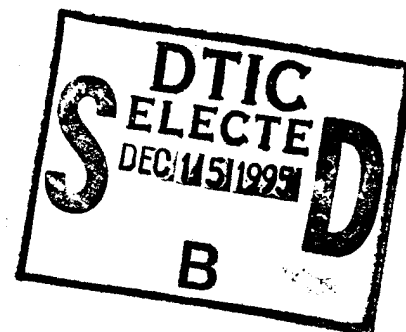


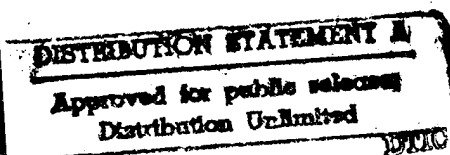
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SELECTED PHYSICAL AND THERMAL PROPERTIES OF VARIOUS FORMULATIONS OF SILICONE POTTING COMPOUNDS

Gary L. Flowers
Bill D. Faubion
Jackie L. Montague
Susan T. Switzer



November 1980



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19951214 081

Process Development
Endeavor No. 102

DEPARTMENT OF DEFENSE
PLASTICS TECHNICAL EVALUATION CENTER
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140355

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Printed in the United States of America
Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161
Price: Printed Copy \$5.00 ; Microfiche 3.50

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-- 1 - AD NUMBER: D433398

-- 5 - CORPORATE AUTHOR: MASON AND HANGER-SILAS MASON CO INC AMARILLO TEX
PANTEK PLANT

-- 6 - UNCLASSIFIED TITLE: SELECTED PHYSICAL AND THERMAL PROPERTIES OF
VARIOUS FORMULATIONS OF SILICONE POTTING COMPOUNDS,

--10 - PERSONAL AUTHORS: FLOWERS,G. L. ;FAUBION,B. D. ;MONTAGUE,J. L. ;
SWITZER,S. T. ;

--11 - REPORT DATE: NOV , 1980

--12 - PAGINATION: 18P

--14 - REPORT NUMBER: MHSMP-80-63

--15 - CONTRACT NUMBER: AC04-76DP-00487

--20 - REPORT CLASSIFICATION: UNCLASSIFIED

--22 - LIMITATIONS (ALPHA): APPROVED FOR PUBLIC RELEASE; DISTRIBUTION
UNLIMITED. AVAILABILITY: NATIONAL TECHNICAL INFORMATION SERVICE,
SPRINGFIELD, VA. 22161, NUCMP 80 00.

--33 - LIMITATION CODES: 1

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Endeavor No. 102

INTRODUCTION

When Dow Corning discontinued production of several of their addition cured silicone potting compounds used by the DOE complex, it became necessary to develop alternate or substitute materials. This task was undertaken jointly by the Lawrence Livermore National Laboratory (LLNL) and Pantex. Several substitutes were developed and tested at both facilities. Much of this work has been reported previously^(1,2,3,4,5,6,7). This report deals with some of the physical and thermal properties of many of these substitutes as well as some of the starting materials.

These substitutes were originally developed as replacements for 93-119, 93-120, 93-122 and mixtures of 93-119/93-120 and 93-119/93-122^a. These substitutes are based on combining various quantities of any or all of six raw materials. These materials include:

Sylgard 186^a - Silicone dioxide filled, high viscosity, slow curing, high strength, two part RTV.

Sylgard 184^a - Medium viscosity, slow curing, high strength, two part RTV.

Q3-6527
Dielectric Gel^a - Low viscosity, very slow curing, very low strength, two part RTV.

^aProduct of Dow Corning, Midland, Michigan

- Q3-6559
Accelerator^a - Low viscosity, vinyl terminated polydimethylsiloxane fluid containing a large concentration of a platinum catalyst.
- DC-1107^a - Low viscosity, silane hydrogen terminated polydimethylsiloxane fluid used as an accelerator.
- Cabosil
MS-75^b - Silicon dioxide filler.

