increased. No clear trends were evident due to varying the trimethylolpropane.

TABLE 99. ELASTOMERS PREPARED FROM A 7,000 M.W. DIOL BLENDED WITH TRIMETHYLOLPROPANE AND DCB (102)

Code	1652-G	1652-I	1652-L	1652-F	1652-E	1652-R	1652-P	1652-N
Diol/Triol Molar Ratio	1:1	1:2	1:2	1:2	1:2	1:2.5	1:3	1:3.3
NH_2/OH Ratio	1.0	0.5	0.625	0.75	1.0	0.526	0.455	0.526
% Diamine	7.33	5.77	7.02	8.22	10.45	6.87	6.77	6.94
Young's Modulus, E								
-65°C.	43,800	19,200	-	68,900	-	-	-	-
-60°C.	13,500	6,230	-	15,300	-	-	-	-
-55°C.	10,900	2,480	6,700	7,530	16,950	5,360	10,400	19,200
-50°C.	-	1,830	6,240	5,510	15,900	3,620	4,770	6,380
-45°C.	6,640	1,290	3,010	-	12,400	3,000	4,590	6,460
Tensile Strength, psi	1080	680	970	1770	1970	1130	880	1130

TABLE 100. ELASTOMERS FROM 14,000 MOLECULAR WEIGHT TRIOL BLENDED WITH TRIMETHYLOLPROPANE AND DCB (102)

Code	1654-G	1654-Y	1654-K	1654-M	1654-O	1654-Q	1654-S	1654-U	1654-W
Triol TMP Molar Ratio	1:1	1:1	1:1	1:2	1:2	1:2.5	1:2.5	1:3	1:3
NH_2 OH Ratio	1.0	1.5	2.0	1.0	1.33	0.75	1.0	0.5	0.75
% Diamine	4.67	7.00	8.66	6.58	8.47	5.79	7.46	4.47	6.43
Young's Modulus, E									
-67°C.	30,700	-	24,100	-	-	-	-	-	-
-60°C.	3,080	-	9,360	9,660	10,500	5,080	10,600	5,240	8,450
-55°C.	-	3,640	5,610	5,960	7,460	4,600	7,610	3,150	5,520
-50°C.	-	2,570	4,020	3,960	4,440	3,075	6,090	2,350	3,910
-45°C.	-	2,180	3,330	3,690	4,520	2,550	5,960	-	-
Tensile Strength, psi	450	720	980	1010	1070	920	1030	690	1010

Table 101 compares the flexibility of various elastomers prepared from blends of high molecular weight polyether diol or triol, dichlorobenzidine, and polytetramethylene glycols reacted with TDI. As can be seen, the low temperature flexibility is little affected by using polytetramethylene glycol in the polyether blend, and in addition room temperature properties are improved.

TABLE 101. ELASTOMERS PREPARED FROM BLENDS OF POLYTETRAMETHYLENE GLYCOL WITH 14,000 M.W. TRIOL (102)

14,000 Molecular Weight Triol: 1 mole
Excess NCO, Equiv./10,000 g.: 1.4

Code	1654-AC	1665-O	1654-AE	1654-Y	1654-G	1665-K	1665-O	1665-S	1665-M	1665-Q	1665-U
Trimethylolpropane, moles	1.5	1	2.14	2	1	1	1	1	1	1	1
Polytetramethylene Glycol 2900, moles	-	3	-	3	-	2	3	4	2	3	4
Av. Equiv. Wt. of Polyol Blend	1900	1900	1530	1530	2380	2000	1900	1840	2000	1900	1840
NH_2/OH Ratio	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.4	1.33	1.28
% Diamine	5.7	5.7	6.8	6.8	-	5.4	5.7	5.8	7.3	7.3	7.3
Wt. % Polytetramethylene Glycol in Polyol Blend	-	37.5	-	37.5	0	28.7	37.5	44.5	28.6	37.5	44.5
Young's Modulus, E											
-60°C.	13,200	6,100	15,500	7,700	3,700	6,000	6,150	7,300	8,800	10,000	10,500
-55°C.	6,700	3,700	8,200	8,000	1,200	4,350	3,700	4,200	4,950	6,000	8,500
-50°C.	2,700	-	4,600	5,700	1,200	3,600	-	-	5,200	4,000	6,900
-45°C.	2,300	2,700	3,600	5,300	1,200	2,500	2,700	3,950	4,500	4,100	6,600
Tensile Strength, psi	730	1400	1000	1300	490	910	1400	1440	1190	1680	1970

Tables 102 and 103 show the effect of the molecular weight of polyether on low temperature flexibility. In Table 102 an 11,000 M.W. triol, 14,000 M.W. triol, 7,000 M.W. diol and a 4,700 M.W. diol each blended with trimethylolpropane to the same equivalent weight and identical amounts of TDI and DCB were compared. As can be noted, the modulus of the 4,700 M.W. diol was considerably higher than the other formulations.

TABLE 102. A COMPARISON OF ELASTOMERS FROM 11,000 M.W. TRIOL AND OTHER HIGH EQUIVALENT WEIGHT POLYOLS (102)

Diamine: DCB
NH_2/OH Ratio: 0.62 (7.0% Diamine)
Excess NCO, Equiv./10,000 g.: 1.4
Ave. Equiv. Wt. of Polyol Blend: 900

Code	1678-C	1654-AA	1666-I	1661-C
Polyol Blend				
11,000 M.W. Triol, moles	1	-	-	-
14,000 M.W. Triol, moles	-	1	-	-
4,700 M.W. Diol, moles	-	-	1	-
7,000 M.W. Diol, moles	-	-	-	1
Trimethylolpropane, moles	3.2	4.5	1.15	2
Theo. Ave./M.W./Crosslink	2300	2300	3280	2730
Young's Modulus, E				
-55°C.	8,500	6,200	21,000	7,900
-50°C.	4,800	3,800	11,000	3,500
-45°C.	3,800	-	5,700	3,300

Table 103 compares the properties of castings prepared from triols of varying molecular weights blended with a 2,900 M.W. polytetramethylene glycol. Again, it was noted that low temperature properties of elastomers derived from the highest equivalent weight polyols were slightly better.

TABLE 103. A COMPARISON OF ELASTOMERS FROM 11,000 M.W. TRIOL AND 2,860 M.W. POLYTETRAMETHYLENE GLYCOL BLENDS WITH SIMILAR BLENDS FROM OTHER HIGH M.W. TRIOLS (102)

NH_2/OH Ratio: 1.0

Excess NCO, Equiv./10,000 g.: 1.4

Code	1650-J	1678-G	1665-Y	1678-K	1665-S	1665-O
Polyol Blend						
6,500 M.W. Triol, moles	1	-	-	-	-	-
11,000 M.W. Triol, moles	-	1	-	1	-	-
14,000 M.W. Triol, moles	-	-	1	-	1	1
Trimethylolpropane, moles	-	1	2	0.39	1	1
Polytetramethylene Glycol, moles	2	3	3	3	4	3
Ave. Equiv. Wt. of Polyol Blend	1670	1590	1530	1912	1840	1905
% Polytetramethylene Glycol in Polyol Blend	44.8	43.7	37.5	44.2	44.5	37.5
% Diamine (DCB)	6.4	6.5	6.8	5.7	5.9	5.7
Young's Modulus, E						
-60°C.	8,600	-	7,700	-	7,300	6,200
-55°C.	8,200	3,400	8,000	10,200	4,200	3,700
-50°C.	6,400	4,300	5,700	5,400	-	-
-45°C.	6,700	3,900	5,400	5,100	3,950	2,700

The effect of ester linkages as shown in Table 104. High molecular weight polyols were prepared by coupling available polyols via ester linkages. Pluracol P-2010 was reacted with sebacyl chloride at a 2:1 molar ratio to give a diol of 4,236 average molecular weight containing two ester linkages. Also, Pluracol P-2010 reacted with sebacyl chloride at a 1:2 molar ratio was used to couple with two moles of Pluracol TP-5640 triol. This reaction yielded a tetrol containing four ester linkages per molecular weight of 13,400.

Dichlorobenzidine - TDI elastomers were prepared from these polyols. The low temperature moduli are shown in Table 104. The elastomers hardened at higher temperatures than were previously found for other elastomers of similar molecular weight containing no ester linkages. Also, these ester linkages when incorporated into the polyether polyols (even at very low concentrations) caused excessively high modulus at -55°C, but offered essentially no advantages at room temperatures.

TABLE 104. ELASTOMERS FROM POLYOLS CONTAINING ESTER LINKAGES (102)

Diisocyanate: TDI
Diamine: DCB

Code	1643-D	1643-F	1643-J	1643-K
Polyol	(a)	(a)	(b)	(b)
NH_2/OH Ratio	1.0	1.5	1.5	2.0
Young's Modulus, E				
-55°C.	14,500	24,900	5,000	19,000
-50°C.	7,200	9,370	2,230	3,500
-45°C.	3,770	7,250	1,530	2,700

(a) 2 Moles Pluracol P-2010 coupled with Sebacyl Chloride.

(b) Trimer of 1 Mole Pluracol P-2010 + 2 Moles Sebacyl Chloride + 2 Moles TP-5640 Polypropylene ether triol.

Table 105 shows the effect of substituting a polytrimethylene oxide diol for the polytetramethylene glycol used in sample 1665-0. As can be seen, these have higher moduli than the 1665-0 sample.

TABLE 105. USE OF POLYTRIMETHYLENE GLYCOL (1,450 M.W.) IN A STANDARD FORMULATION (102)

14,000 M.W. Triol, moles: 1

Code	1665-O	1685-D	1685-F
Polyol Blend			
Trimethylolpropane, moles	1	1	-
1,500 M.W. Trimethylene ether glycol, moles	3	1.2	4.2
Ave. Equiv. Wt. of Polyols	1900	1900	1800
NH_2 OH Ratio	1.0	1.0	0.9
% Diamine	5.7	5.7	5.7
% Polytrimethylene ether glycol in elastomer	37.5	9	25
Young's Modulus, E			
-55°C.	3,700	7,400	6,800
-50°C.	-	5,800	4,000
-45°C.	2,700	4,100	3,200

Table 106 compares elastomers containing tetrahydrofuran-ethylene oxide copolymers, DCB and TDI with one containing a 2,900 M.W. polytetramethylene glycol. The triol samples have good low temperature flexibility and excellent oil resistance. However, the tensile values were somewhat low. The diol samples had very low tensile strength.

