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Low-Temperature Differential Scanning Calorimetry of Polysiloxanes

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Interim Report

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20. ABSTRACT (*Continue on reverse side if necessary and identify by block number*)

Low-temperature differential scanning calorimetry (DSC) was used to determine the temperature region of glass formation or the crystallization of polysiloxanes or both. Research- and commercial-grade polysiloxanes were characterized by DSC to determine where significant physical property changes, such as embrittlement or shrinkage, had occurred. The effect of the thermal history, cooling-heating rate, and molecular weight distribution on thermodynamic behavior was investigated. An illustrative example of the failure analysis of a low-temperature adhesive used on a solar panel is included.

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CONTENTS

FIGURES

TABLES

I. INTRODUCTION

Polysiloxane polymers, both linear and branched, have been increasingly used in aerospace applications. These unique properties of high-temperature stability, low variation of physical properties with both low and high temperature, and environmental stability are the reasons for this growth in popularity in the aerospace industry. Many polymeric systems have been synthesized with the polysiloxane structure for the main chain

$$(0 - \mathrm{Si})_n$$

These systems may incorporate various types of substituents, branches, and atoms of other elements whose presence gives the broad spectrum of properties in the product polymer.[1, 2] The polydimethylsiloxane rubbers and polymers based on this structure are known to crystallize readily at low temperatures.[3] The crystallization is so facile that even brief immersion in liquid nitrogen produces thermomechanical curves indicative of a crystalline phase.[4] Helmer and Polmanteer observed crystalline behavior with the use of differential thermal analysis at cooling rates of 52°C/sec.[5] These effects are representative of high chain mobility and very low cohesive energy.

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[1] K. A. Andrianov, Vspekhi. Khim. 26, 895, 1957.

[2] K. A. Andrianov, Vspekhi. Khim. 27, 1257, 1958.

[3] K. A. Andrianov, G. Ye. Golubkov, and V. I. Yelinek, Polymer Sci. USSR 4, 760, 1964.

[4] G. L. Slonimskii and V. Yu. Levin, Vysokomol. Soedin. 11, 1936 (1966).

[5] J. D. Helmer and K. E. Polmanteer, J. Appl. Polymer Sci. 43, 2113 (1969).

Studies of the crystallization rate of polydimethylsiloxanes indicate that low-molecular-weight (mol wt 10,000) polydimethylsiloxane crystallizes much more slowly than the high-molecular-weight variety (mol wt 400,000).[4] High-molecular-weight polydimethylsiloxane rubber crystallizes at an appreciable rate at temperatures slightly below the melting point (~-46°C), whereas at approximately -60°C, this rate is very high, and, in fact, the crystallization process is half complete after only 2.5 min.

The calorimetric measurement of polydimethylsiloxane indicates that two melting peaks are present at approximately -38°C. The relative magnitude of the peaks depends on the rate of cooling.[5] The two melting peaks are the result of the different crystalline phases, an acicular and a spherulitic modification. The crystallization kinetic data of Warrick[6] on unstretched and stretched polydimethylsiloxane filled elastomers indicate that spherulitic growth predominated for the unstretched sample and acicular growth predominated for the stretched sample. The data indicate further that the rate of crystal growth of the acicular crystals was considerably greater than for spherulitic growth and that crystallization took place at a higher temperature. Lee et al.[7] found that the melting temperature increased as the cooling rate decreased. The slower cooling rate produced crystals that are more nearly perfect.

Crystallization of polydimethylsiloxane starts at -60°C and reaches a maximum at -80°C under isothermal conditions.[8] The highest degree of crystallization on a polydimethylsiloxane polymer of mol wt 25,000 was 70%.

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[6] E. L. Warrick, J. Polymer Sci. 27, 19 (1958).

[7] C. L. Lee, O. K. Johannson, O. L. Flaningan, and P. Hahn, Polymer Preprints 10, 1311 (1969).

[8] M. Sh. Yagfarov, Z. G. Gizatullina, and V. S. Ionkin, Vysokomol. Soedin. 11, 815 (1969).

The contributions of each peak in the double melting peak of polydimethylsiloxane to the overall degree of crystallization was reported by Yagfarov et al.[8] The concentration of the crystalline formation with a high melting temperature noticeably increases with decreasing thermostatting temperature and reaches a maximum of 58% at -105°C. The concentration of crystalline formations with a lower melting temperature decreases with decreasing thermostatting temperature, and it disappears completely at -110°C. It appears that acicular crystal concentration increases with decreasing thermostatting.

If approximately 5 to 15% of the methyl groups of dimethylpolysiloxanes are replaced by phenyl groups, a polysiloxane polymer with exceptionally good low-temperature mechanical properties is obtained. The improved properties are the result of the effect of the randomly distributed and relatively bulky phenyl groups in the molecular chain. These phenyl groups reduce intermolecular forces and retard the tendency of the polymer to crystallize. There is a limit to the improvement that can be obtained in this manner inasmuch as increasing the phenyl-methyl ratio causes a reduction in molecular flexibility, and the high-phenyl high-molecular-weight polysiloxanes approach the solid crystalline state. In fact, the completely phenyl-substituted system (that with no methyl substituents) has a melting point of +367°C.[9]

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[9] D. J. Fischer, J. Polymer Sci. 5, 436 (1961).

II. EXPERIMENTAL

A. MATERIALS

Thermal studies were performed on secondary standards of polydimethylsiloxane and commercial materials. Since commercial siloxanes contain unknown amounts of proprietary additives and cross-linking agents, the initial investigation was focused on three secondary standards of uncrosslinked polydimethylsiloxane (Table 1). The secondary standards were obtained from Aldrich Chemical Company. The weight average $\overline{M}_w$ and number average $\overline{M}_n$ were determined by light-scattering and gel-permeation chromatographic measurements, respectively. The relatively large polydispersity of these samples is representative of commercial-grade siloxanes.

The commercial samples that were investigated are listed in Table 2. They were formulated according to the manufacturer's recommendations.

Table 1. Polydimethylsiloxanes

Sample	Weight Average $\overline{M}_w$, mol wt	Number Average $\overline{M}_n$, mol wt	Polydispersity Ratio $\overline{M}_w/\overline{M}_n$
PDS-01	77,400	30,800	2.51
PDS-02	166,000	47,000	3.52
PDS-03	609,000	150,000	4.05

Table 2. Commercial Silicone Products

Sample	Manufacturer	No. of Components	Color	Generic Type
RTV 732	Dow-Corning	1	Transparent	Dimethyl
RTV 3145	Dow-Corning	1	Gray	Dimethyl
DC 93-072	Dow-Corning	2	White	Methyl-vinyl-phenyl
DC 93-500	Dow-Corning	2	Light straw	Methyl-vinyl-phenyl
DC 6-1104	Dow-Corning	1	Translucent white	Dimethyl
Silastic E	Dow-Corning	2	White	Dimethyl
Sylgard 182	Dow-Corning	2	Transparent	Dimethyl
RTV-118	General Electric	1	Transperent	Dimethyl
RTV-560	General Electric	2	Red	Dimethyl
RTV-566	General Electric	2	Red	Dimethyl
RTV-567	General Electric	2	Transperent	Dimethyl
RTV-630	General Electric	2	Blue	Dimethyl

B. CARD DIFFERENTIAL SCANNING CALORIMETRY

The calorimeter studies were performed with a Perkin-Elmer Model DSC-2 calorimeter equipped with subambient accessories (Fig. 1). Liquid nitrogen was used as the coolant. Dry helium was purged through the calorimeter chamber at a rate of 20 cm^3/min. The glove-box area above the chamber was purged with dry nitrogen at a rate of 20 cm^3/min. The relative humidity of the chamber was maintained at 3% during the determination. The sample size for this investigation was 9 to 12 mg.