For this particular study, 19 formulations were selected. They include raw material only formulations as well as formulations of products currently in use by the DOE and products considered for future use by the DOE. The test data reported here are comprised of the following categories:

PHYSICAL PROPERTIES

Disk or punch shear strength
Lap shear strength
Butt tensile strength
Compressive strength
Bulk tensile strength
Durometer hardness
Dynamic shear modulus
Density
Crosslink density

THERMAL PROPERTIES

Coefficient of thermal expansion
Thermal conductivity
Specific heat
Thermal diffusivity

All samples were prepared and cured at 25 C. Unless otherwise stated, all samples were tested at 25 C after a minimum cure time of 30 days.

^aProduct of Dow Corning, Midland, Michigan

^bProduct of Cabot Corporation

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<i>A-1</i>	

Material Number	Description*	10-minute Viscosity (Pa.s)	Gel Time (minutes)	Comments	"A" Component	"B" Component
1	APC 2.5	2.5	1 - 97 1A - 140 1B - 67	Based on low viscosity lots of Sylgard 184 and Dielectric Gel	Sylgard 184A (Lot 89) - 100 pbw** Dielectric Gel A (Lot 31) - 5 pbw Accelerator 1 - 7 pbw 1A - 4 pbw 1B - 10 pbw	Sylgard 184B (Lot 89) - 10 pbw Dielectric Gel B (Lot 31) - 5 pbw
2	APC 2.5	2.5	2 - 93 2A - 121 2B - 61	Based on high viscosity lots of Sylgard 184 and Dielectric Gel	Sylgard 184A (Lot 36) - 100 pbw Dielectric Gel A (Lot 38) - 5 pbw Accelerator 2 - 8 pbw 2A - 4 pbw 2B - 10 pbw	Sylgard 184B (Lot 36) - 10 pbw Dielectric Gel B (Lot 38) - 5 pbw
3	APC 2.5	3.0	79		Sylgard 184A (Lot 36) - 100 pbw Dielectric Gel A (Lot 31) - 2 pbw	Sylgard 184B (Lot 36) - 10 pbw Dielectric Gel B (Lot 36) - 2 pbw DC-1107 - 1.5 pbw
4	APC 2.5	3.3	72		Sylgard 186A (Lot 47) - 100 pbw Dielectric Gel A (Lot 31) - 50 pbw	Sylgard 186 B (Lot 47) - 10 pbw Dielectric Gel B (Lot 31) - 50 pbw DC-1107 - 1.25 pbw
5	APC 5.0	5.6	91		Sylgard 186A (Lot 64) - 37 pbw Sylgard 184A (Lot 36) - 63 pbw Accelerator - 4 pbw	Sylgard 186B (Lot 64) - 3.7 pbw Sylgard 184B (Lot 36) - 6.3 pbw
6	APC 10.0	9.4	85	Evaluates maximum amount of Sylgard 184 in 184/186 blend	Sylgard 186A (Lot 64) - 52 pbw Sylgard 184A (Lot 89) - 48 pbw Accelerator - 4 pbw	Sylgard 186B (Lot 64) - 5.2 pbw Sylgard 184B (Lot 89) - 4.8 pbw
7	APC 10.0	10.3	88	Evaluates minimum amount of Sylgard 184 in 184/186 blend	Sylgard 186A (Lot 52) - 63 pbw Sylgard 184A (Lot 36) - 37 pbw Accelerator - 4 pbw	Sylgard 186B (Lot 52) - 6.3 pbw Sylgard 184B (Lot 36) - 3.7 pbw
8	APC 10.0	Variable	Variable	Hydrogen bonding of Cabosil causes viscosity decrease and gel time change as material absorbs atmospheric water. Not suitable for production use.	Sylgard 184A (Lot 36) - 100 pbw Accelerator - 3 pbw Cabosil - 6.25 pbw	Sylgard 184B (Lot 36) - 10 pbw

*APC XXX is an identifier assigned to the material by LLNL and represents "Addition Cured Potting Compound" and XXX is the nominal viscosity in Pa.s.
Sylgard is registered trademark of Dow Corning.