TABLE 106. ELASTOMERS CONTAINING TETRAHYDROFURAN-ETHYLENE OXIDE COPOLYMERS, DCB AND TDI (102)

Excess NCO, Equiv. 10,000 g.: 1.43

Code		1686-A	1689-E	1689-J		1689-N	1689-P	1689-U
Polytetramethylene ether Glycol, moles	2900 M.W. diol[1]	1	-	-	4400 M.W. triol[1]	1	-	-
	2400 M.W. diol[1]	-	1	-	3900 M.W. triol[1]	-	1	-
	3500 M.W. diol[1]	-	-	1	5900 M.W. triol[1]	-	-	1
Ave. Equiv. Wt. of Polyols		1430	1205	1750		1350	1300	1980
DCB, moles		1	0.84	1.22		1.0	1	1.5
NH_2 OH Ratio		1.0	0.84	1.22		1.03	1.0	1.5
Young's Modulus, E								
-55°C.		63,000	8,100	6,400		6,800	6,500	-
-50°C.		51,000	7,300	5,400		6,100	5,200	-
-45°C.		39,000	6,600	6,500		6,000	4,700	-
Tensile Strength, psi		2840	290	380		770	850	900

[1] Tetrahydrofuran-ethylene oxide copolymers.

Table 107 shows the modulus of elastomers prepared from a 3,900 M.W. EO-THF triol and a 5,900 M.W. triol blended with trimethylolpropane to the same hydroxyl number. Each of these elastomers also had equal amounts of DCB and TDI. The 5,900 M.W. triol and trimethylolpropane had slightly better flexibility.

TABLE 107. COMPARISON OF ELASTOMERS PREPARED FROM EO-THF POLYETHERS (102)

Ave. Equiv. Wt. of Polyols: 1300
DCB, moles: 1.35
NH_2/OH Ratio: 0.9
Excess NCO, Equiv./10,000 g.: 1.4

Code	1689-P	1689-W
Polyol Blend		
3,900 M.W. Triol, moles	1	-
5,900 M.W. Triol, moles	-	1
TMP, moles	-	0.5
Young's Modulus, E		
-55°C.	6,500	5,500
-50°C.	5,200	4,400
-45°C.	4,700	3,500

Electrical and Thermal Properties

The volume resistivity of a urethane casting material at interested temperatures is given below.

Casting Material	Parts of Weight	Volume Resistivity, ohm-cm* (82)		
Solithane 113 Polyurethan	100	-65°F	73.5°F.	160°F
Catalyst 320	26.5	7×10^{15}	3×10^{14}	1×10^{13}

*ASTM D-257; 1/8 " thick specimen at 600 volts, reading taken 1 minute after applied voltage.

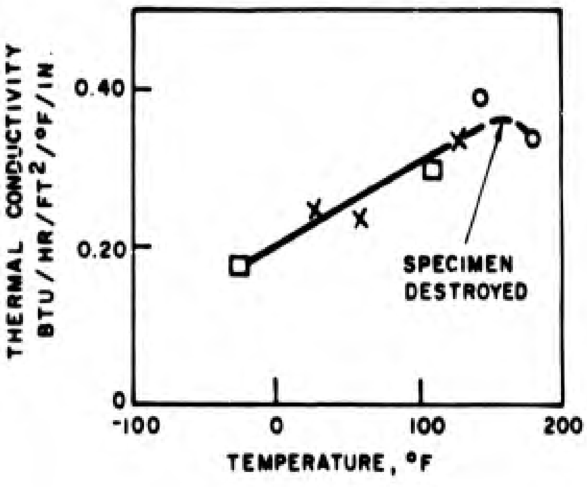
The average coefficient of linear expansion on the same material is given as 7×10^{-5} in/in/°F over the temperature range -65° to 160°F.

PART III - FOAMS

The properties of foams are a function of base plastic resin, the density, type of cells (open or closed), blowing agent and cell size in addition to temperature. Density, cell wall size and orientation primarily influence the mechanical properties. The type of cells, density and gas within the cells affect the thermal conductivity. Density and blowing agents also influence electrical properties.

SECTION XXIII. EPOXY

Figure 215 plots the thermal conductivity of an epoxy foam. (83) Values were low, ranging from 0.18 Btu/hr/ft^2/° F/ in at -30° F to 0.37 Btu/hr/ft^2/° F/ in at 150° F. It should also be noted that the specimen began to collapse at about 150° F.



14 inch Apparatus
□ Gum Rubber Filler
○ Fiberfrax Filler

7 inch Apparatus
X Gum Rubber Filler

Figure 215. Thermal conductivity in the thickness direction of 10 lb/ft^3 epoxy foam (Scotchcast) (83)

SECTION XXIV. PHENOLICS

Thermal Properties

The thermal expansion of a rigid phenolic foam was measured in a quartz tube dilatometer of Bureau of Standards Design. (92) The results in Figure 216 show that the specimen permanently deformed upon cooling from room temperature to -50° F. That is, it did not return to its original length upon reheating to room temperature. The material also began to collapse at 180° F. The total expansion and coefficient of the phenolic foams were significantly smaller than other foams tested. (92)

The thermal conductivity of the same material is given in Figure 217. The value ranged from .2 Btu/hr/ft^2/° F/ in at -50° F to .32 Btu/hr/ft^2/° F/in at 200° F. The values were as expected due to the low densities. That is, the void content has a greater effect on conductivity than the resin.

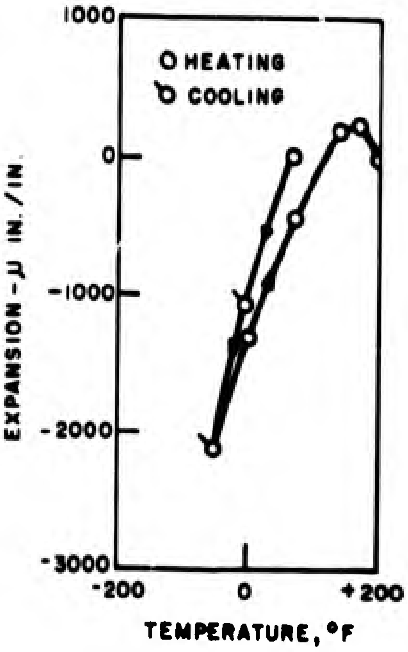
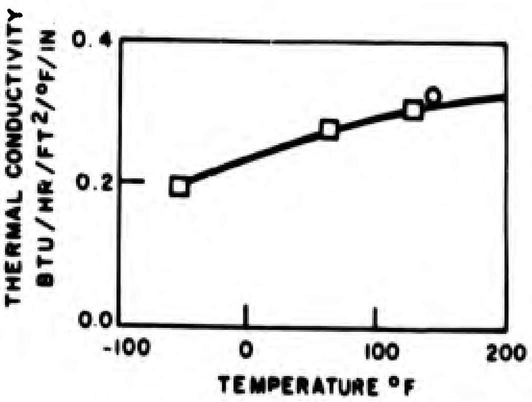


Figure 216. Thermal expansion in the longitudinal direction of rigid (9.8 lb/ft^3 density) phenolic foam (92)



14 inch ASTM C177 Apparatus

☐ Gum Rubber Filler

O Fiberfrax Filler

Figure 217. Thermal conductivity in the thickness direction of rigid (9.8 lb/ft^3) phenolic foam (92)

SECTION XXV. POLYETHYLENE

Mechanical Properties

Figure 218 is a typical stress-strain curve for a low-density (2 lb. per cu. ft.) polyethylene foam. The strength increases with decreasing temperatures as with other polyethylene materials (molding, film, etc.).

Figure 219 plots typical deceleration-load data for low-density polyethylene foam from 30 inch drops on 2, 3, 4 and 5 inch thick materials at two temperatures, -65° F. and 155° F. The reduction in cushioning with decreasing temperatures is expected due to the stiffening of the foam.

An item suspended on a foam cushioning material has a natural frequency of vibration. When an item is vibrated at that frequency it will vibrate many times harder than at a frequency just above or below that frequency. This movement can cause the item tobe damaged or the vibrational force may break down the foam.

Figure 220 gives the relationship between natural frequency and static stress for four thicknesses of polyethylene foams at three temperatures. These data were obtained by empirical calculation and should be regarded as only first approximations. They do, however, clearly demonstrate the increase in frequency with decreasing temperatures.

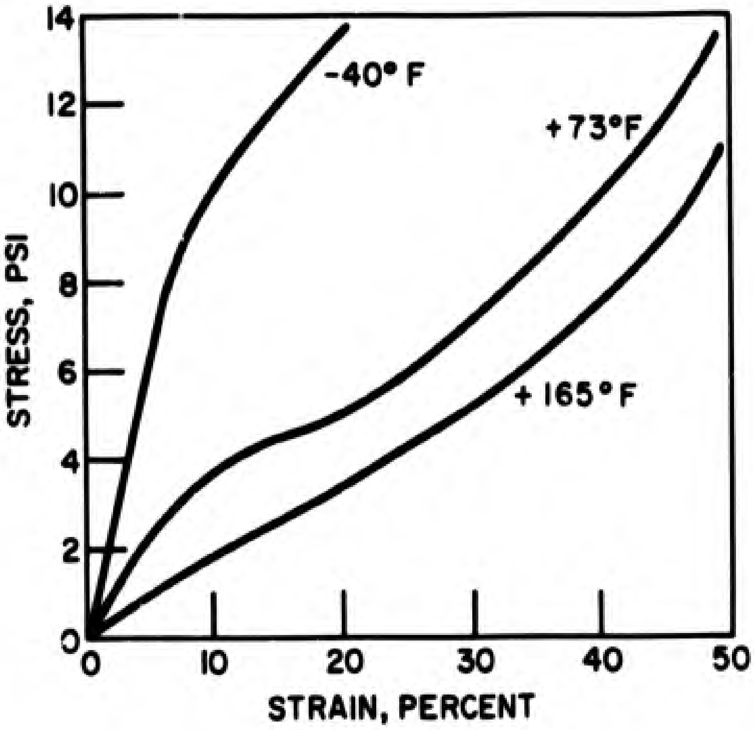


Figure 218. Compressive stress-strain of low density polyethylene foam at various temperatures (103)