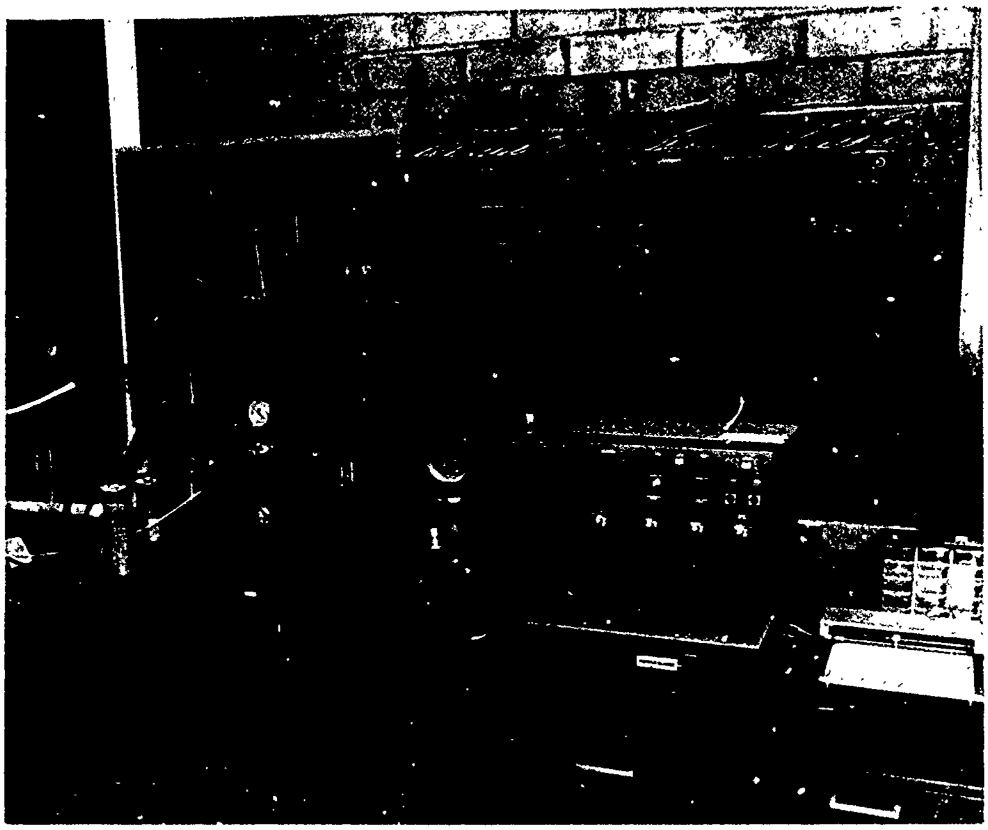

Fig. 1. Perkin-Elmer Differential Scanning Calorimeter Model-2

III. RESULTS

The DSC thermograms of the three standards are shown in Fig. 2. The specimens were cooled at a rate of 10°C/min to -180°C, equilibrated for 5 min, and heated to room temperature at 10°C/min. Each thermogram was recorded during the heating cycle and represents the amount of energy required to increase the temperature of the material at a constant rate. The endothermic peak recorded at -120°C is designated the glass transition peak and represents the energy required to "melt" the polysiloxane glass. The cold crystallization or supercooling region depicted by the exothermic peak at -80°C in Fig. 2 results from the crystallization that occurs when the specimen is heated and then rapidly cooled. As heating continues, the crystalline regions of the system melt, which yields the endothermic peaks at about -40°C. These two peaks are indicative of the two distinct crystalline regions:[6] the peak at -45°C is attributed to strain-induced acicular crystallization, and the peak at -35°C is attributed to bulk spherulitic crystallization. The effect of the molecular weight and the cooling rate is clearly evident in the PDS-03 specimen. At the standard cooling rate, the system would not crystallize into the two distinct crystal modifications. A slower cooling rate for PDS-03 yields a thermogram similar to that shown for PDS-01.

The thermal response of the polydimethylsiloxanes can be demonstrated by repetitive DSC scans with the use of the temperature program previously described. A 10-mg sample of PDS-02 yielded the DSC thermogram shown in Fig. 3. The samples were permitted to equilibrate at room temperature between runs. The spherulitic crystal content appears to increase with each scan. This phenomenon is in conflict with polymer literature in which it is reported that the thermal history is destroyed once the crystal is melted. Theoretically, each scan should have produced identical DSC thermograms. The "memory effect" could be destroyed by heating to 100°C at 10°C/min.

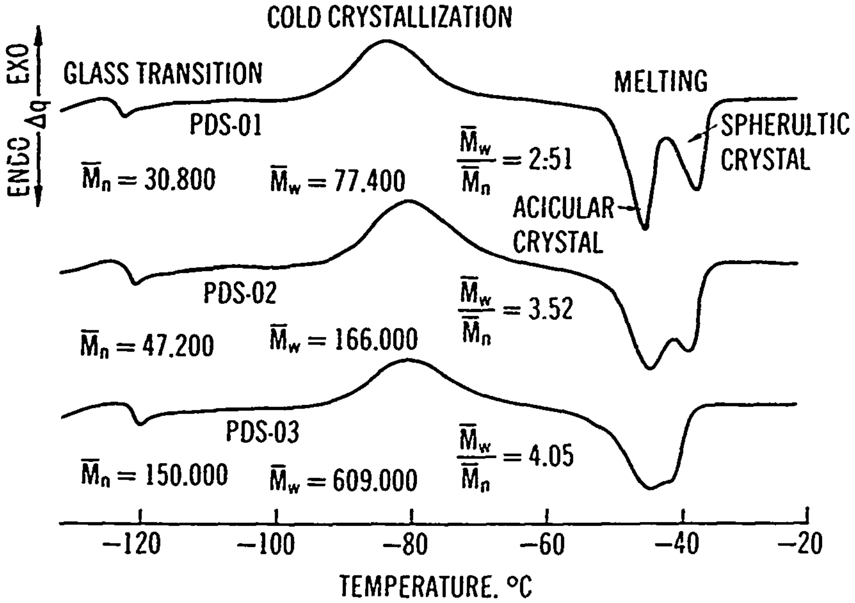

Fig. 2. Effect of Molecular Weight Distribution on Thermodynamic Behavior of Polydimethylsiloxane

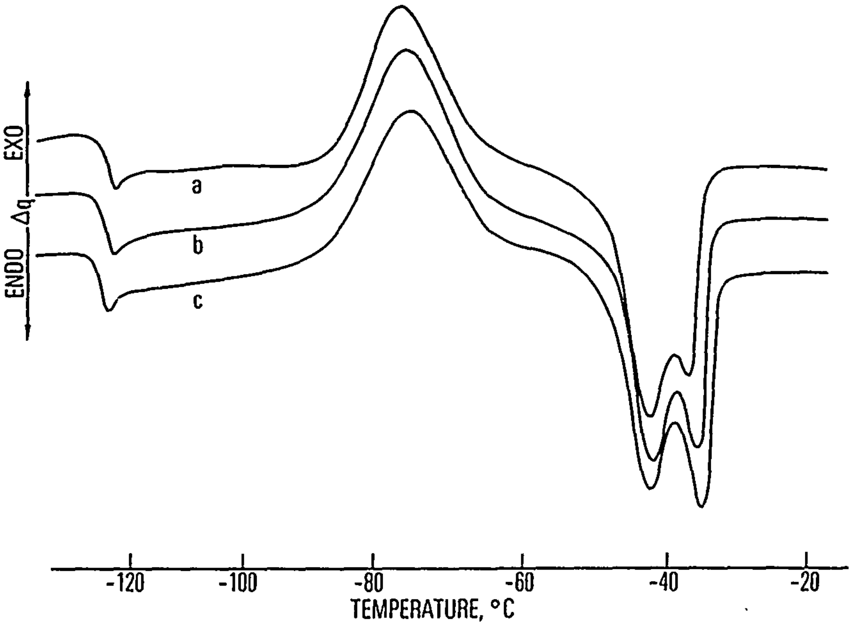

Fig. 3. Effect of Thermal History on Polyelimethylsiloxane a. PDS-02, initial run. b. PDS-02, second run at 21.5 hr. c. PDS-02, third run at 71.5 hr.