**pbw - parts by weight

Material Number	Description*	10-minute Viscosity (Pa.s)	Gel Time (minutes)	Comments	"A" Component	"B" Component
9	APC 10.0	10.3	108		Sylgard 186A (Lot 64) - 53 pbw Sylgard 184A (Lot 36) - 47 pbw	Sylgard 186B (Lot 64) - 5.3 pbw Sylgard 184B (Lot 36) - 4.7 pbw DC-1107 - 0.3 pbw
10	APC 10.0	12.0	97		Sylgard 186A (Lot 47) - 85 pbw Dielectric Gel A (Lot 31) - 15 pbw	Sylgard 186B (Lot 47) - 8.5 pbw Dielectric Gel B (Lot 31) - 15 pbw DC-1107 - 0.34 pbw
11	APC 300	N/A	N/A		Sylgard 186A (Lot 64) - 100 pbw Accelerator - 10 pbw Cabosil MS-75 - 3 pbw	Sylgard 186B (Lot 64) - 10 pbw DC-1107 - 0.15 pbw
12	APC 300	N/A	N/A		Sylgard 184A (Lot 31) - 100 pbw Accelerator - 5 pbw Cabosil MS-75 - 9.653 pbw	Sylgard 184B (Lot 31) - 10 pbw DC-1107 - 1.2 pbw
13	APC 300	N/A	N/A		Dielectric Gel A (Lot 31) - 100 pbw Accelerator - 5 pbw Cabosil MS-75 - 6 pbw	Dielectric Gel B (Lot 31) - 100 pbw DC-1107 - 3 pbw Cabosil MS-75 - 6 pbw
14	Accelerated Sylgard 184	3.9	80		Sylgard 184A (Lot 31) - 100 pbw Accelerator - 5 pbw	Sylgard 184B (Lot 31) - 10 pbw
15	Sylgard 184	3.1	Est. 10	High viscosity lot of Sylgard 184	Sylgard 184A (Lot 31) - 100 pbw	Sylgard 184B (Lot 31) - 10 pbw
16	Sylgard 184	2.0	243	Low viscosity lot of Sylgard 184	Sylgard 184A (Lot 36) - 100 pbw	Sylgard 184B (Lot 36) - 10 pbw
17	Sylgard 186	46.0	N/A	High viscosity lot of Sylgard 186	Sylgard 186A (Lot 47) - 100 pbw	Sylgard 186B (Lot 47) - 10 pbw
18	Sylgard 186	41.0	N/A	Low viscosity lot of Sylgard 186	Sylgard 186A (Lot 64) - 100 pbw	Sylgard 186B (Lot 64) - 10 pbw
19	APC 1.0	N/A	19	Initial viscosity approximately 0.2 Pa.s	Dielectric Gel A (Lot 31) - 50 pbw Accelerator - 10 pbw	Dielectric Gel B (Lot 31) - 50 pbw DC-1107 - 1.03 pbw

*APC XXX is an identifier assigned to the material by LNL and represents "Addition Cured Potting Compound" and XXX is the nominal viscosity in Pa.s.
Sylgard is registered trademark of Dow Corning.

**pbw - parts by weight

Physical Properties

DISC OR PUNCH SHEAR STRENGTH

The punch shear test (similar to ASTM 0732) employed a 1.59 mm thick test sample located between a punch and die. The punch sheared the sample into the die at a rate of 1.27 mm/min during testing.

Material Number	Average (KPa)	Standard Deviation (KPa)	Material Number	Average (KPa)	Standard Deviation (KPa)
1	1100	85	11	2314	135
2	1178	34	12	3057	157
3	1514	53	13	938	43
4	1381	36	14	1782	57
5	1553	37	15	1789	136
6	1774	91	16	1462	22
7	1831	46	17	2292	54
8	2174	62	18	1941	49
9	1602	35	19	-	-
10	1760	44			

NOTE: Sample Size $n = 5$

LAP SHEAR STRENGTH

The lap shear test (ASTM D-1002) employed two aluminum panels (25.4 mm x 101.6 mm) which overlapped 12.7 mm with an adhesive thickness of 0.127 mm. These assemblies were tested in shear at a rate of 1.27 mm/min.