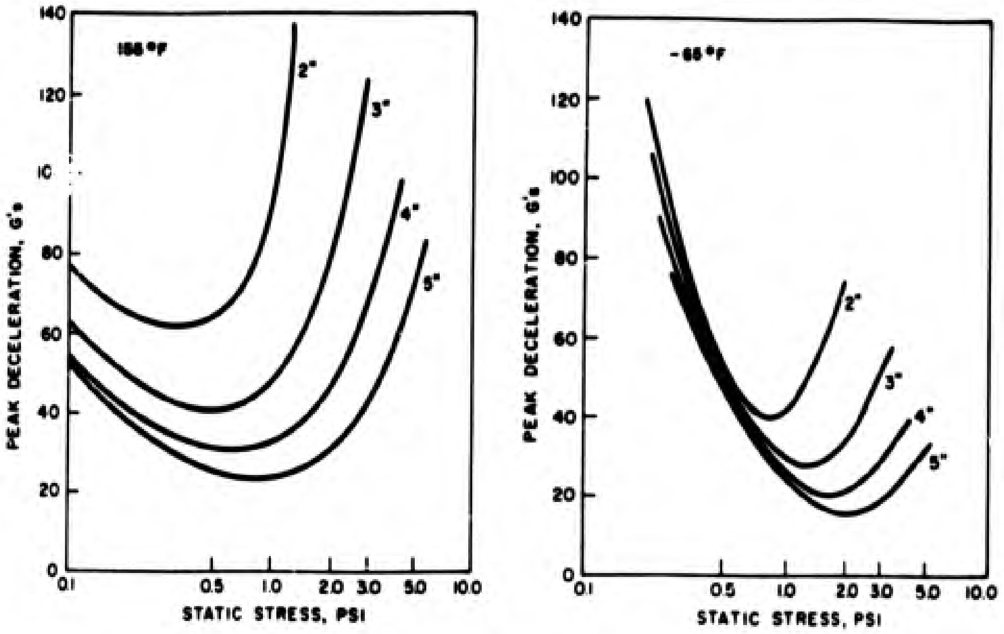
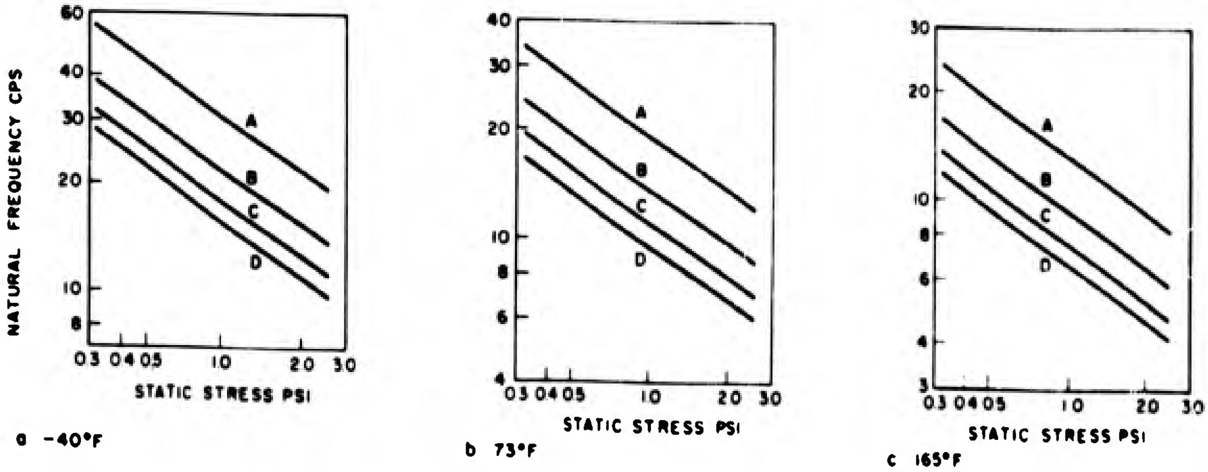


Figure 219. Dynamic cushioning of low-density polyethylene foam from a 30" drop height at 155° F. and -65° F. (103)



a -40°F

b 73°F

c 165°F

A - 1" Thickness
B - 2" Thickness

C - 3" Thickness
D - 4" Thickness

Figure 220. Natural frequency vs static stress of polyethylene (Ethafoam) foam at -40°, 73°, and 165° F. (104)

SECTION XXVI. POLYSTYRENE

Mechanical Properties

Table 108 shows some average values for mechanical properties of various density molded expandable polystyrene in our interested temperature range. As can be seen, properties are more affected by density than temperature. The strength increases with both density and decreasing temperature.

TABLE 108. TYPICAL PROPERTIES OF MOLDED EXPANDABLE POLYSTYRENE (103)

	Density, lb. per cu. ft.				
	1.0	2.0	3.0	4.0	5.0
Tensile strength, psi (speed - 0.05 in./min.)					
-25° F.	30	60	81		-
73° F.	33	49	67		128
160° F.	30	50	68		-
Ultimate elongation, %	4	5	3	2	
Compressive strength, psi (speed -0.05 in./min.)					
-25° F.	18	41	46		-
73° F.	19	29	45		92
160° F.	12	26	38		-
Flexural strength, psi (speed - 0.10 in./min.)					
-25° F.	41	73	107		-
73° F.	45	72	100		260
160° F.	36	69	100		-

An investigation is reported which involved the static testing of resilient expanded polystyrene foams (Resilo-Pak) at low temperature (-80° F.), room temperature (70° F.) and 165° F. (105) The stress-strain curves in Figure 221 show the increase in strength with decreasing temperatures and increasing density.

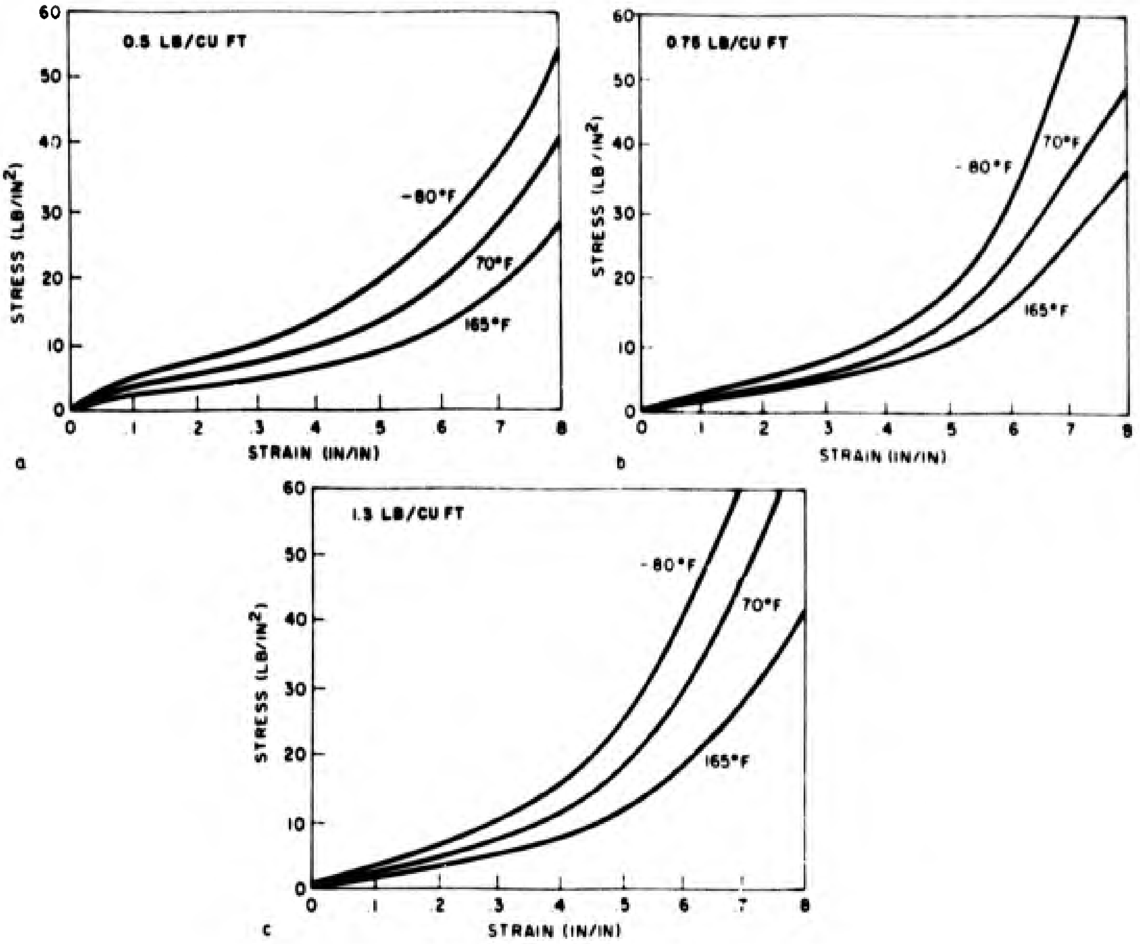


Figure 221. Static stress vs strain curves for resilient expanded polystyrene foam (Resilo-Pak) at temperature extremes (0.5, 0.75 and 1.3 lb./cu. ft. densities) (105)

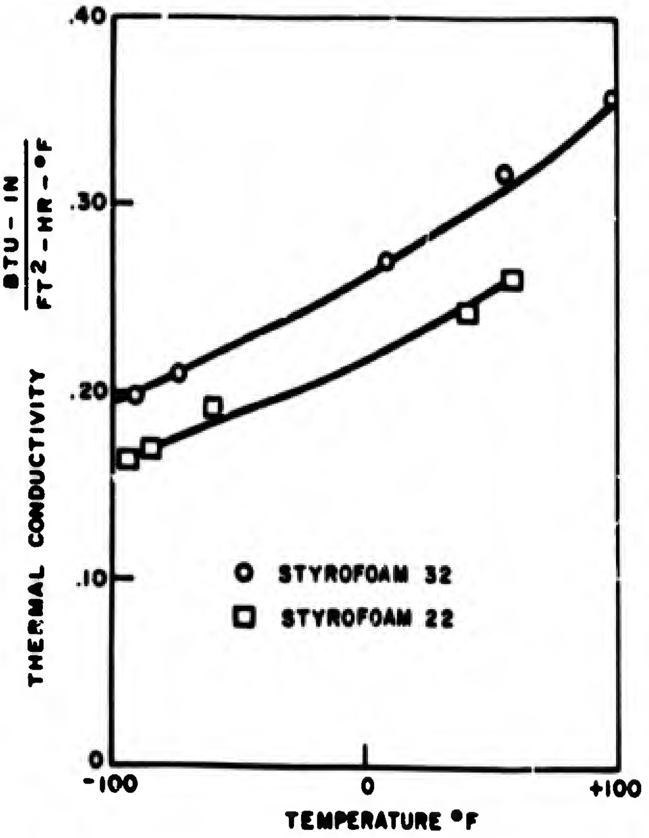
Electrical and Thermal Properties

The volume resistivity (ohm-cm) of self-expanding polystyrene foam (Styrofoam) is reported as: $>4 \times 10^{16}$ at -65° F, 73.5° F, and 160° F.