Helmer and Polmanteer[5] report that a cooling rate of at least 96°C/min was necessary to observe cold crystallization or supercooling of PDS when subsequently heated at 10°C/min. Their PDS had an average molecular weight of 450,000 and was free of a polymerization catalyst. Lee et al.[7] have shown that a cooling rate of at least 4.2°C/min is necessary to observe cold crystallization on PDS samples with a polydisperity ratio $\bar{M}_w/\bar{M}_n$ of 1.1/1.3. In this investigation, supercooling or a cold crystallization was observed for PDS-01, -02, and -03 when cooled at 10°C/min, soaked at liquid nitrogen temperatures for 5 min, and subsequently heated at 10°C/min. Evidently, the polydispersity ratio is involved in the crystallinity behavior. A large polydispersity ratio means a broader distribution of molecular sizes, which results in low crystallizability because of the steric and "ponderal effects"[10] on chain alignment for crystallization. The system can still achieve local crystallization of its nearest neighbor atom without chain movement in the amorphous state, i.e., cold crystallization. A large distribution of molecular weights could facilitate local crystallization because of the greater number of sites, which would explain the facile cold crystallization effect observed with PDS-01, -02, and -03.

The influence of thermal history on the thermodynamic behavior of a secondary standard of polydimethylsiloxane is shown in Fig. 4. A sample of PDS-01 was programmed (cooled) from 30 to -170°C at 10°C/min (Fig. 4a). During cool-down, a single transition (glass transition) was observed at approximately -126°C. The sample was immediately heated to -25°C at 10°C/min (Fig. 4b). A glass transition was again observed at approximately -126°C, followed by an exothermic transition (cold crystallization) at approximately -80°C. Most of the crystalline melting occurs at from -50 to -40°C. Two major crystal modifications melt in this region: a spherulitic crystal at approximately -49°C and an acicular crystal at approximately -43°C. The smaller endothermic melting areas are typical of the polydimethylsiloxane

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[10] De la Mare, Fowden, Hughes, Ingold, and Mackie, J. Chem. Soc. 3196 (1955).

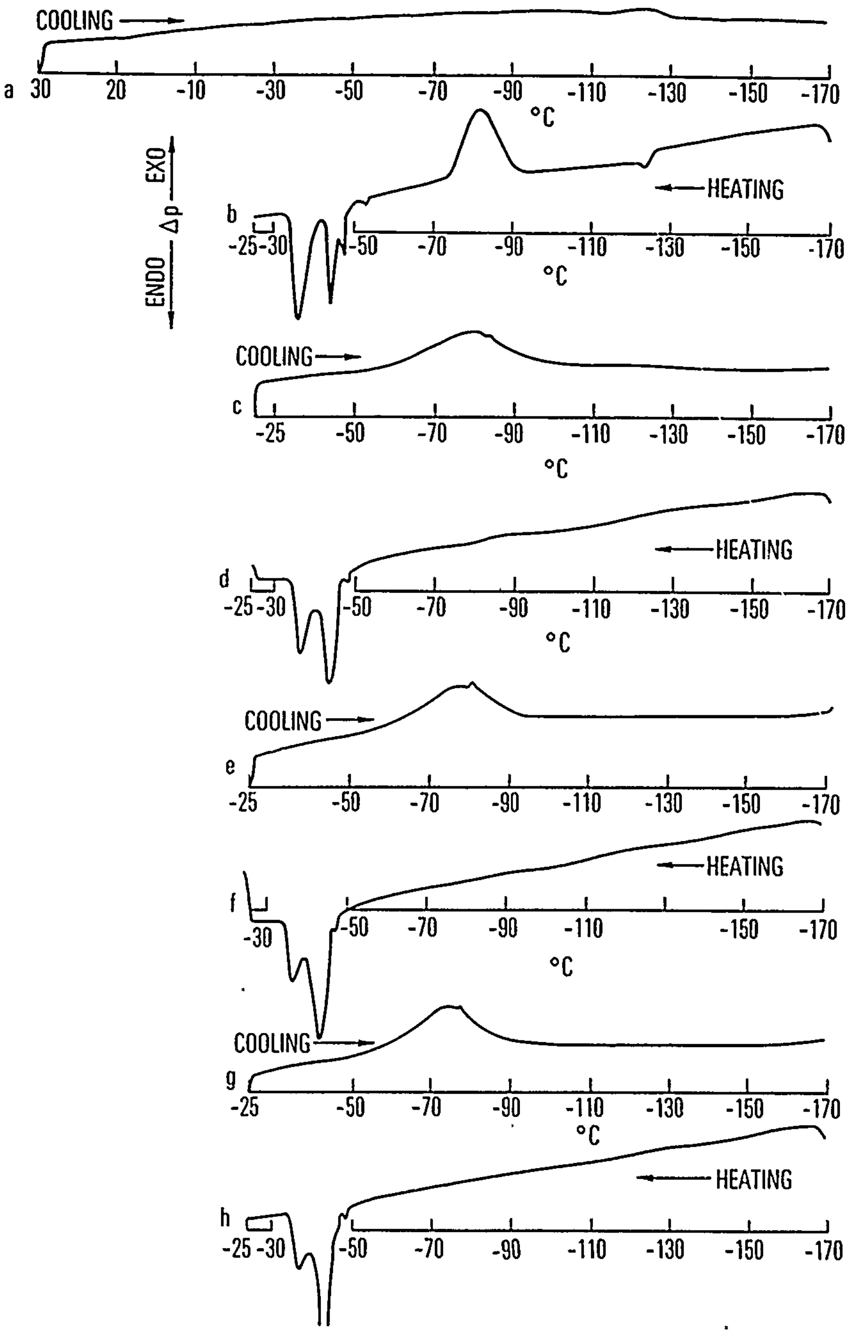

Fig. 4. Effect of Thermal History on Thermodynamic Behvaior of PDMS-01. a. 30 to 70° C at 10°C/min. b. -25° C. c. -80° C. d. -170 to +25° C. e, f, g, and h. -170° C

system and have not been assigned to a crystalline order. Immediately cooling the sample from -26 to -170°C produced a single exothermic or freezing peak at approximately -80°C (Fig. 4c). The occurrence of this exothermic peak in Fig. 4c and its absence in Fig. 4d indicate a structural ordering, which was maintained when the temperature did not exceed -26°C. This type of behavior could easily be missed in a single adhesive qualification evaluation if it were cycled between room-temperature and liquid-nitrogen temperature. However, this behavior may have serious mechanical performance ramifications in a satellite application where the temperature could be maintained within the low-temperature limits, which caused the exothermic peak shown in Fig. 4c. Focal planes and infrared sensors on satellites are typically maintained at low temperatures. Heating the sample from -170 to -25°C (Fig. 4d) did not reveal any cold crystallization. The absence of the cold crystallization peak at approximately -80°C in Fig. 4d during heating confirms the existence of some molecular ordering. This ordering restricts the local orientation of atoms on adjacent chains, which normally causes cold crystallization. The melting region shown in Fig. 4d indicates that there is an increase in the acicular crystal modification content (43°C). Continued cycling between -25 and -170°C (Figs. 4e, 4f, 4g, and 4h) did not reveal much change in the cooling thermograms (Figs. 4e and 4g). During the heating cycles (Figs. 3f and 3h); however, a continued growth of the acicular crystal modification is evident. This modification is a strain-induced crystallization. Apparently, the continued melting and freezing within the low temperature limits are increasing the strain within the polymer structure. This feature is of significant importance in the evaluation of commercial adhesives that exhibit the two crystal modifications.

