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HIGH TEMPERATURE ELECTRICAL CONDUCTIVITY AND THERMAL DECOMPOSITION OF PHENOLIC- AND SILICON-BASED DIELECTRICS FOR FIRESET HOUSINGS

R. T. Johnson, Jr. & R. M. Biefeld

August 1981

Sandia National Laboratories

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ABSTRACT

The temperature dependence of the electrical conductivity and thermal decomposition characteristics of several phenolic- and silicone-based materials of interest for fireset case housings have been measured to 600-700°C. The materials are phenolic or silicone resins reinforced with glass chopped fabric or cloth. The conductivity temperature dependence was measured during decomposition in a nitrogen atmosphere at a heating rate of ∿10°C/minute. Applied electric fields were from 4×10^2 to 4×10^3 volts/cm. Thermal decomposition characteristics were investigated by mass spectroscopy in vacuum and thermal gravimetric analysis in nitrogen and air. Nearly ohmic voltage-current characteristics were obtained, except where decomposition and/or outgassing was pronounced. For the silicone materials, the conductivity increases nearly monotonically with increasing temperature (to ∿400°C), approaching $\sim 10^{-8}$ (ohm-cm)$^{-1}$ at 400-600°C. The phenolics exhibit conductivity peaks at 250°C and ∿400°C. The peak magnitudes [maximum value $\sim 3 \times 10^{-8}$ (ohm-cm)$^{-1}$] and positions depend upon the material formulation, age, and post-cure conditions. Postcuring to 250°C for 1 h eliminates the conductivity peak at 250°C. The temperature dependence of the mass loss and decomposition products correlates with pronounced changes (peaks) in electrical conductivity. Results show that the silicone materials have better thermal stability and are better high temperature electrical insulators than the phenolic materials. Furthermore, the excellent correlation obtained between thermal and electrical properties suggests that electrical conductivity can be used as a thermal analytical tool in characterizing these materials.

TABLE OF CONTENTS

	Page No.
INTRODUCTION	5
MATERIALS	6
Phenolic Materials	6
Silicone Materials	7
EXPERIMENTAL METHOD	9
Electrical Measurement Techniques	9
Thermal Analysis Techniques	14
ELECTRICAL RESULTS	15
Phenolic Materials	15
Silicone Materials	28
THERMAL RESULTS	36
CORRELATION OF ELECTRICAL AND THERMAL RESULTS	46
CONCLUSIONS	53
ACKNOWLEDGEMENTS	54
REFERENCES	55
APPENDIX A: FOUR TERMINAL CONDUCTIVITY EXPERIMENTS	57
APPENDIX B: MASS SPECTRA RESULTS	61

TABLE OF CONTENTS

	Page No.
INTRODUCTION	5
MATERIALS	6
Phenolic Materials	6
Silicone Materials	7
EXPERIMENTAL METHOD	9
Electrical Measurement Techniques	9
Thermal Analysis Techniques	14
ELECTRICAL RESULTS	15
Phenolic Materials	15
Silicone Materials	28
THERMAL RESULTS	36
CORRELATION OF ELECTRICAL AND THERMAL RESULTS	46
CONCLUSIONS	53
ACKNOWLEDGEMENTS	54
REFERENCES	55
APPENDIX A: FOUR TERMINAL CONDUCTIVITY EXPERIMENTS	57
APPENDIX B: MASS SPECTRA RESULTS	61

I. INTRODUCTION

Nuclear safety concerns over the possibility that a weapon may be subjected to an accident involving a fuel fire and that a high voltage source may be present indicated the need for information about the high temperature electrical insulating characteristics of fireset case materials. The materials of interest included those based on phenolic and silicone resins reinforced with glass chopped fabric or cloth which could be molded or formed into complex shapes suitable for fireset case housings.

There is very little information in the literature on the high temperature electrical properties of these materials. Some data are available on changes in the dielectric properties of resins during the curing process (to temperatures of $\sim$210°C) [1-4], and on the properties of pyrolyzed polymers (material obtained after pyrolysis to temperatures $\gtrsim$400°C) [5-10]. Except for limited data on other materials [11], there is practically no information available on changes in electrical conduction characteristics during the decomposition process [12,13] as the temperature is increased to 600-700°C. The purpose of the studies reported herein was to obtain such information on the materials of interest for fireset applications under conditions that approximate those expected in a fuel fire environment.

Depending upon the fuel fire accident scenario, values for the temperature, voltage and time will vary. For our investigations, conductivity changes for temperature increasing

to 600-700°C at a rate of ∿10°C/minute were examined. Applied voltages were up to 1000 V dc across flat plate specimens ∿3 mm in thickness (electric fields up to ∿4 x 10^3 volts/cm). The electrical experiments were conducted in a nitrogen environment since the atmosphere could be oxygen deficient during a fuel fire. In the materials examined, thermally-induced chemical and structural changes result in material decomposition at high temperature [12,13]. Consequently, the electrical data are taken when the material is in a state of non-equilibrium. Information on decomposition products and material mass loss was obtained using mass spectroscopy (in vacuum) and thermal gravimetric analysis (in nitrogen and air). The thermal data are correlated with the conductivity results and provide insight into relationships between electrical conduction and thermal decomposition.

II. MATERIALS

Two phenolic and three silicone based materials reinforced with glass chopped fabric or cloth were examined in this study [14,15]. The phenolic and silicone resins which are highly crosslinked were chosen for their high temperature stabilities [15]. The samples examined herein used the same starting materials and molding or forming conditions as specified for fireset housings.