The thermal conductivity of two polystyrene foams, Styrofoam 22 and fire-retardant Styrofoam 33 were measured by the hot plate method using ice water, dry ice and alcohol as constant temperature baths to maintain a fixed outside wall temperature of the test samples. (106) Specimens were 8" x 8" x 1/2" thick. Figure 222 shows that the fire-retardant Styrofoam 33 was a poorer insulator than Styrofoam 22.

Figure 223 plots the thermal conductivity of extruded polystyrene foam versus temperature. At relatively low densities, the conductivity is a function of cell size and mean temperature.

Figure 224 illustrates the effect of density and temperature on thermal conductivity of molded polystyrene foam.



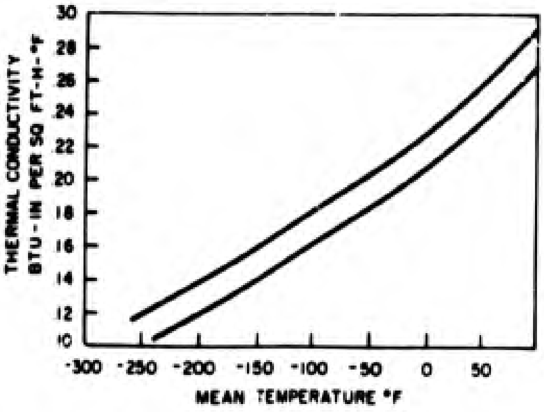


Figure 223. Thermal conductivity of Type A extruded polystyrene foam vs mean temperature (103)

Figure 222. Thermal conductivity of polystyrene foam vs mean temperature (106)

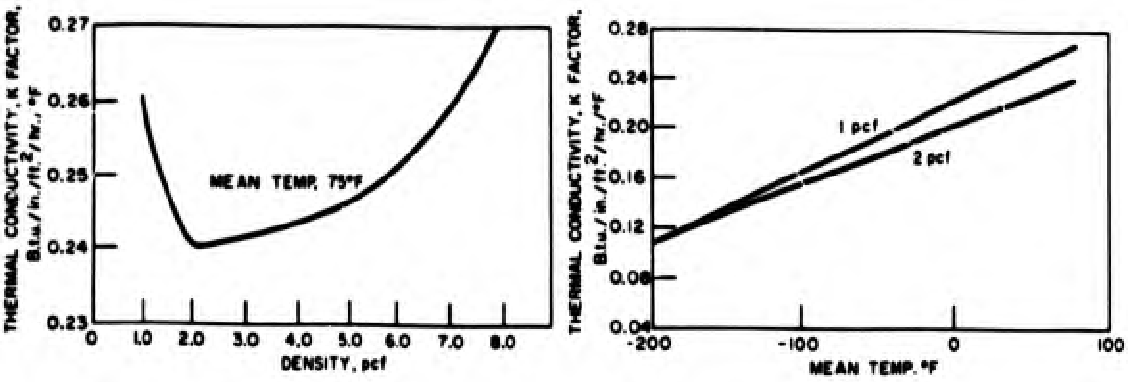


Figure 224. Thermal conductivity of molded polystyrene foam vs density (left) and temperature (right) (5)

SECTION XXVII. POLYURETHANES

Mechanical Properties

Load-compression, resiliency, and hardness tests were conducted on different resilient polyurethane foams in the density range of 2 to 3 lbs. per cubic foot.(107) The material was stored at 74 ±2° F and 50 ±3 percent R.H. prior to low temperature conditioning for 5 ±1/4 hours at -20, -40 and -67°F. before testing.

The specimens for all load-compression tests were 1-5/8 in. in diameter by about 1 in. They were cut with a knife blade held at proper radius in a drill press using the procedure described in Method 12141 of Fed. Test Method Std. No. 601, except that they were not lubricated or frozen.

The specimens were compressed twice to 25 ±5 percent of their "as received" height, rested for 10 ±5 minutes and then measured with a dial gage micrometer equipped with a 1/4 in. diameter cylindrical foot without the 3 ounce weight. An aluminum shim, having a diameter of 1-5/8 in., a thickness of 0.124 in., and a weight of 11.3 grams, was centrally positioned on the specimen to prevent indentation of the micrometer foot into the specimen. The height of the specimens following low temperature conditioning was measured as described above except that the specimens were not compressed prior to this measurement.

The Yerzley Oscillograph was used to measure the load compression in both the initial and low temperature conditions. The data is given in Table 109.

The percent compression values in the table are those at -20, -40 and -67° F at the loading rate required to obtain a 25 percent compression at room temperature.

The data show that the compression of the polyurethane foam materials at low temperatures was very small when subjected to the same load which produced 25 percent compression at room temperature. Only two materials exhibited more than 7.5 percent compression after 5 hours at -20°F. At -40°F and -67°F, it produced about 2 percent and 1 percent compression, respectively. In general, the polyester foams were affected more by low temperature than the polyether foams.

A Pandux hardness gage was used to measure the relative resistance to indentation of specimens 1-5/8" in diameter, 1" thick in the initial condition and at -20° F, -40° F and -67° F. The Pandux gage was placed on top of the specimen with no external load and the hardness was measured after a contact period of 15 seconds. The average of two readings, one from each side of the specimen, was taken as the Pandux hardness. The results are shown in Table 110.

The Pandux hardness readings are approximately proportional to the compression resistance values of the foam materials. It was pointed out that once the relationship between Pandux hardness and compression resistance of a material is known, it may be possible to substitute Pandux readings, which take less than one minute to obtain, for Yerzley compression resistance values which take about one hour to obtain.

TABLE 109. COMPRESSION AND CHANGE IN COMPRESSION AT LOW TEMPERATURES OF POLYURETHANE FOAM MATERIALS (107)

Material	After 5 hours at -20 ± 2F		After 5 hours at -40 ± 2F		After 5 hours at -67 ± 2F	
	Compression %	Change in Compression at -20F, %	Compression %	Change in Compression at -40F, %	Compression %	Change in Compression at -67F, %
Suprether Uniform (1)	7.20	71.2	4.20	83.2	1.82	92.7
MS Flexible Stafoam (2)	4.13	83.5	1.10	95.6	0.68	97.3
MH Flexible Stafoam (1)	6.30	74.8	2.55	89.8	1.40	94.4
Polyester SRP-C-114 (1)	6.40	74.4	4.10	83.6	1.55	93.8
Polyester SRP-1104P-C106 (2)	5.45	78.2	2.30	90.8	1.30	94.8
Nopcofoam S 20-3 (2)	3.40	86.4	1.57	93.7	1.40	94.4
Nopcofoam SC 20-3 (2)	2.14	91.4	1.80	92.8	0.88	96.5
Nopcofoam TX 20-1 (1)	3.20	87.2	2.50	90.0	0.98	96.1
High Grade (2)	2.25	91.0	0.87	96.5	0.66	97.4
Hewitex H2002 (1)	8.00	68.0	4.18	83.3	1.74	93.0
Hewitex A2516 (2)	2.33	90.7	1.42	94.3	0.68	97.3
LD 314 (1)	3.87	84.5	2.23	91.1	1.18	95.3
Polyfoam Type 1211 (1)	7.50	70.0	2.51	90.0	0.92	96.3
Cush-N-Foam 0.04 (2)	1.48	94.1	0.83	96.7	0.47	98.1
Cush-N-Foam 0.03 (2)	1.72	93.1	0.87	96.5	0.70	97.2
Cush-N-Foam 0.06 (2)	2.50	90.0	0.57	97.7	0.42	98.3
152-A-826 (1)	4.83	80.7	2.78	88.9	2.12	91.5
Vibrafoam (2)	3.25	87.0	0.95	96.2	0.54	97.8
SR-100 (2)	29.3	88.3	1.78	92.9	0.59	97.6

(1) - Polyether Urethane
(2) - Polyester Urethane
Note: Compression - Percent Compression at loading rate required to obtain 25% compression at room temperature.
Change in Compression - Percentage change in compression when subjected to the load required to obtain 25% compression at room temperature.

The Bashore resilience was measured on the specimens immediately following the Pandux hardness determinations. In this test, the specimen (1-5/8 in. diameter) was placed on the anvil and the hammer was allowed to fall freely to strike and rebound from the center of the specimen. The height of rebound on the third consecutive fall was taken as the Bashore resilience. (See Table 111.)

The data indicates that these foams are not very resilient. The increase in resiliency of the materials at low temperatures is probably due to hardening of the specimens.

Table 112 gives compression-deflection data for polyester-type urethane foam over our interested temperature range. Data was obtained on an Instron tester after 2 hours' conditioning at the test temperature.

TABLE 110. PANDUX HARDNESS OF THE POLYURETHANE FOAM MATERIALS IN THE INITIAL CONDITION AND AFTER CONDITIONING AT LOW TEMPERATURES (107)

	Pandux Hardness, Points			
		After Conditioning 5 Hours At		
Material	Initial	-20 ° F.	-40 ° F.	-67 ° F.
Suprether Uniform (1)	28	56	76	94
MS Flexible Stafoam (2)	67	95	98	97
MH Flexible Stafoam (1)	43	76	88	96
Polyester SRP-C-114 (1)	41	68	78	96
Polyester SRP-1104-C106 (2)	95	98	98	98
Nopcofoam S20-3 (2)	58	95	97	98
Nopcofoam SC 20-3 (2)	61	98	97	97
Nopcofoam TX 20-1 (1)	18	92	82	95
High Grade (2)	73	95	98	98
Hewitex H2002 (1)	21	57	77	94
Hewitex A2516 (2)	71	95	97	96
LD 314 (1)	43	82	92	96
Polyfoam Type 1211 (1)	32	61	88	96
Cush-N-Foam 0.04 (2)	50	95	98	98
Cush-N-Foam 0.03 (2)	52	95	98	98
Cush-N-Foam 0.06 (2)	62	95	98	97
152-A-826 (1)	45	85	92	92
Vibrafoam (2)	62	94	98	98
SR-100 (2)	65	88	97	98

(1) - Polyether Polyurethane
(2) - Polyester Polyurethane

As can be seen, low-temperature flexibility of this material is poor. Also, these tests were conducted down to only -40° F, not to the specified military requirement of -65° F.

Figure 225 plots the stress versus deflection (d/T: deflection of specimen, in inches, divided by specimen thickness, in inches) for a 3" thick polyurethane foam at -65° F and 165° F. The effect of density at low temperatures is apparent.

Figure 226 illustrates the effect of low temperature on the accelerations of a typical flexible polyurethane foam of a specific thickness and density. As expected, the accelerations will generally be higher for a drop test at -65° F. than at 70° F.