The results of a crystallization study of the three secondary standards of polydimethylsiloxane are given in Table 3. The average peak areas can be used to calculate the amount of crystallization that occurs during cooling. The results of this calculation versus the polydispersity ratio are shown in Fig. 5.

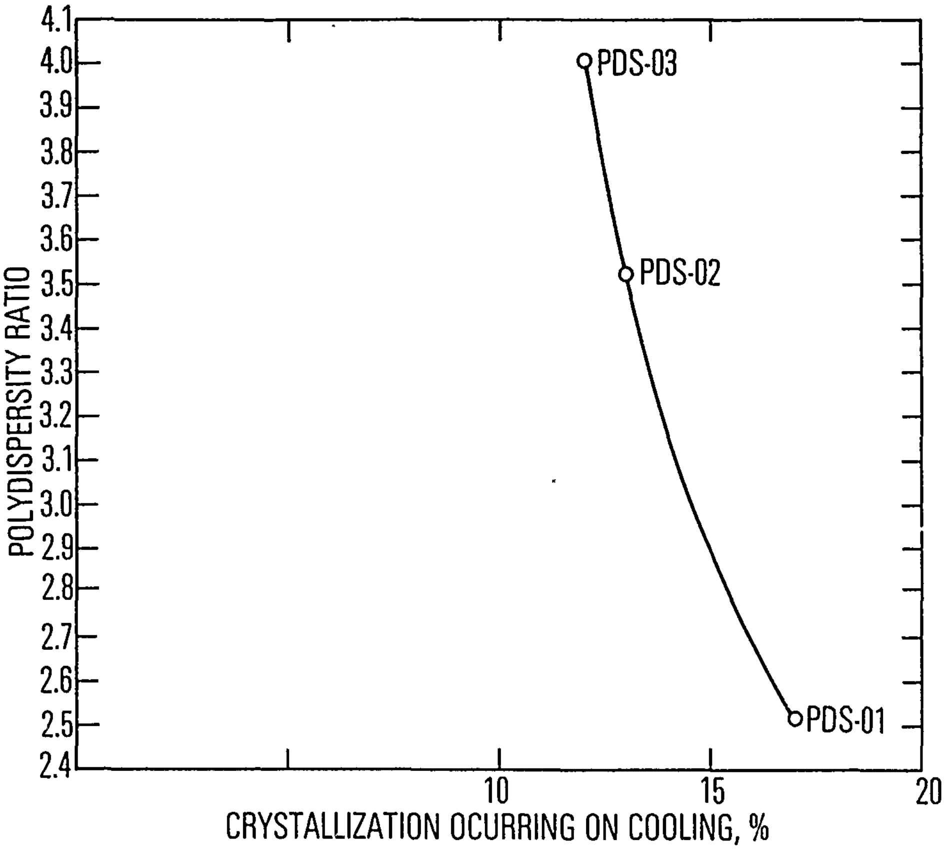

Fig. 5. Crystallization Behavior

Table 3. DSC Results for Polydimethylsiloxane with Cooling-Heating Rate of 10°C/min

Sample	Trial	Area of Supercoupled Peak, in.^{2}	Area of Melting Peak, in.^{2}	Glass Transition Temperature Tg, °C
PDS-01	1	0.953	1.006	-126
	2	0.993	1.023	-126
	3	0.900	1.030	-125
PDS-02	1	0.913	9.900	-127
	2	0.836	0.913	-126
PDS-03	1	0.160	0.200	-126
	2	0.210	0.196	-126

In the PDS-01 system, 17% crystallization occurs during cooling, the result of the greater mobility of the shorter chain of the polymer, i.e., the lower polydispersity ratio.

The effect of varying the heating rate on PDS-01 is demonstrated in Fig. 6. Each specimen was cooled at a rate of 25°C/min to -190°C and subsequently heated at rates of 5, 15, 30, and 60°C and, again, at 5°C/min (Figs. 6a through 6e). The effect of heating rate apparently has little effect on the melting temperature of the glass, i.e., -127°C. The exothermic peak, cold crystallization or supercooling, at -80°C results from crystalline growth during the heating of the specimen. Apparently, little crystallization occurs in PDS-01 when cooled at 25°C/min, as evident in all five thermograms, because (1) the endothermic heat of melting at approximately -40°C is approximately equal to the exothermic peak of formation that occurs at -80°C and (2) when heating the specimen at 60°C/min, there is insufficient time for cold crystallization to occur, and, consequently, very little melting of a crystalline form can be measured at -40°C.

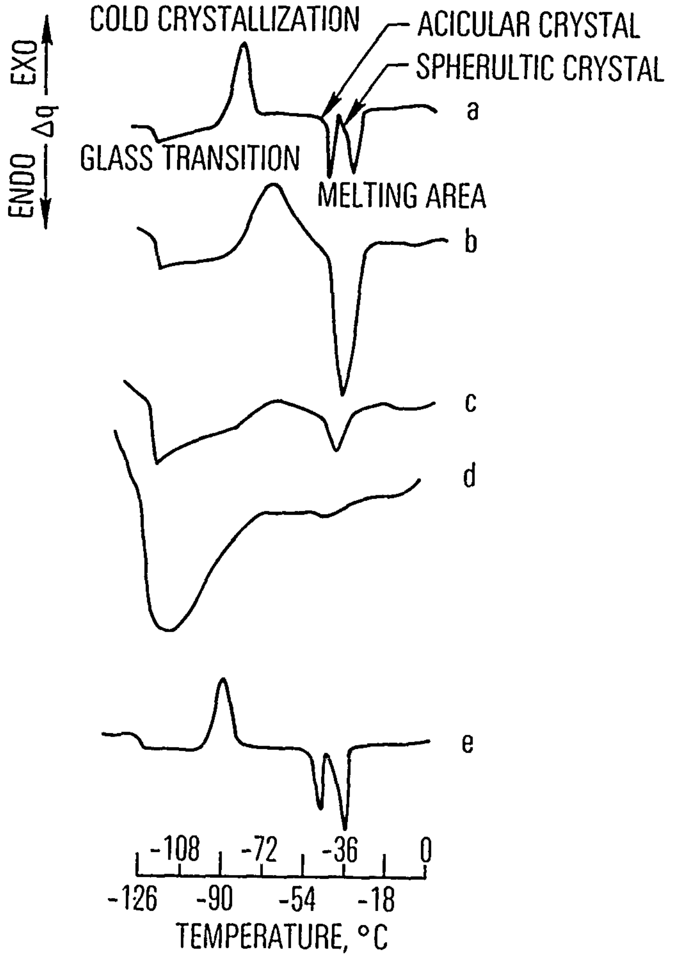

Fig. 6. Effect of Heating Rate on Thermodynamic Behavior Polydimethylsiloxane PDS-01.
a. 5°C/min. b. 15°C/min. c. 30°C/min.
d. 60°C/min. e. 5°C/min.