Material Number	Average (KPa)	Standard Deviation (KPa)	Material Number	Average (KPa)	Standard Deviation (KPa)
1	167	16	11	-	-
2	-	-	12	-	-
3	322	72	13	-	-
4	157	26	14	329	51
5	209	8	15	151	18
6	192	20	16	-	-
7	-	-	17	374	65
8	363	66	18	-	-
9	357	93	19	-	-
10	475	92			

NOTE: Sample Size $n = 5$

BUTT TENSILE STRENGTH

The butt tensile test (ASTM D-2094) employed two aluminum cylinders (28.66 mm diameter x 38.1 mm high) butt bonded with an 0.127 mm thick adhesive bond. These samples were then tested to failure in tension at a rate of 1.27 mm/min.

<u>Material Number</u>	<u>Average (KPa)</u>	<u>Standard Deviation (KPa)</u>	<u>Material Number</u>	<u>Average (KPa)</u>	<u>Standard Deviation (KPa)</u>
1	1293	163	11	-	-
2	-	-	12	-	-
3	1666	424	13	-	-
4	1708	189	14	2508	270
5	946	80	15	1329	140
6	971	90	16	3575	-
7	-	-	17	3575	345
8	1923	174	18	-	-
9	3178	451	19	-	-
10	3026	486			

NOTE: Sample Size $n = 5$

COMPRESSIVE STRENGTH

The compressive strength test was performed on cylindrical test specimens 20.27 mm diameter x 25.4 mm high. These were tested to failure in compression at a compression rate of 2.5 mm/min.

<u>Material Number</u>	<u>Average (KPa)</u>	<u>Standard Deviation (KPa)</u>	<u>Material Number</u>	<u>Average (KPa)</u>	<u>Standard Deviation (KPa)</u>
1	23.1	10.3	11	-	-
2	33.3	13.4	12	-	-
3	42.9	5.3	13	-	-
4	73.2	20.4	14	45.7	5.6
5	76.8	7.9	15	58.3	7.5
6	76.5	13.0	16	59.4	6.7
7	200.0	32.9	17	276.0	-
8	7.9	1.5	18	276.0	-
9	104.1	13.7	19	1.0	0.49
10	182.2	27.8			

NOTE: Sample Size $n = 5$

DUROMETER HARDNESS

Durometer hardness was determined via a Shore A2 durometer according to ASTM D-1706. The samples were 25.4 mm x 25.4 mm x 6.35 mm patties.

Material Number	Average		Material Number	Average	
	24 Hours	30 Days		24 Hours	30 Days
1	37.6	49.5	11	31.0	37.0
2	31.2	44.5	12	44.7	54.5
3	31.3	44.0	13	35.5	45.0
4	29.7	38.5	14	33.6	49.0
5	26.9	36.5	15	34.3	46.0
6	29.1	41.0	16	29.7	41.5
7	28.2	39.0	17	24.4	40.0
8	40.6	52.0	18	11.0	35.0
9	26.6	35.0	19	20.9	33.5
10	13.1	36.5			

DYNAMIC SHEAR MODULUS

A Rheometrics 7200 Mechanical Spectrometer was employed for all measurements. The test specimens were 25.4 mm diameter X 5.7 mm high disks clamped under load between two parallel plates with serrated faces. The top plate oscillated clockwise/counterclockwise at a selected frequency while the lower plate (to which the load and torque transducers were attached) remained stationary. For a given sample, the following test parameters could be varied: clamping load, test temperature, oscillation frequency and maximum strain amplitude. Since the shear modulus is essentially independent of strain amplitude as long as strain amplitude is small and the material is in the linear portion of the viscoelastic curve, a small strain amplitude of 0.5% was selected. Since oscillation frequency can be changed quite readily, an automatic scan from 100 mhz to 10 hz was selected. Temperature is very time consuming to vary so three temperatures were selected: -40, 25 and 100 C. Load plays a very important part in determining modulus, i.e., as load increases, so does the modulus. Therefore, load was varied from 100 to 2000 g or 0.204 to 4.074 g/mm². Fig. 1 shows a typical modulus versus clamping load for material No. 15 tested at 1 hz.