A test program on urethane foams was conducted to: (1) determine the effects of moisture at low temperature on cushioning; (2) evaluate changes in load-bearing

TABLE 111. BASHORE RESILIENCE OF THE POLYURETHANE FOAM MATERIALS IN THE INITIAL CONDITION AND AT LOW TEMPERATURES (107)

	Bashore Resilience, Height of Rebound, Units			
		After Conditioning 5 Hours At		
Material	Initial	-20° F.	-40° F.	-67° F.
Suprether Uniform (1)	1	4	2	2
MS Flexible Stafoam (2)	3	6	6	5
MH Flexible Stafoam (1)	3	7	7	8
Polyester SRP-C-114 (1)	1	7	6	0
Polyester SRP-1104-C106 (2)	7	16	13	15
Nopcofoam S 20-3 (2)	3	14	8	9
Nopcofoam SC 20-3 (2)	4	9	8	9
Nopcofoam TX 20-1 (1)	1	8	6	6
High Grade (2)	11	5	4	1
Hewitex H2002 (1)	0	5	3	3
Hewitex A2516 (2)	4	6	6	1
LD 314 (1)	1	6	5	4
Polyfoam Type 1211 (1)	1	5	5	4
Cush-N-Foam 0.04 (2)	1	5	6	5
Cush-N-Foam 0.03 (2)	2	6	8	9
Cush-N-Foam 0.06 (2)	3	4	1	1
152-A-826 (1)	0	11	11	13
Vibrafoam (2)	2	3	2	2
SR-100 (2)	2	5	3	1

(1) - Polyether Polyurethane
(2) - Polyester Polyurethane

TABLE 112. COMPARISON OF COMPRESSION-DEFLECTION DATA FOR A POLYESTER-TYPE URETHANE-FOAM FLEXED AT VARIOUS TEMPERATURES (105)

Test Temperature	-40° F.	0° F.	+40° F.	+80° F.	+100° F.	+120° F.	+140° F.	+160° F.
Compression-deflection at 25%, psi	7.4	2.1	1.7	0.9	1.0	1.0	1.0	1.0
Compression-deflection at 50%, psi	7.8	2.5	2.0	1.1	1.2	1.2	1.2	1.2
Compression-deflection at 75%, psi	16.0	5.8	4.6	2.6	2.7	2.7	2.7	2.7

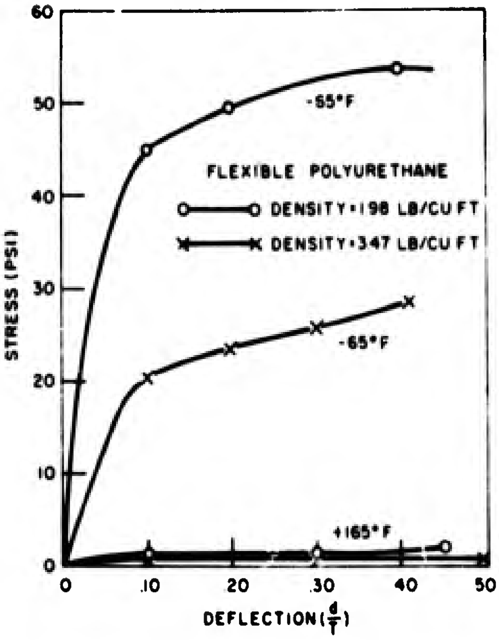


Figure 225. Static load-deflection curves for polyurethane foams at temperature extremes (105)

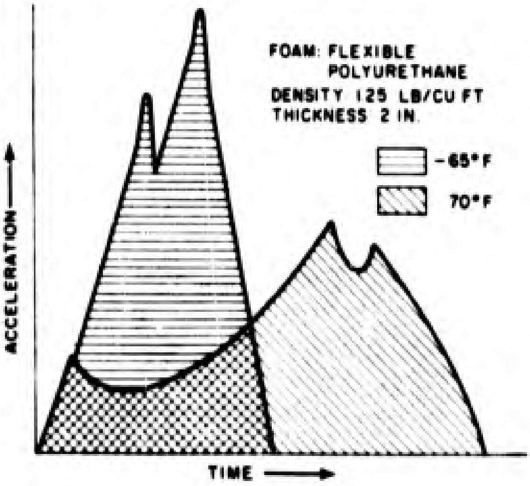


Figure 226. Acceleration vs time traces for a typical flexible polyurethane cushioning material dynamically loaded at 70 ° F. and -65 ° F. (105)

capacity by the static load-deflection and dynamic drop test versus temperature; (3) determine if a correlation exists between static compression and dynamic drop tests data; (4) and to evaluate the dynamic cushioning properties of other foam materials at -65 ° F. (108)

The material selected for the low temperature comparison of static and dynamic testing was a polyurethane foam of 4.1 ±0.4 pounds per cubic foot, an ultimate strength of 30 ± 3.0 psi with approximately 210 percent elongation.

Figure 227 shows the results of static tests compressed at a constant rate of 2 inches per minute. These load deflection curves show a yield point* and the hysteresis effect (energy absorbed by the foam material during compression). The authors explain that this method of testing is considered static because the compression is relatively slow, allowing the foam to nearly reach equilibrium condition of load for a given deflection during the progress of the tests. It is pointed out that the rate of compression of the foam changes the load-deflection curve obtained. Rapid compression raises the curve slightly; however, within the compression rates of 0.2 to 4.0" per min., the flow curve can be considered as constant and the test static. The test pads were 4.5" x 4.5" x 3" thick.

Consideration was given to the possible effect of moisture from laboratory air (40 - 70 percent R.H.) on the flow curve of the urethane foam during low temperature compression tests. It was felt that fine layers of ice in the foam matrix could significantly affect the load versus deflection curve.

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* The yield point is defined as the maximum load obtained prior to elastic breakdown of the foam, resulting in a rapid deflection for a slight increase in load.

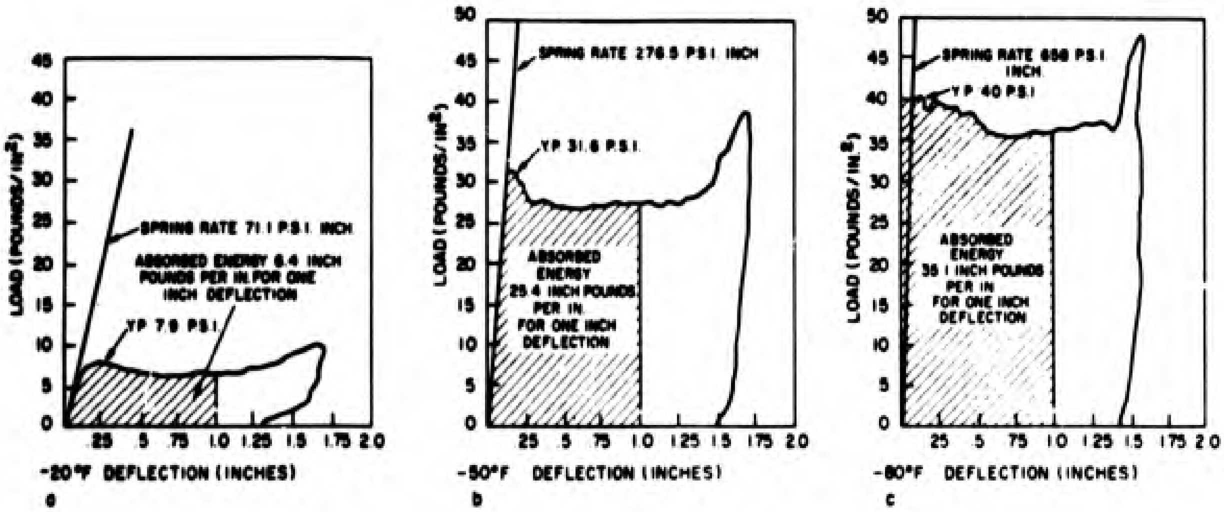


Figure 227. Urethane foam static compression curves at -20°, -50° and -80 °F. (108)

Compression curves were run on samples containing the moist laboratory air and on dried pads (equilibrated in atmosphere of dry nitrogen, dew point -60 °F and dry argon, dew point -130 ° F) at temperatures of -65 °, -50 ° and -35 ° F. The flow curves obtained with moisture laden foams were identical to the dried foam curves. From this it was concluded that moisture had a negligible effect on the flow curve of the foams measured at temperatures below zero.

Although not stated in the original work, this author believes these samples were of the closed cell construction which could account for this result.

The spring rate (psi per inch of deflection) shown in Figure 227 was obtained from the initial linear portion of the load deflection curve. These become more difficult to measure at low temperature since the slopes become so steep that small errors are greatly magnified. This function was plotted against temperature in Figure 228. It should be noted that at -10 ° F the curve begins to rise rapidly with decreasing temperature, showing increase in resistance to deformation of the foam samples.

The yield point, another function of the load deflection curve, is plotted in Figure 229. This curve is similar to the spring rate curve in that it begins to increase sharply at -10 ° F.

The last measure of increase in hardness of foams versus temperature is the measure of energy absorbed during the compression of the foam. Figure 230 shows a plot of absorbed energy versus temperature. Again, a rapid increase in total energy absorbed during a compression cycle is obtained as temperature is lowered below -10 ° F.

As can be seen, at -10 ° F, all three functions rise with decreasing temperature, until at -55 ° F to -65 ° F they flatten out and tend to remain constant with decreasing temperature. Based on the above it would be expected that at -10 ° F, the cushioning ability of urethane would begin to degrade. Dynamic drop tests were conducted to verify this assumption.