The diverse behavior of commercial polysiloxanes is shown in Fig. 7. The six DSC thermograms were recorded during heating at 10°C/min; all specimens were previously cooled at 20°C/min to liquid nitrogen temperature and soaked for 5 min. The absence of crystallization, which results in a more pronounced glass transition, is indicated by the Dow-Corning DC 182 and the General Electric GE 567 specimens. The remaining materials, GE 630, DC 6-1104, DC 118, and DC 732, all crystallized at approximately -40°C; the exothermic peak for DC 6-1104 and DC 118 at -85°C is the result of supercooling. Additional experiments indicated that cooling DC 6-1104 and DC 118 at a slightly lower rate, e.g., 10°C/min, removes the supercooling peaks shown in Fig. 7. Similarly, GE 630 and DC 732 display supercooling characteristics if a slightly higher cooling rate is used. Thus, the extent of crystallization is highly dependent on the cooling rate. No crystallization occurred in the DC 182 and GE 567 (and GE 560, not shown) materials at the cooling rates used. The absence of crystallization probably results from the substitution of limited amounts of phenyl and vinyl substituents for the methyl groups. Such substitutions affect the molecular symmetry of the molecule and inhibit the molecular packing necessary for crystallization.

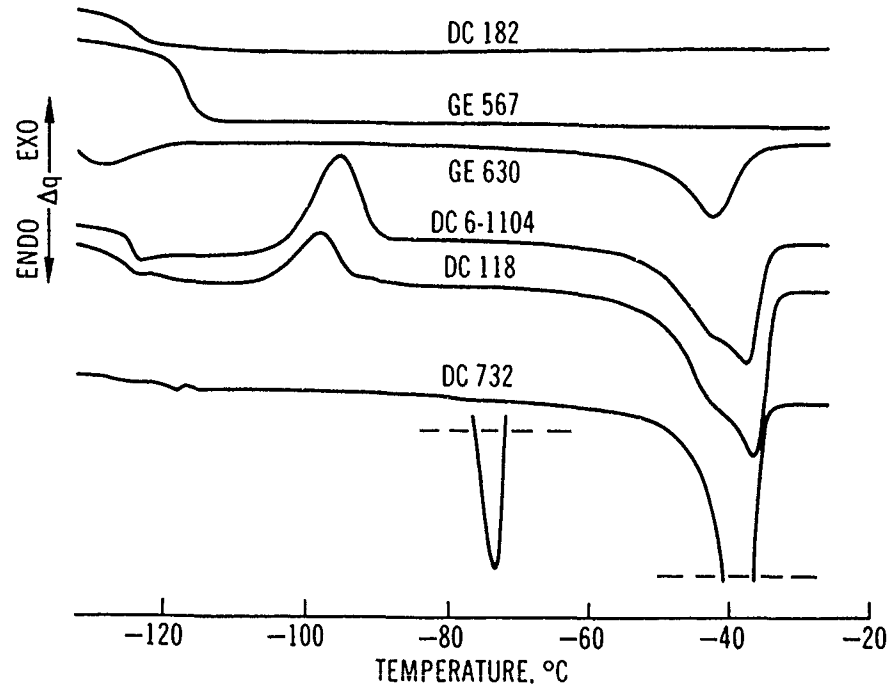

Fig. 7. Effect of 20° C/min Cooling and 10°C/min Heating Rate on Commercial Polysiloxanes

IV. APPLICATIONS

An example of the application of these thermal measurements to actual satellite problems is the recent failure of a diode on a GPS solar panel. Silicone diodes attached to the solar panels with Dow Corning DC 6-1104 had routinely survived quench-cooling to -195°C during thermal-vacuum testing. However, it was discovered that, when a cooling rate of 10 to 15° C/min was applied, diode cracking (Fig. 8) occurred.

This phenomenom was explained by the thermal behavior of DC 6-1104 (Appendix). When cooled rapidly, DC 6-1104 does not crystallize and consequently remains pliant to -123°C, its glass-transition temperature. Thus, during quench-cooling, the DC 6-1104 exhibits rigid behavior only below -123°C; apparently, the diode was capable of withstanding the thermal stresses developed in the 72°C temperature excursion below -123°C. However, slower cooling of the order of 10 to 15°C/min permitted sufficient time for the molecular rearrangements necessary for substantial crystallization to occur in the region of -60°C. Complementary mechanical measurements on similar siloxane materials indicate that partial crystallization of these materials is accompanied by a substantial increase in modulus, or rigidity, as would be expected. In addition, the more efficient molecular packing of the crystalline regions results in a bulk shrinkage of the material. Both phenomena, higher modulus and bulk shrinkage, cause higher stresses in the diode when crystallization occurs. It was concluded that the additional thermal stresses occurring in the diode as a result of crystallization of the DC 6-1104 caused the diode to fail. When a noncrystallizing silicone encapsulant (GE 560) was substituted, no further diode cracking occurred. Government-Industry Data Exchange Program Alert F1-A-76-08 was issued.

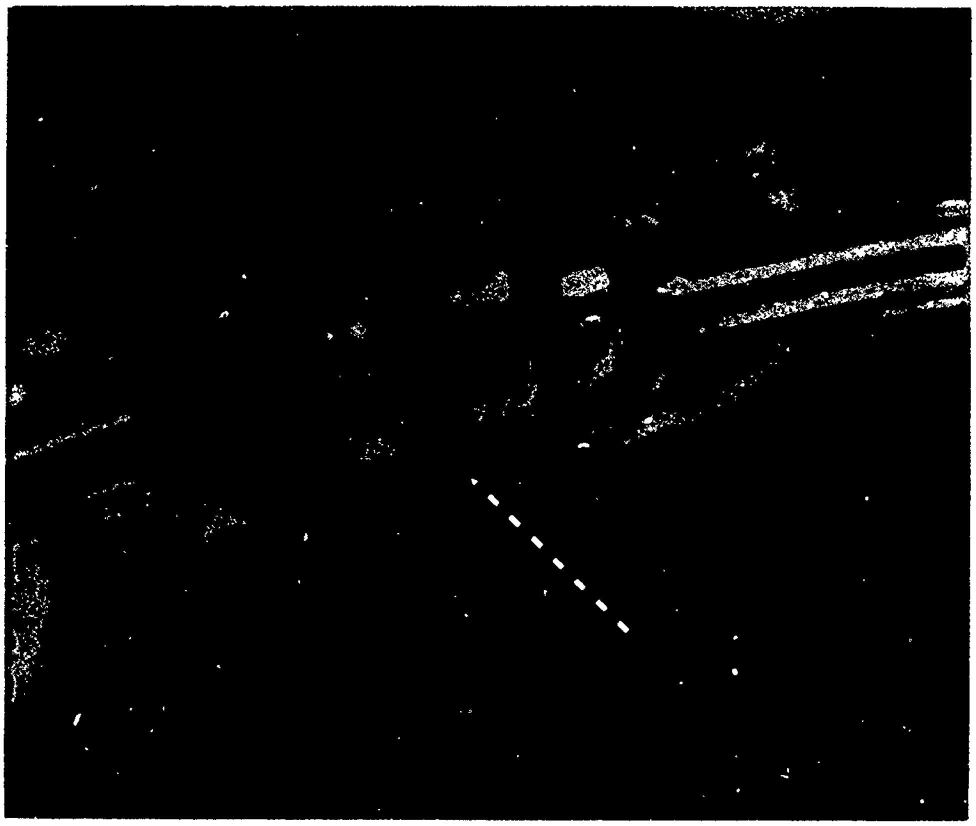

Fig. 8. Crack in Unitrode IN551 Silicon Diode Caused by Thermal Stresses of Silicone Adhesive That Crystallized at Low Temperature. (Crack has been opened for illustrative purposes.)