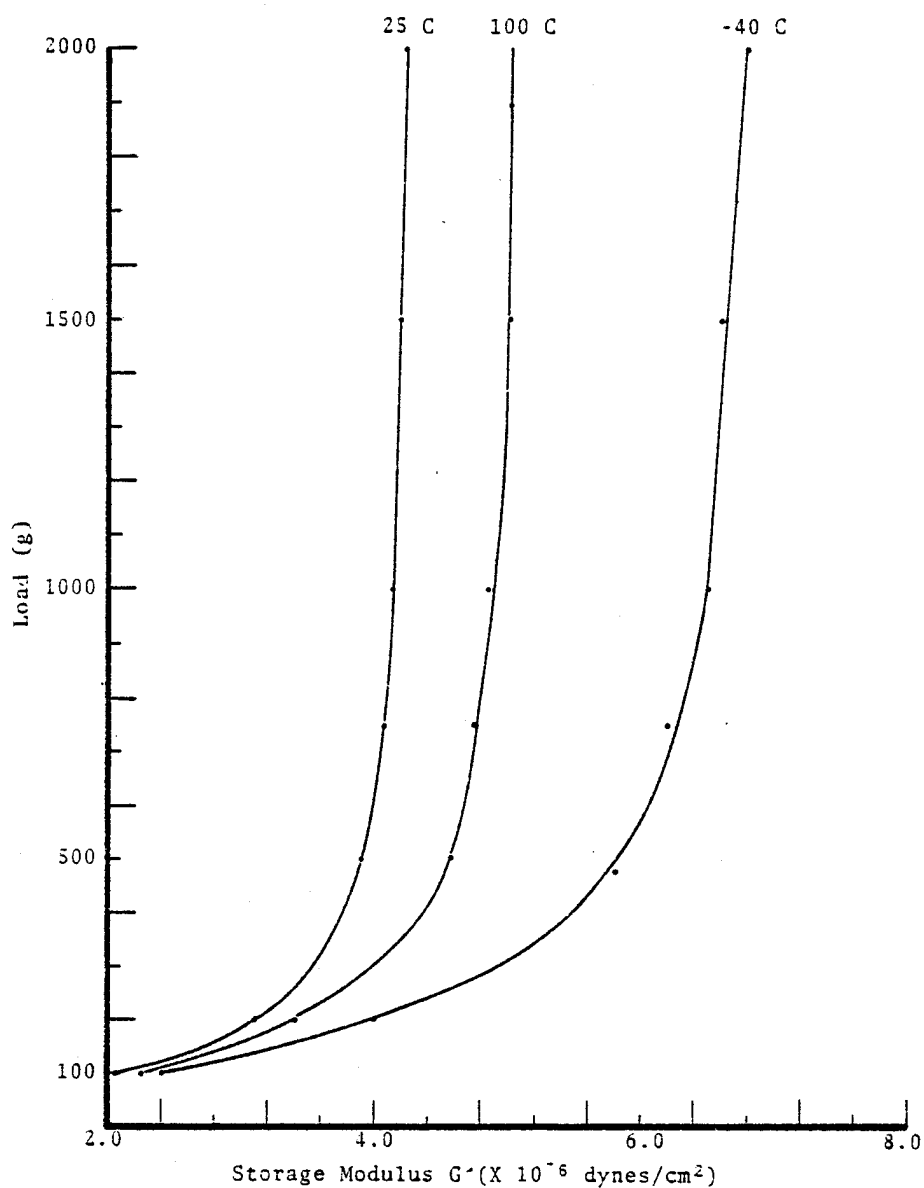


Fig. 1. Storage Modulus (G') Versus Clamping Load for Material No. 15

Modulus normally decreases rapidly above the glass transition point as the temperature increases. Therefore, several questions arose when the 100 C curve consistently had a higher modulus than either 25 or -40 C. This occurred for several materials. A special run was made on material No. 15 where a constant load of 2000 g, strain amplitude of 0.5%, and a

test frequency of 1 hz was used and the temperature was varied from -140 to 100 C. These data, shown in Fig. 2, explain why the storage modulus at 100 C was greater than the storage modulus at either 25 or -40 C.