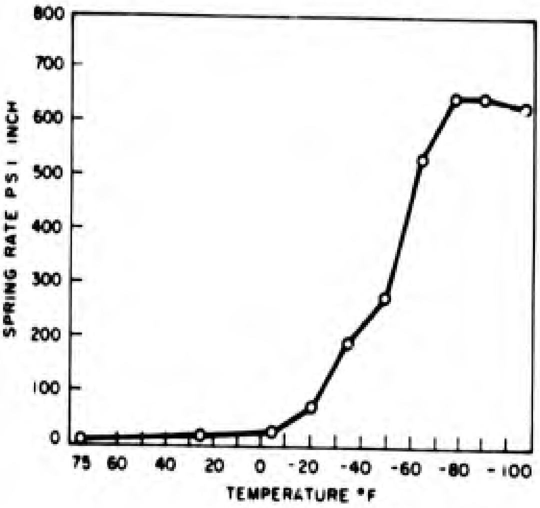


Figure 228. Urethane foam spring rate versus temperature (108)

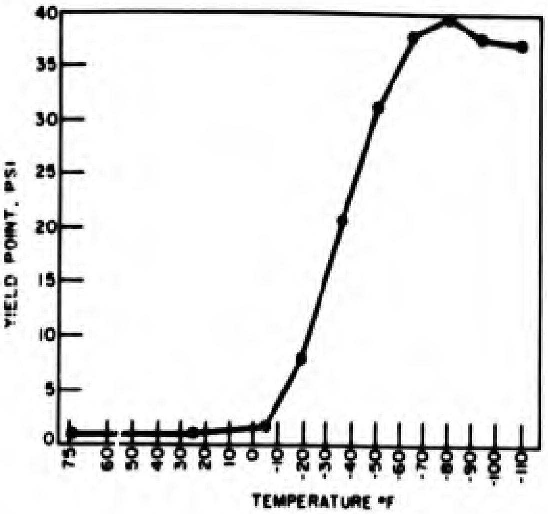


Figure 229. Urethane foam yield point versus temperature (108)

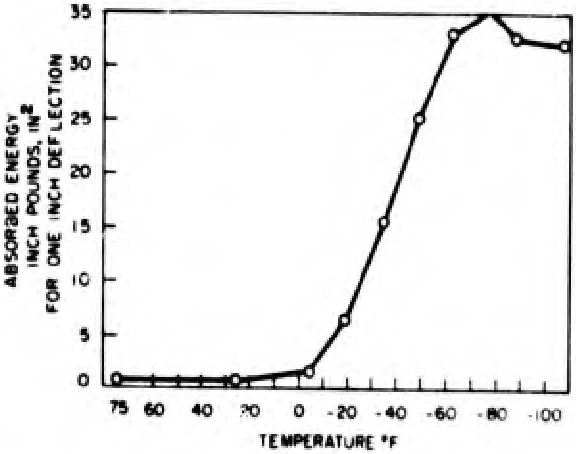


Figure 230. Urethane foam absorbed energy versus temperature (108)

Urethane foam pads 6" x 10" x 3" were positioned in a Hughes Aircraft Company's drop test jig with a 0.5 psi load and a 15 percent precompression. This was to approximate a missile dead weight loading on the bottom of the pad and the additional precompression when the container lid was closed. Three drops were made from 8, 12 and 16 inches at eight temperatures from +75° F to -85° F. The total time elapse (removal from cold box to end of test) was approximately three minutes.

The initial 8" drop was chosen since Hughes' missile specifications frequently limit the shock load to the 30 g's in the temperature range +160° F to -65° F and it was found that the 8" drop resulted in an average of 24 g's. It was pointed out that the g values at the other heights may contain slight errors as a result of softening of the pads due to the energy input from the previous drop; and softening from the slight warm up of the fixture.

Figures 231 and 232 show peak acceleration versus temperature for 8, 12 and 16" drops.

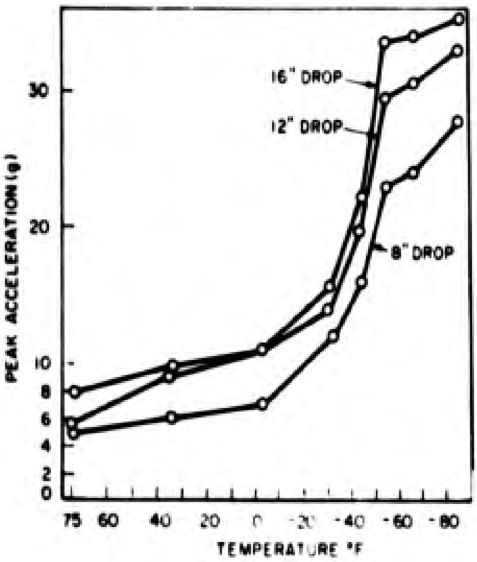


Figure 231. Peak acceleration versus temperature for 8, 12 and 16-inch drop (108)

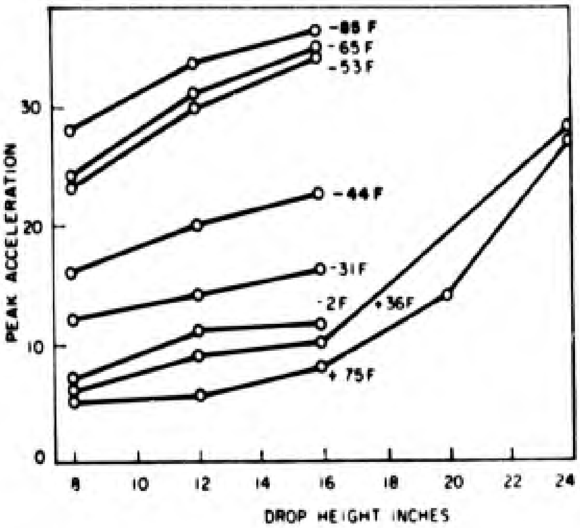


Figure 232. Peak acceleration versus drop height (108)

The curves are very similar to the static test functions of spring rate, yield point and absorbed energy versus temperature. Both the static and dynamic functions increase as the temperature is lowered (sharply around -10° F) to values of -55° F to -65° F; the functions remain constant to -85° F and 110° F respectively.

Figure 232 shows the effect of increased drop height on the peak shock of the cushioned simulated missile section. As the temperature is lowered, the curve for each series of drops moves to higher values of shock.

Based upon the above data, static compression tests were conducted on the below listed materials. (108) Only two classes were found to have superior low temperature properties: the silicones and a fiberglass flexible pad material. All others showed a large increase in yield point at -65° F. It was stated that some of these may have better cushioning at some intermediate temperature, but tests were only run at -65° F.

Material Class

Polyethylene

PVC

Polyurethane

Acrylonitrile rubber

Neoprene rubber

Silicone

Fiberglass flexible material

The load-deflection curves of the three silicone and the fiberglass flexible material were found to be identical at -65 ° F and +75 ° F. However, both materials have other limitations.

Figure 233 compares the flexibility of flexible urethane foams with latex foam rubber in terms of Clash-Berg torsional stiffness at various temperatures. (103) As can be noted, the adipic acid polyester foams began to stiffen significantly at about -10 ° to -25 ° C. The dimer acid foam stiffened gradually. However, in the temperature range of -10 ° to -30 ° C it reached a given modulus at about 15-20 degrees lower than the adipic acid polyester foam. The polyether foam showed an additional 10 ° advantage. It reached an apparent modulus of 2000 psi at -40 °, compared to -30 ° for dimer acid and -10 ° for adipic acid polyester foam. The latex foam reached this modulus at -53 ° C.

The Clash-Berg torsional curves in Figure 234 for foams prepared from polyether triols demonstrate the effect of molecular weight on low temperature flexibility. The higher molecular weight materials had the best low temperature modulus. Also, foams (of equal aromatic and urethane contents) with the highest crosslink density will better retain their flexibility at lower temperatures. (103)

Change in temperature does not affect the physical properties of rigid urethane foam in the same way nor to the same extent. Figures 235 and 236 show the tensile strength decreases very slowly at lower temperatures, then reaches a maximum slightly above room temperature and decreases rapidly above that point. The elongation at break increases quite rapidly with temperature. The compressive strength does not exhibit a maximum, but decreases with increasing temperature over the entire interested temperature range. In general, the strain at the yield point in compression is not greatly affected by temperature in the range, -100 ° to 250 ° F.

Although not shown, the shear strength, shear modulus and elastic modulus in compression follow the compressive strength curve closely. They all increase with decreasing temperature.

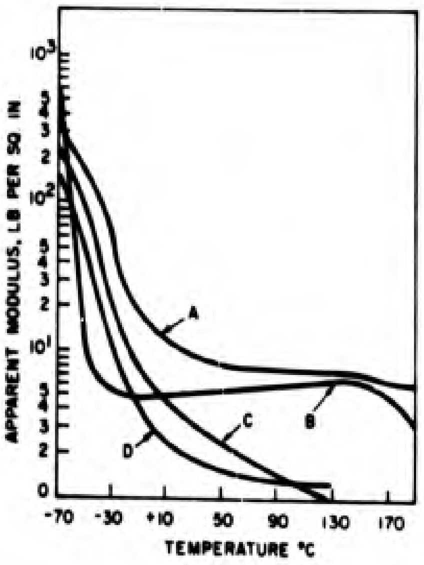


Figure 233. Torsional stiffness properties of flexible foam (Clash-Berg) (103)

A - Adipic acid polyester urethane foam
B - Latex foam rubber
C - Dimer acid polyester urethane foam
D - Polyether urethane foam

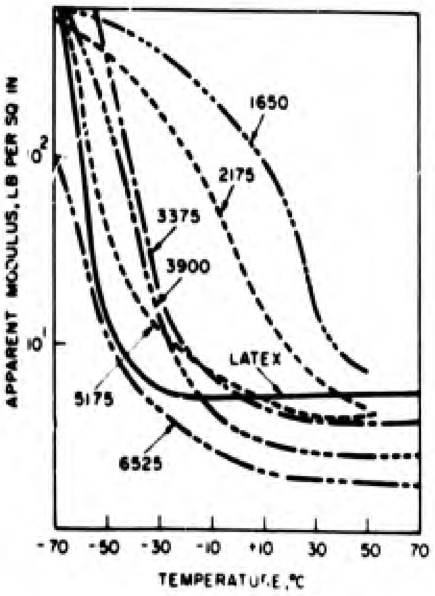


Figure 234. Clash-Berg torsional curves, showing variations of flexibility in function of temperature, for various molecular weight formulations (103)

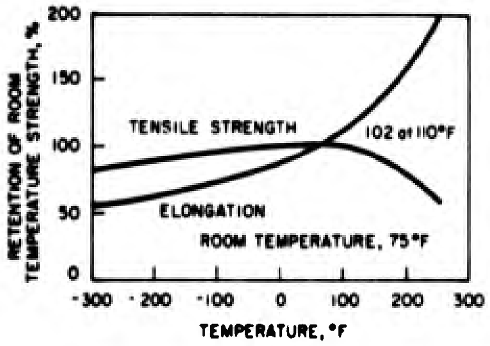


Figure 235. Effect of temperature on tensile strength and elongation of rigid urethane foam. Density: 1.8 lb per cu ft. High cross-link density (calculated average distance between cross-links = 350) (103)

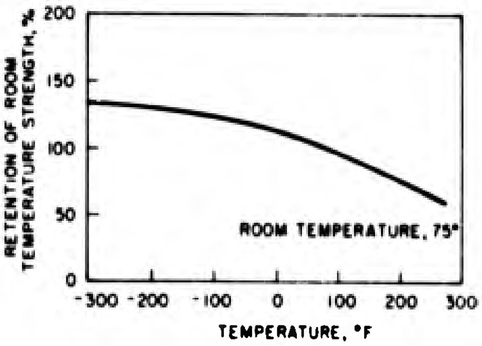


Figure 236. Effect of temperature on compressive strength of rigid urethane foam (103)

As previously stated, the foam density has an effect on the rate and extent of change of a physical property with temperature. The higher density foams are influenced less by temperature than low density foams. Tensile strength is not affected by temperature as much as compressive strength. The effect of temperature on shear strength and flexural strength is about intermediate between tensile and compressive strengths. Elastic modulus in compression or tension shows about the same change with density.