APPENDIX

DIFFERENTIAL SCANNING CALORIMETRY THERMOGRAMS

Sample	Figure
RTV 732	A-1
RTV 3145	A-2
DC 93-072	A-3
DC 93-500	A-4
DC 6-1104	A-5
Silastic E	A-6
Sylgard 182	A-7
RTV-118	A-8
RTV-511	A-9
RTV-560	A-10
RTV-566	A-11
RTV-567	A-12
RTV-630	A-13

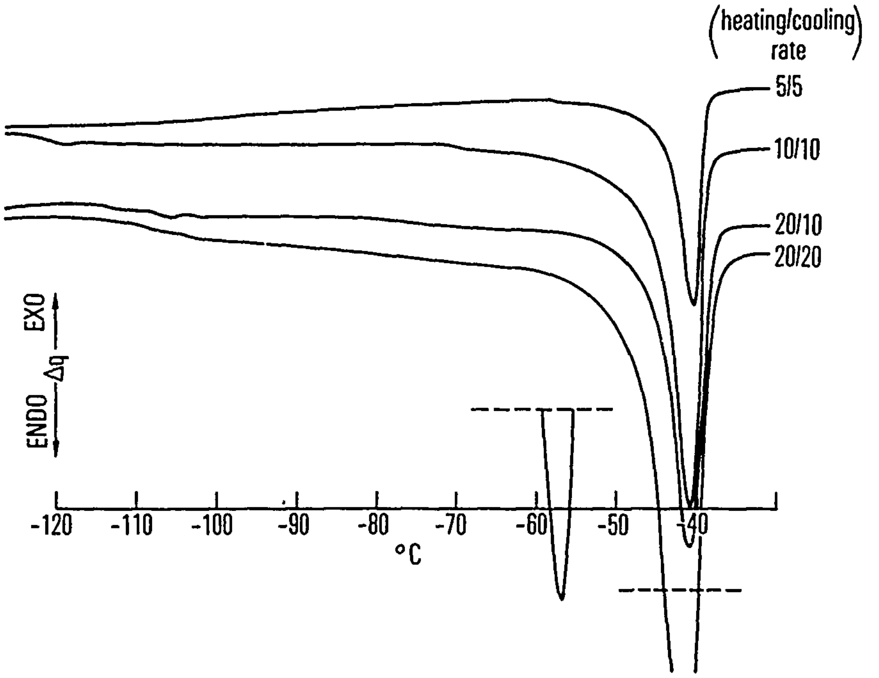

Fig. A-1. RN 732

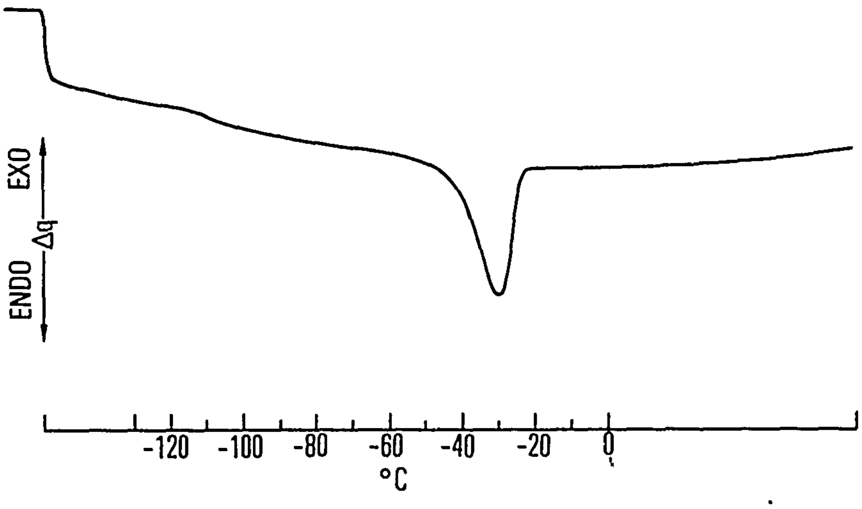

Fig. A-2. RTV 3145. Heating rate = 20° C/min.

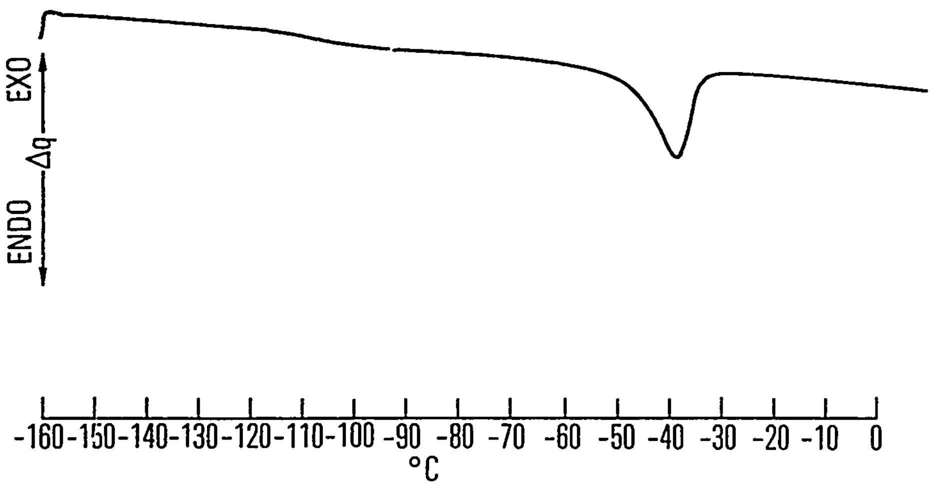

Fig. A-3. DC 93-072. Heating rate = 10° C/min.

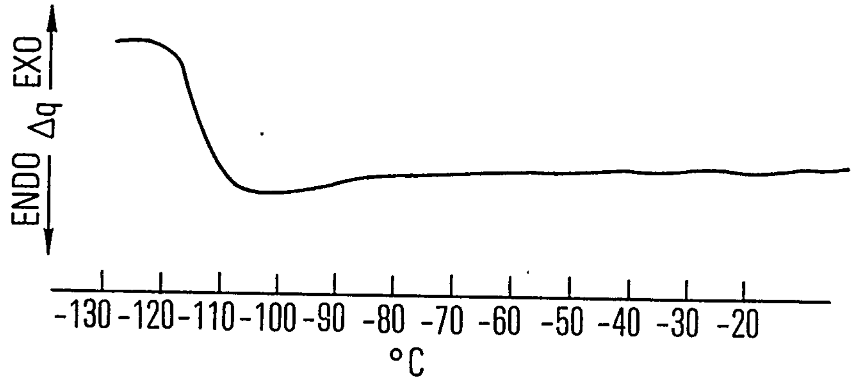

Fig. A-4. DC 93-500. Heating rate = 10° C/min.