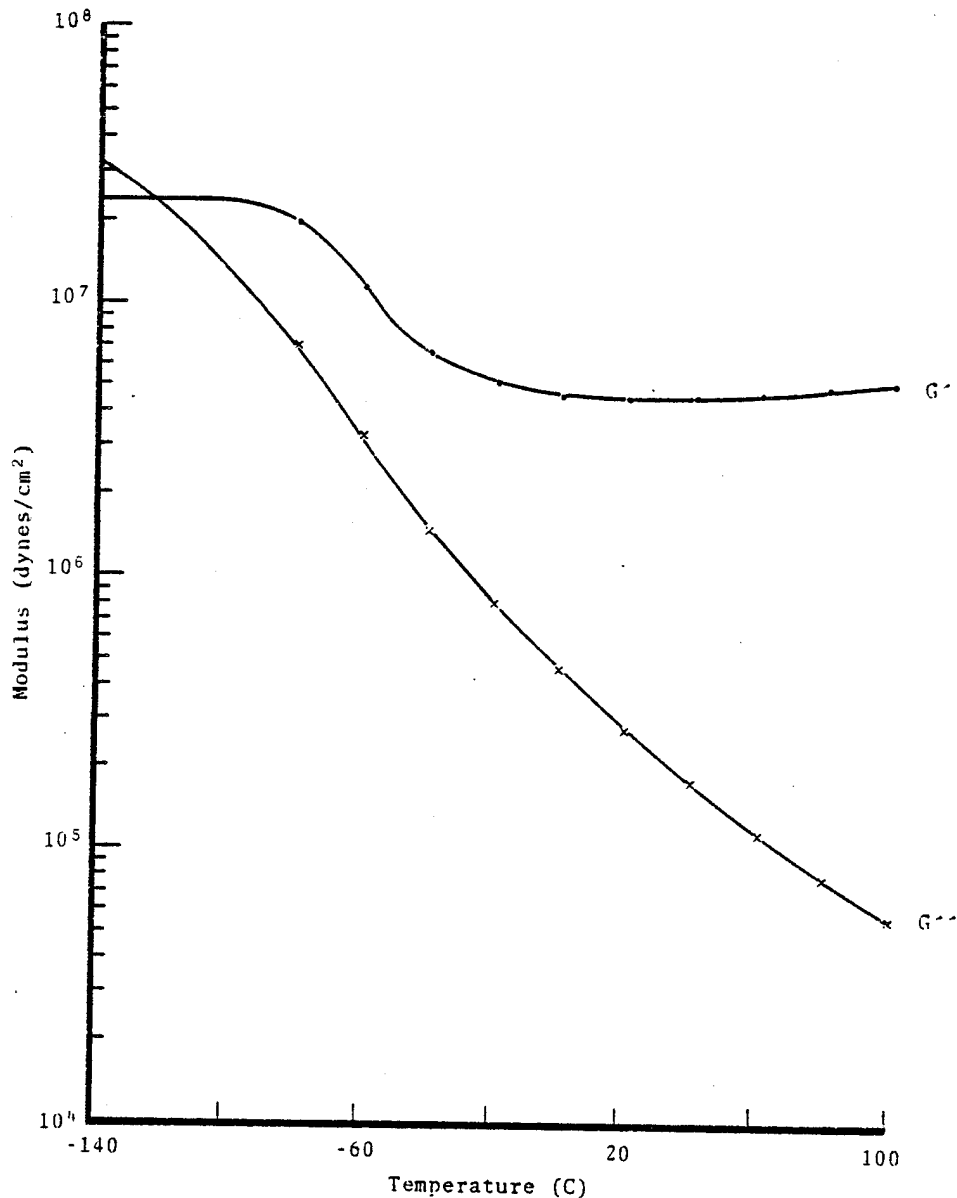


Fig. 2. Modulii Versus Temperature for Material No. 15

It was decided that for a comparison test between samples that the following parameters should be chosen; maximum strain amplitude of 0.5 percent, clamping load of 1000 g or 2.037 g/mm², test frequency of 1 hz

and a test temperature of 25 C. Under these conditions, the following data were generated. Some data were also available at these conditions except at -40 and 100 C.