The ability of rigid urethane foam to maintain dimensional stability under various conditions is influenced to a large extent by the base polyol used. The average crosslink density is determined by the polyol and the NCO/OH* ratio used. Usually, foams having low crosslink density have poor dimensional stability at both low and high temperatures. (Figure 237.) Those having high crosslink density have good dimensional stability. However, if the crosslink density is too high, the foams may become brittle.

The dimensional stability of foams formulated to an NCO/OH ratio of 1.05 to 1.10 was found better than those made with NCO/OH ratios of 0.95 to 1.00, under all exposure conditions studied. (103) (See Figures 237 and 238.)

*NCO/OH ratio refers to the isocyanate hydroxyl ratio.

Cure: 7 days at 75° F.
Exposure: 48 hours under
indicated conditions

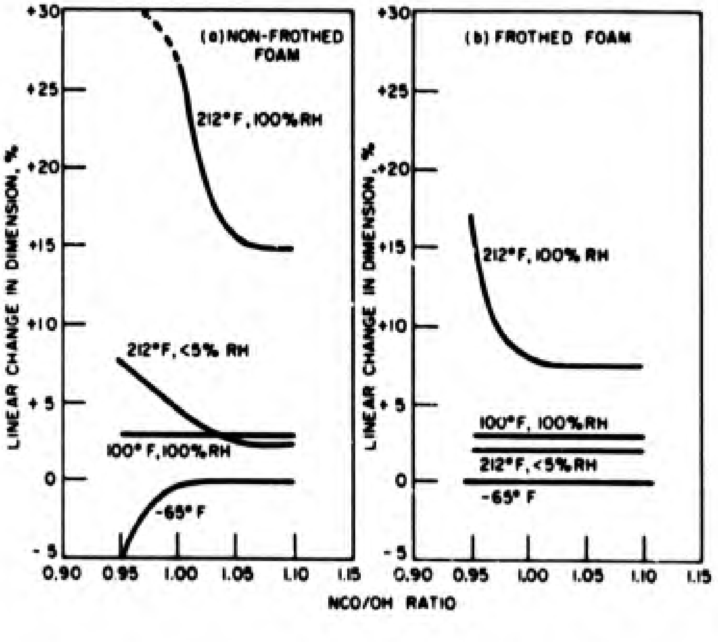


Figure 237. Effect of NCO/OH ratio on dimensional stability (103)

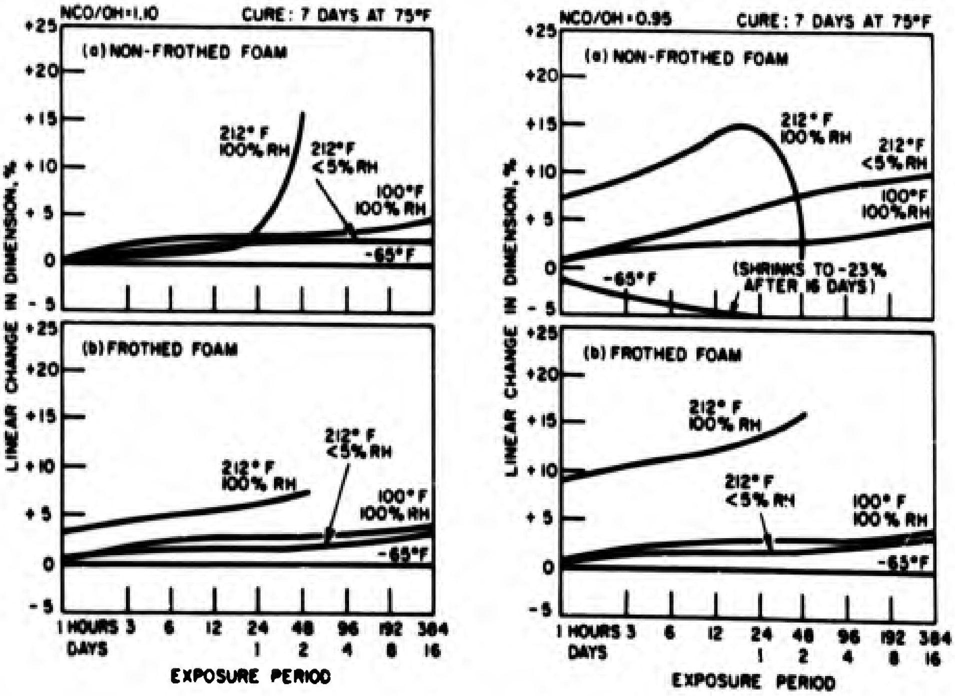


Figure 238. Dimensional stability of rigid urethane foam under various exposure conditions (103)

Poor dimensional stability is also caused by excessive shrinkage at low temperatures (below 75 ° F). Shrinkage occurs when the blowing agent condenses, leaving a partial vacuum in the foam cells, and the foam structure is not rigid enough to withstand the external pressure. This phenomenon was observed in the non-frothed foam formulated to an NCO/OH ratio of 0.95, when it was exposed at -65 ° F. (103)

Electrical Properties

Urethanes are widely used for foaming-in-place. As previously stated, their electrical properties vary with density and density can range from 2 to 20 lb. per cu. ft. Either polyester- or polyether-based urethane foam is used. However, polyethers are generally preferred since they have a smoother compression load resistance curve, have better low-temperature flexibility (at a few degrees below zero) and a better resistance to humidity and to solvents.

The effect of temperature on both dielectric constant and loss tangent is shown in Figure 239. The dielectric constant changes very little with temperature, whereas the loss tangent changes rapidly.

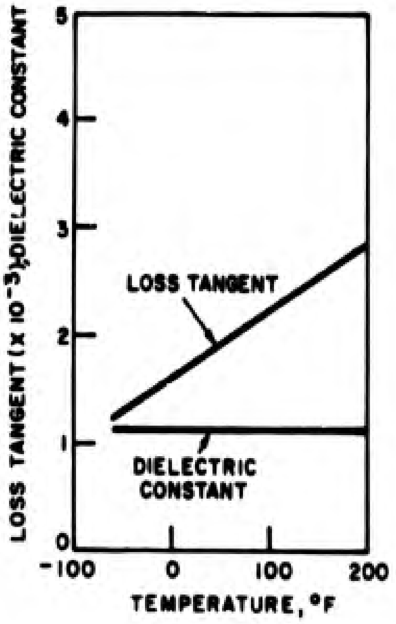


Figure 239. Effect of temperature on dielectric constant and loss tangent of rigid urethane foam (halogenated foam) (103)

The volume resistivity of a rigid polyurethane foam (49 parts Lockfoam BH 610-R resin; 51 parts Lockfoam BH 610-T foaming agent and catalyst) is

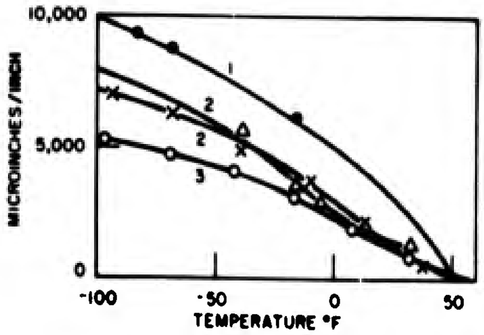
Volume resistivity ohm-cm (82)

-65° F	73.5° F	160° F
1×10^{16}	1×10^{16}	3×10^{15}

Thermal Properties

The coefficient of thermal expansion of Polycel 420 (a $1.84/ft^3$ Freon-blown polyurethane) was measured. (109) The foam was rigid having approximately 96 percent closed cells.

Measurements were made using a Leitz Dilatometer which was modified for low temperature by replacing the furnace with a liquid nitrogen jacket. Measurements were taken under the following conditions: evacuated and purged with dry nitrogen; dry helium atmosphere at atmospheric pressure; atmospheric pressure - air; and vacuum (10-micron). The data is plotted in Figures 240 and 241. Two samples were measured in Figure 241 to give some idea of variation between specimens. These samples were both cut from the same block of material.



1. Dry helium atmosphere-atmospheric pressure
2. Vacuum
3. Atmospheric pressure

Figure 240. Expansion of polyurethane foam (Polycel 420) in various environments (109)

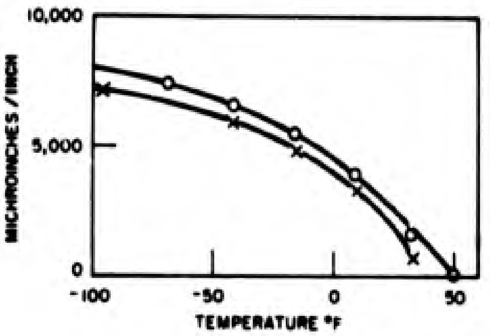
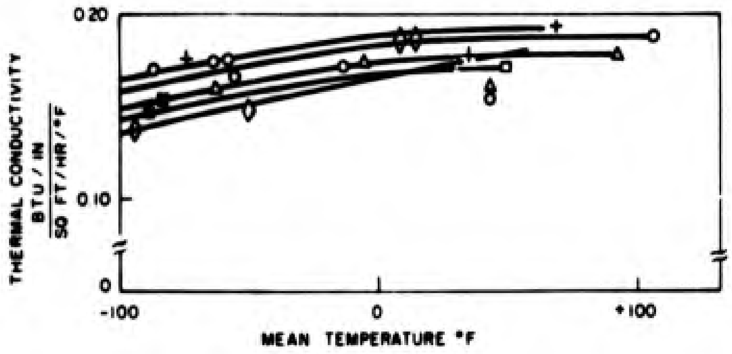


Figure 241. Expansion of Polycel 420, atmospheric pressure - air (two samples) (109)

A hot plate method was designed to measure the thermal conductivities of polyurethane foam insulation materials at low temperatures. (106) Ice water, dry ice and alcohol were used as constant temperature baths to maintain a fixed outside wall temperature of the material being tested. The thickness of all materials was 1/2" and the test panel was 8" square.

Figure 242 compares five Freon-blown polyurethane foams. All except Stafoam AA-3102 are polyether-base foams. The lower thermal values for Stafoam AA-1602 than Stafoam AA-402 is attributed to better molding characteristics and cell structure due to its slower reaction time.