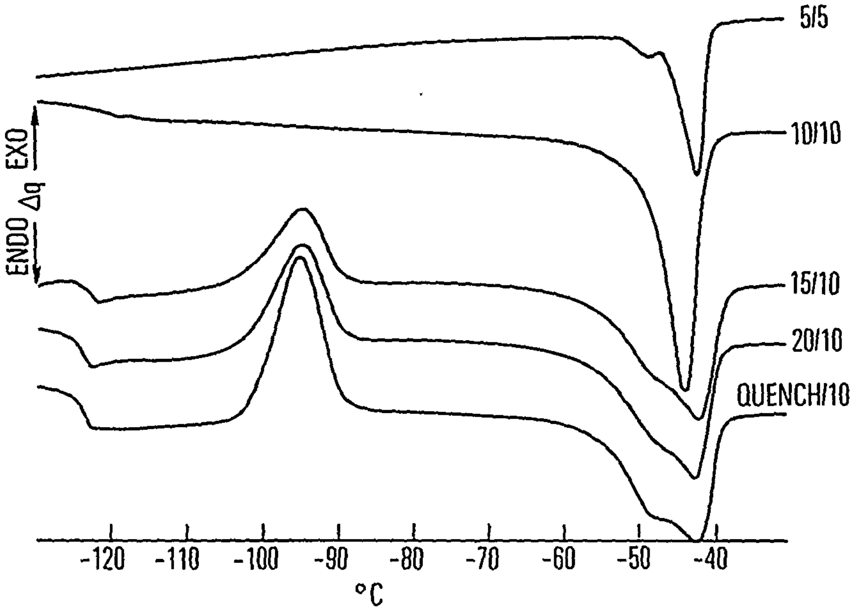

Fig. A-5. DC 6-1104

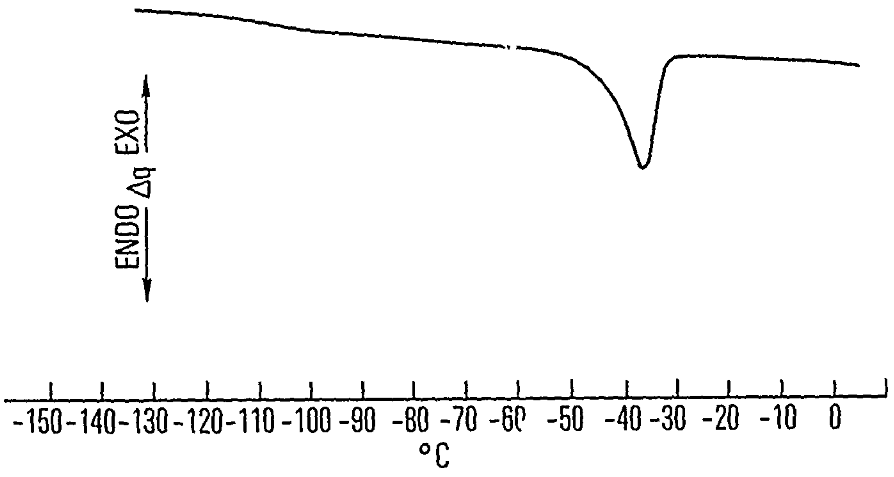

Fig. A-6. Silastic E. Heating rate = 10°C/min.

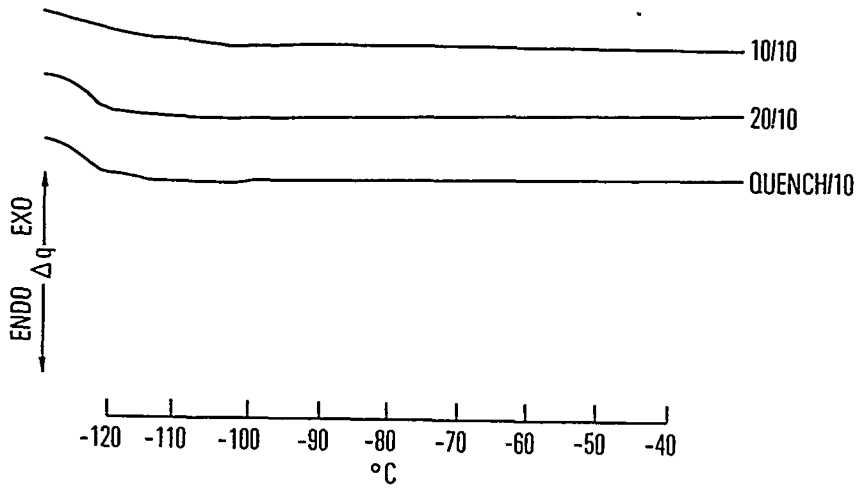

Fig. A-7. Sylgard 182

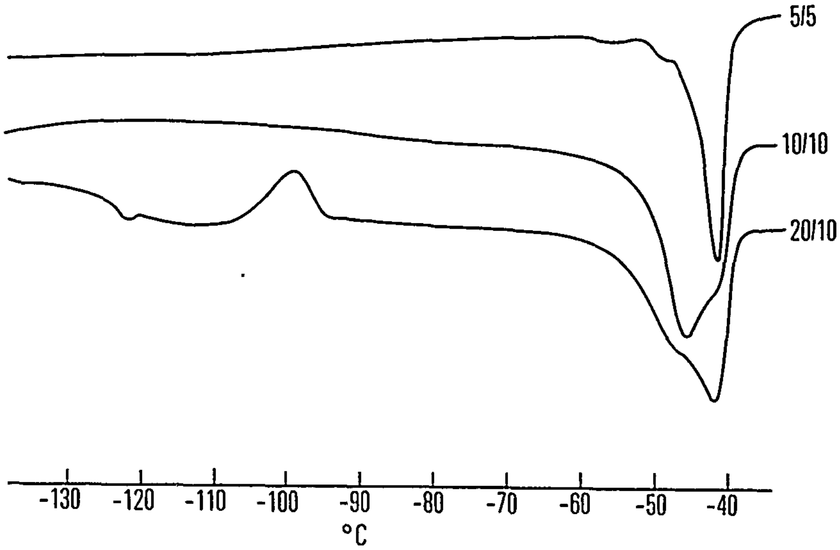

Fig. A-8. RTV-118

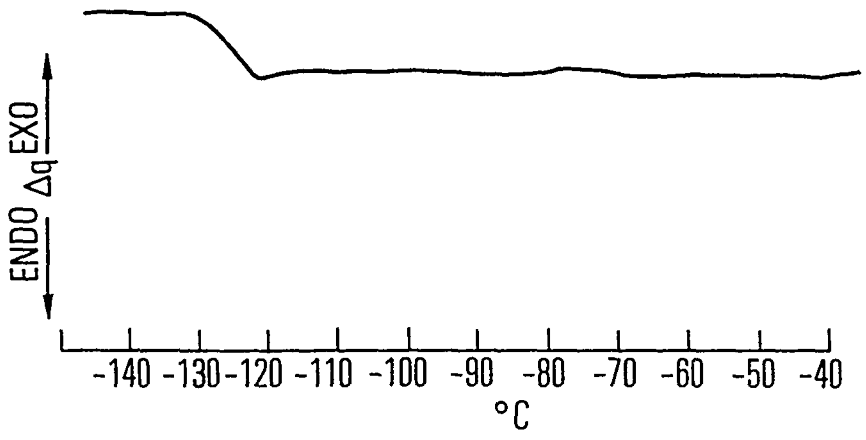

Fig. A-9. RTV-115. Heating rate = 10° C/min.