Material Number	Storage Shear Modulus $G' (X 10^{-6} \text{ dynes/cm}^2)$			Loss Shear Modulus $G'' (X 10^{-4} \text{ dynes/cm}^2)$			Tan $\delta (X 10^2)$		
	-40 C	25 C	100 C	-40 C	25 C	100 C	-40 C	25 C	100 C
1	-	5.07	-	-	14.00	-	-	2.60	-
1A	5.56	5.05	6.10	87.70	16.60	7.17	15.40	3.30	1.18
1B	5.09	5.02	6.20	66.00	13.40	5.46	13.00	2.60	0.88
2	-	3.75	-	-	3.90	-	-	1.00	-
2A	3.12	3.59	4.49	23.40	4.90	2.63	7.49	1.36	0.59
2B	3.34	3.78	4.84	21.30	4.10	2.83	6.39	1.07	0.59
3	-	4.07	-	-	6.30	-	-	1.60	-
4	-	3.35	-	-	3.58	-	-	1.12	-
5	-	3.17	-	-	7.29	-	-	2.31	-
6	3.67	3.80	4.57	31.60	10.50	6.79	8.61	2.88	1.49
7	3.72	3.91	4.02	25.00	9.62	10.90	6.70	2.47	2.70
8	-	5.88	-	-	14.60	-	-	2.48	-
9	-	-	-	-	-	-	-	-	-
10	-	3.89	-	-	7.25	-	-	1.86	-
11	-	3.57	-	-	17.20	-	-	4.82	-
12	-	10.20	-	-	59.40	-	-	5.83	-
13	-	4.91	-	-	5.10	-	-	1.04	-
14	-	5.09	-	-	2.16	-	-	4.25	-
15	6.50	4.35	4.85	144.50	2.26	6.77	22.20	5.21	1.40
16	-	3.22	-	-	6.80	-	-	2.11	-
17	4.40	4.40	4.23	47.70	18.60	12.10	10.80	4.41	2.89
18	-	2.92	-	-	15.10	-	-	5.17	-
19	2.25	2.90	3.55	1.79	0.71	0.68	0.80	0.24	0.19

DENSITY

Density was measured on small samples of materials using a standard pycnometer.

Material Number	Density (g/cm ³)	Material Number	Density (g/cm ³)
1	1.031	11	-
2	1.020	12	-
3	1.026	13	-
4	1.044	14	1.036
5	1.051	15	1.038
6	1.068	16	1.024
7	1.075	17	1.109
8	1.054	18	1.105
9	1.067	19	0.980
10	1.074		

CROSSLINK DENSITY

The molecular weight between crosslinks (M_c) and the effective number of moles of networks crosslink per cubic centimeter (V_c) can be determined by a solvent swell technique employing hexane(8,9).

<u>Material Number</u>	<u>$M_c (M_w/\text{crosslink})$</u>	<u>$V_c(\text{crosslink moles/cm}^3 \times 10^{-4})$</u>
1	1256	7.96
2	1855	5.37
3	1825	5.48
4	4115	2.43
5	3268	3.06
6	2717	3.68
7	4184	2.39
8	1387	7.21
9	3731	2.68
10	4329	2.31
11	-	-
12	-	-
13	-	-
14	1346	7.43
15	1822	5.49
16	2141	4.67
17	4695	2.13
18	5747	1.74
19	4505	2.22

Thermal Properties

COEFFICIENT OF THERMAL EXPANSION {LINEAR}

Linear CTE measurements were made using the 940 Thermalmechanical Analyzer plug-in module for the DuPont 900 Thermal Analyzer. A 4mm diameter quartz expansion probe was used with a 5 g loading. Samples were nominally 6.35 mm diameter right circular cylinders cast from Lexan molds. The exact height of each sample was measured at ambient temperature with a micrometer. Temperature was increased from -80 to 90 C at 5 degrees/minute and the vertical sample expansion recorded. The CTE was calculated by comparing the slope of the sample curve to that of an aluminum standard. The slope was measured over a 10° temperature ranged centered at the reported temperatures. The values for the CTE of aluminum at these temperatures were taken from the American Institute of Physics Handbook, Second Edition. The CTE values reported are the average of at least four determinations with the variation given at $\pm 1\sigma$.