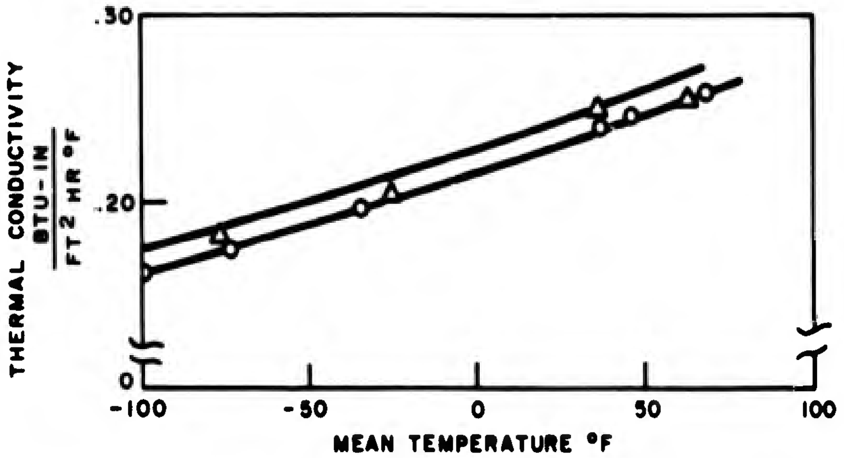


○ Stafoam AA-1602, 2.0 lbs/ft^3
+ Stafoam AA-402, 2.0 lbs/ft^3
△ Polycel 440, 4.0 lbs/ft^3
□ Stafoam AA-3102
◊ Apco 1414

Figure 242. Thermal conductivity of freon-blown polyurethane foams vs mean temperature (106)

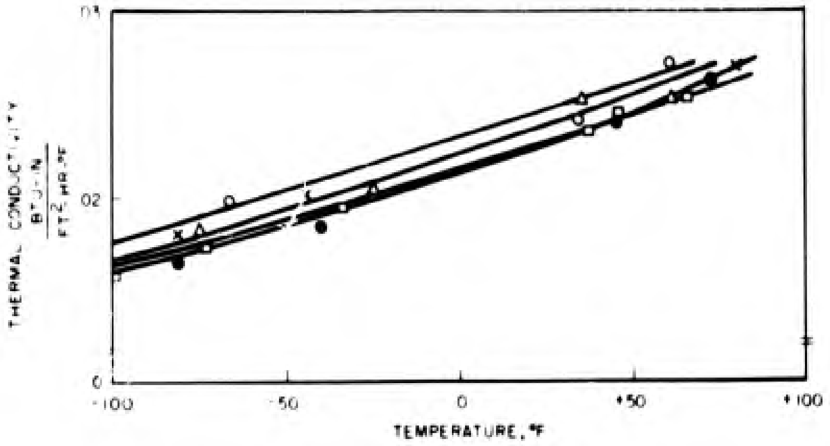
Although higher densities usually result in higher thermal conductivities, the values obtained with the 4.0 lb/ft^3 Polycel 440 were lower than those obtained with Stafoam AA-402 (2.0 lb/ft^3). A comparison of the effects of density on thermal conductivity is shown in Figure 243. PAPI-1008 foam, (2.2 lb/ft^3 density) resulted in a k-factor versus mean temperature curve which was slightly lower and parallel to the curve obtained with the 3.0 lb/ft^3 PAPI-1008 foam. The thermal conductivities of the carbon dioxide (CO_2) blown, high temperature resistant polyurethane foams (Figure 244) and the Freon-blown polyurethane foams were fairly approximate at -100° F, but at 50° F the CO_2 blown foams were about 50 percent higher than the Freon-blown foams. A composite insulation consisting of one-inch fiberglass honeycomb core cells filled with a CO_2 blown foam resulted in a curve similar to those obtained with the PAPI-CO_2 blown foams.

Figure 245 shows the data obtained with two fire-retardant polyurethane foams. The k-factors of both foams at -100° F were close, but the polyester-based casting formulation resulted in a k-factor almost 25 percent higher than the polyether-based spray formulation at 50° F. This is attributed to the finer cell structure of the spray foam.



○ Density - 2.2 lbs/ft^3 △ Density - 3.0 lbs/ft^3

Figure 243. Thermal conductivity of (PAPI foam 1008) polyurethane foam vs mean temperature (110)

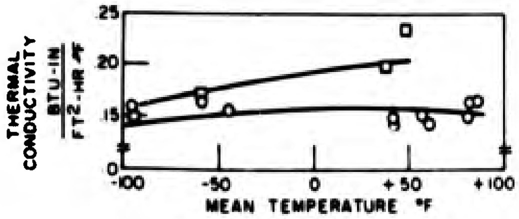


○ X-226-32A, 2.0 lbs/ft^3 } (CO$_2$ blown)
□ PAPI-1008, 2.2 lbs/ft^3
△ PAPI-1008, 3.0 lbs/ft^3
X Laboratory Sample (Composite insulation) 3.05 lbs/ft^3
● Production sample (Composite insulation) 2.92 lbs/ft^3

Figure 244. Thermal conductivity of high temperature insulations vs mean temperature (106)

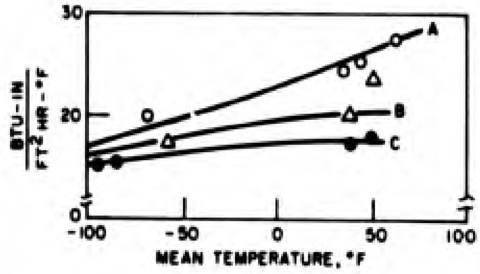
Figure 246 plots the thermal conductivity versus mean temperature of various polyurethane foam insulations, investigated for the Centaur vehicle. (110) Again, the effect of density and blowing agent on thermal conductivity is evident.

Figure 247 plots the thermal conductivity versus temperature for a 10 lb/ft^3 rigid polyurethane foam. Values range from 0.16 Btu/hr/ft^2/° F/in at -50° F to 0.32 at 170° F.



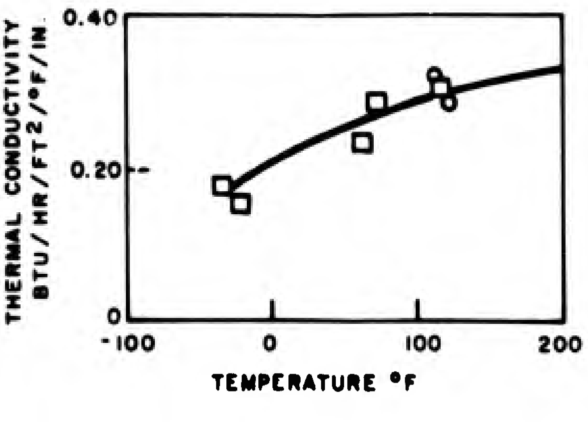
□ Stafoam AA-1802 (Polyester base)
1.80 lb/ft^3
○ Stafoam AA-2802 (Polyether base)
1.99 lb/ft^3

Figure 245. Thermal conductivity of rigid fire retardant freon-blown polyurethane foams vs mean temperature (106)



A = CO_2-blown, polyether (2.0 density)
B = Freon-blown, fire retardant polyester (1.8 density)
C = Freon-blown (1.4 density)

Figure 246. Thermal conductivity of rigid polyurethane foams vs temperature (X-226-32A, APCC 1414, Stafoam AA-1802) (110)

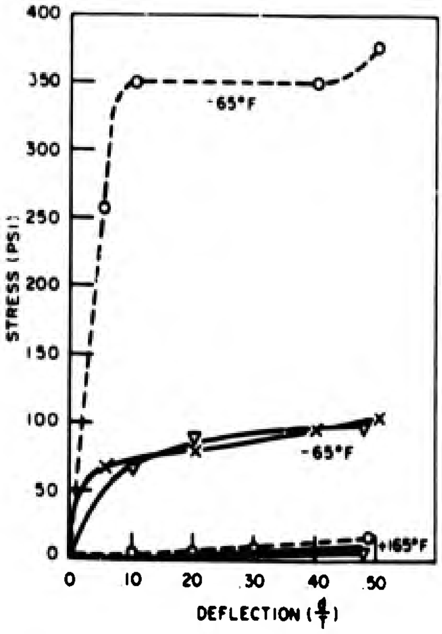


14 inch Apparatus
□ Gum Rubber Filler
○ Fiberfrax Filler

Figure 247. Thermal conductivity in the thickness direction of 10 lb/ft^3 rigid polyurethane foam (Nopcofoam G510)(83)

Mechanical Properties

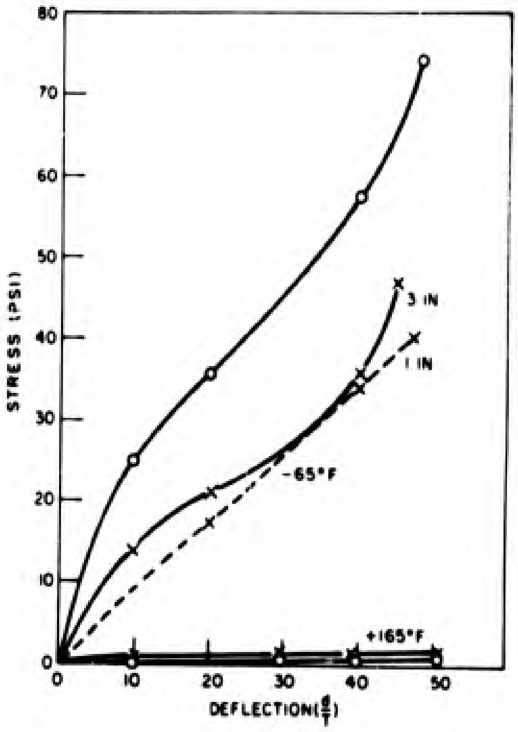
Figures 248 and 249 are static load-deflection curves for various density flexible vinyl foams at two temperature extremes. The effect of density and specimen thickness are much more significant at low temperatures than at high temperatures.



Foam: Flexible Polyvinyl Chloride (Modified)

O---O Density = 10.8 lb/cu ft.
X——X Density = 5.84 lb/cu ft.
∇——∇ Density = 5.01 lb/cu ft.

Figure 248. Static load deflection curves for 3" flexible polyvinyl chloride (modified) foams (105)



Foam: Flexible Vinyl

O——O Density = 7.97 lb/cu ft.
X----X Density = 7.93 lb/cu ft. (1 in.)
X——X Density = 7.93 lb/cu ft. (3 in.)

Figure 249. Static load-deflection curves for flexible vinyl foams, at temperature extremes (105)

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13. ABSTRACT

The effects of low temperature on the mechanical, electrical and thermal properties of plastics is discussed. Data are given where available at three temperatures; namely, low temperature, about -65°F, room temperature and around 160°F to permit complete evaluation. The material is presented by plastic family (in alphabetical order) and is divided into three parts: thermoplastics, thermosets and foams.

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