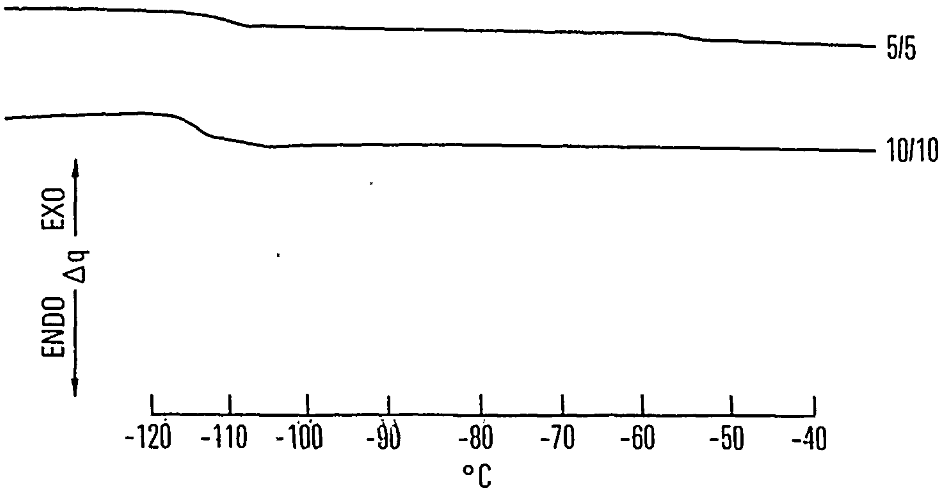

Fig. A-10. RTV-560

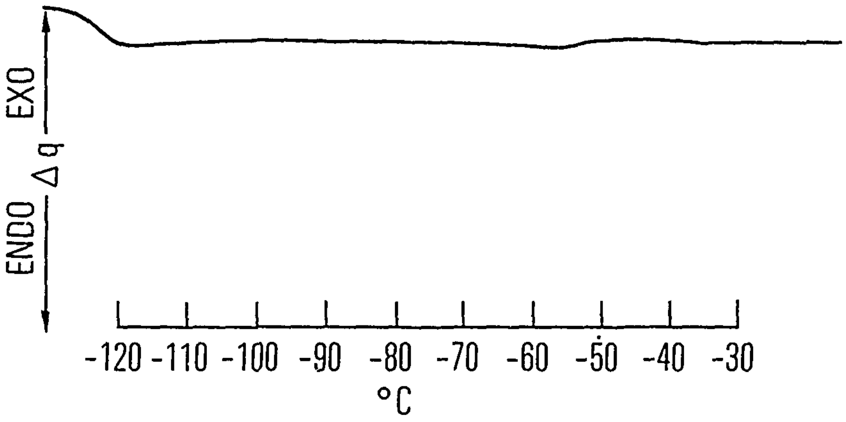

Fig. A-11. RTV-566. Heating rate = 10° C/min.

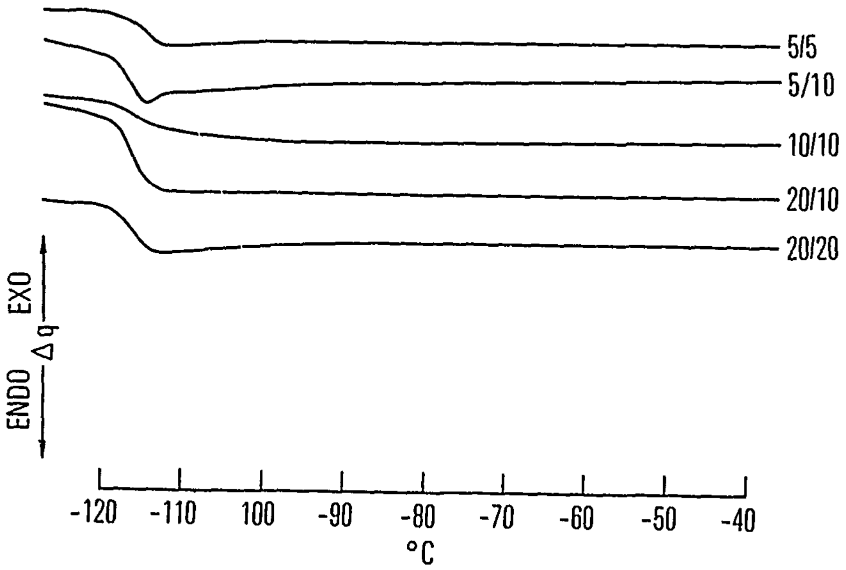

Fig. A-12. RTV-567

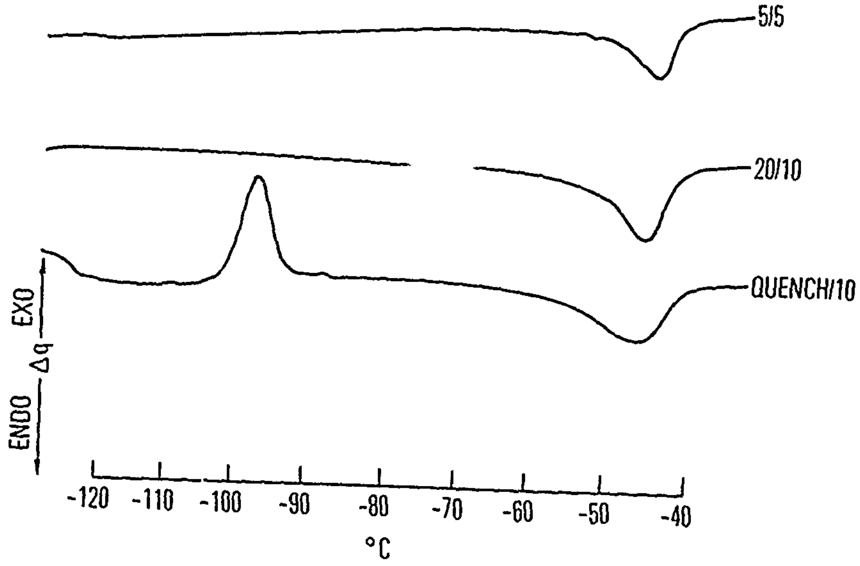

Fig. A-13. RTV-630

THE IVAN A. GETTING LABORATORIES

The Laboratory Operations of The Aerospace Corporation is conducting experimental and theoretical investigations necessary for the evaluation and application of scientific advances to new military concepts and systems. Versatility and flexibility have been developed to a high degree by the laboratory personnel in dealing with the many problems encountered in the nation's rapidly developing space and missile systems. Expertise in the latest scientific developments is vital to the accomplishment of tasks related to these problems. The laboratories that contribute to this research are:

Aerophysics Laboratory: Launch and reentry aerodynamics, heat transfer, reentry physics, chemical kinetics, structural mechanics, flight dynamics, atmospheric pollution, and high-power gas lasers.

Chemistry and Physics Laboratory: Atmospheric reactions and atmospheric optics, chemical reactions in polluted atmospheres, chemical reactions of excited species in rocket plumes, chemical thermodynamics, plasma and laser-induced reactions, laser chemistry, propulsion chemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, photosensitive materials and sensors, high precision laser ranging, and the application of physics and chemistry to problems of law enforcement and biomedicine.

Electronics Research Laboratory: Electromagnetic theory, devices, and propagation phenomena, including plasma electromagnetics; quantum electronics, lasers, and electro-optics; communication sciences, applied electronics, semiconducting, superconducting, and crystal device physics, optical and acoustical imaging; atmospheric pollution; millimeter wave and far-infrared technology.

Materials Sciences Laboratory: Development of new materials; metal matrix composites and new forms of carbon; test and evaluation of graphite and ceramics in reentry; spacecraft materials and electronic components in nuclear weapons environment; application of fracture mechanics to stress corrosion and fatigue-induced fractures in structural metals.

Space Sciences Laboratory: Atmospheric and ionospheric physics, radiation from the atmosphere, density and composition of the atmosphere, aurorae and airglow; magnetospheric physics, cosmic rays, generation and propagation of plasma waves in the magnetosphere; solar physics, studies of solar magnetic fields; space astronomy, x-ray astronomy; the effects of nuclear explosions, magnetic storms, and solar activity on the earth's atmosphere, ionosphere, and magnetosphere; the effects of optical, electromagnetic, and particulate radiations in space on space systems.

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