Material Number	Coefficient of Thermal Expansion ($\mu\text{m/m} - \text{Deg C}$)					
	-23 C		27 C		77 C	
	Average	Standard Deviation	Average	Standard Deviation	Average	Standard Deviation
1	314	13	344	13	346	15
2	-	-	-	-	-	-
3	-	-	-	-	-	-
4	-	-	-	-	-	-
5	379	14	325	6	329	2
6	379	16	324	10	325	8
7	-	-	-	-	-	-
8	308	19	316	13	315	15
9	-	-	-	-	-	-
10	383	10	337	15	330	15
11	<i>a</i>	<i>a</i>	377	4	390	16
12	273	16	300	18	318	9
13	380	20	318	1	331	2
14	302	7	301	7	319	2
15	291	18	322	18	353	12
16	-	-	-	-	-	-
17	302	1	289	6	332	15
18	-	-	-	-	-	-
19	347	5	316	10	370	16

NOTE: Sample Size $n = 4$

*a*Phase transition

THERMAL CONDUCTIVITY

Thermal conductivity (K) was determined using a comparative method adapted for the Perkin-Elmer DSC(10,11). The same samples were used for both thermal conductivity and the CTE Measurements. In this method the difference in the heat flow required to maintain a 10° temperature gradient across the sample and a Teflon reference of similar dimensions is measured. Thermal conductivity is calculated using these values, the sample and reference dimensions and the thermal conductivity of the reference. Measurements were made with the DSC set at 45 C and the cold surface temperature maintained at 35 C using a button heater and temperature controller. A thin film of thermal conductive paste (Thermacote Thermal Joint Compounds, Thermallog Co., Dallas, Texas) was used to insure good thermal contact between the ends of the sample and the hot and cold plates.

Material Number	(K) ($\times 10^4$ cal/cm-sec- $^\circ$ C)		Material Number	(K) ($\times 10^4$ cal/cm-sec- $^\circ$ C)	
	Average	Standard Deviation		Average	Standard Deviation
1	5.33	0.01	11	5.32	0.01
2	-	-	12	5.27	-
3	-	-	13	5.74	0.05
4	-	-	14	5.48	0.15
5	5.54	0.07	15	5.55	0.07
6	5.57	0.10	16	-	-
7	-	-	17	5.68	0.09
8	5.52	0.06	18	-	-
9	-	-	19	5.69	0.07
10	5.48	0.05			

NOTE: Sample Size $n = 4$

SPECIFIC HEAT {C_p}

Specific heat or heat capacity (C_p) was determined using a Perkin-Elmer DSC-1 following the standard Perkin-Elmer procedure. Specific heat was determined at both 40 and 50 C.

Material Number	Specific Heat (C _p) (cal/g - deg C)			
	40 C		50 C	
	Average	Standard Deviation	Average	Standard Deviation
1	0.366	-	0.389	-
2	-	-	-	-
3	-	-	-	-
4	-	-	-	-
5	0.324	0.006	0.313	0.002
6	0.330	0.010	0.330	0.010
7	-	-	-	-
8	0.354	0.007	0.363	0.003
9	-	-	-	-
10	0.332	0.004	0.344	0.002
11	0.358	0.004	0.379	0.002
12	0.340	0.0005	0.345	0.003
13	0.370	0.006	0.383	0.007
14	0.327	0.002	0.324	0.003
15	0.388	0.002	0.386	-
16	-	-	-	-
17	0.340	0.010	0.350	0.020
18	-	-	-	-
19	0.480	0.020	0.491	0.004

NOTE: Sample Size n = 4

THERMAL DIFFUSIVITY

Thermal diffusivity (α) is calculated based on the following relationship:

$$\alpha = K / \delta C_p$$

where

K = Thermal Conductivity

δ = Density

C_p = Heat Capacity or Specific Heat

<u>Material Number</u>	<u>Thermal Diffusivity (α) (X 10^3 cm²/sec) 45 C</u>	<u>Material Number</u>	<u>Thermal Diffusivity (α) (X 10^3 cm²/sec) 45 C</u>
1	1.369	11	-
2	-	12	-
3	-	13	-
4	-	14	1.625
5	1.655	15	1.382
6	1.580	16	-
7	-	17	1.414
8	1.461	18	-
9	-	19	1.196
10	1.510		

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