A. Phenolic Materials

FM-5135

The starting material, U. S. Polymeric FM-5135, contains the Ironside Chemical Co. phenolic resin system 91LD with

a chopped half-by-half inch glass fiber reinforcing filler [16]. The material is molded at Bendix Corporation, Kansas City, at a cure temperature of approximately 149°C, pressure of 3185 psi for a period of 25 minutes. The material is a single stage phenolic which utilized ammonia as a catalyst. Ammonia is liberated during the curing process. This is of concern in utilizing this material for fireset applications because ammonia that may be liberated during long term storage may induce compatibility problems with other fireset components.

MXB-71

The starting material is Fiberite Corp. MXB-71 which is a blend of two resins. It is formulated using the Monsanto resin system SC-1008, a nylon modifier incorporated for toughness, and chopped half-by-half inch glass fabric reinforcing. This material is also a one stage phenolic and utilizes a proprietary amine as a catalyst. The material is molded at Bendix, Kansas City, under the same conditions as for the FM-5135 material (i.e. 149°C, 3185 psi, 25 minutes). No ammonia has been detected as a result of the curing process [17].

B. Silicone Materials

DC-302

Dow-Corning DC-302 is a fiberglass-filled high temperature silicone resin. The starting material consists of chopped glass fabric coated with a polyphenylmethysiloxane resin in which is dispersed an iron-oxide/silicon-oxide filler [18]. The composition is approximately 32% resin, 46% fiber, and 22% particulate [19]. The material is molded at Bendix,

Kansas City under approximately the same conditions as for the phenolics.

BRP-50201

This material is a Bendix, Kansas City formulation of a glass mat impregnated with a silicon resin [20]. It is based on Dow Corning's QR-4-3136 silicone resin and on a continuous strand fiberglass mat (M-8610) from Owens-Corning Fiberglas Corporation. It consists of 53% glass, 32% resin and 15% fillers and additives. This material was developed by Bendix as a replacement for DC-302, since DC-302 is being discontinued by Dow Corning as a commercial item. The molding process is the same as for the phenolics and DC-302.

CS-4202

This material is a silicone-glass composite which utilizes a pre-pregnated continuous glass fiber cloth. The starting material is supplied by Ferro Corp. and is a composite of a silicone resin formulated by Ferro Corp. (CS-4202, based on Dow Corning DC-2106) which is impregnated on a continuous woven fiber cloth (7781E-glass from Corning). The material is supplied as a roll of pre-pregnated glass fabric. The cloth is cut to desired shape and parts are processed (at Sandia National Laboratories in Albuquerque) in an autoclave to a 12 ply thickness. The material is vacuum sealed in a plastic bag and experiences a time, temperature, pressure cycle during processing. A two-stage processing operation in the autoclave is used. The first stage involves 150 psi pressure, 177°C for one hour. The material is then postcured in the autoclave for

3 hours at 177°C. This is followed by a postcure under atmospheric pressure at 204°C for 2 hours. The material is then further postcured at 249°C for times which are selected to vary from 4 to 24 hours. This material was developed by Kevin Hirschbeuhler in the Composite Materials Development Division at Sandia National Laboratories [21].

III. EXPERIMENTAL METHOD

A. Electrical Measurement Techniques

Flat specimens, ∿0.3 cm thick in the shape of squares ∿3 cm on a side were used. Electrodes for two-terminal conductance (or resistance) measurements were circular (∿1.1 cm diameter) and were placed on opposite faces of the sample as shown in Fig. 1a. The guard-ring electrode configuration of Fig. 1b (outer guard ring grounded) was used to evaluate effects of surface currents. The fully electroded configuration (Fig. 1c) was used to determine effects of fringe fields associated with the Fig. 1a arrangement.

Electrodes were prepared using silver paint, evaporated gold, or aquadag (graphite). There was no significant dependence of the conductance on electrode material. However, the best adhesion was obtained with the silver electrodes. As a result, silver electrodes were used for most of the experiments reported herein. There is an indication that volatiles are partially trapped by the electrodes (see Appendix A). The configuration of Fig. 1a was found to be adequate for these experiments, in that there did not appear to be any serious electrode problems related to volatilization.

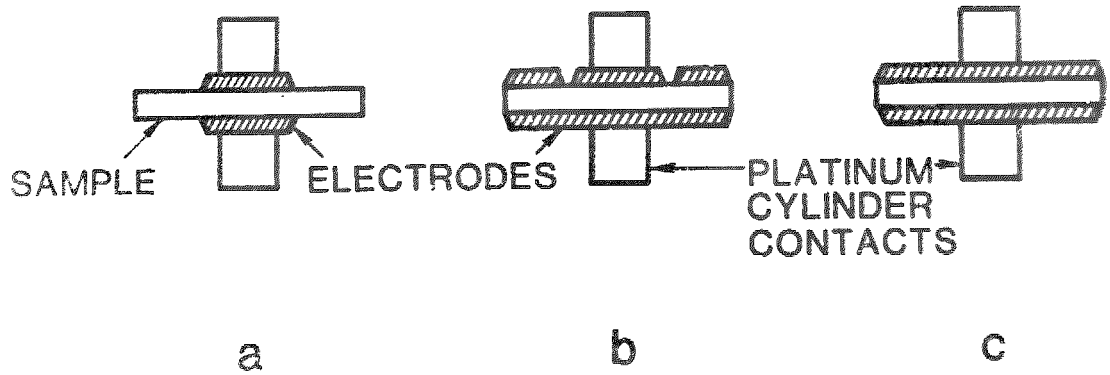


Figure 1. Sample and electrode configuration for electrical conductivity measurements: a) standard measurement arrangement, b) guard ring configuration (outer guard ring grounded to minimize surface current effects), and c) fully electroded (to measure effects of fringe fields associated with arrangement in part a).

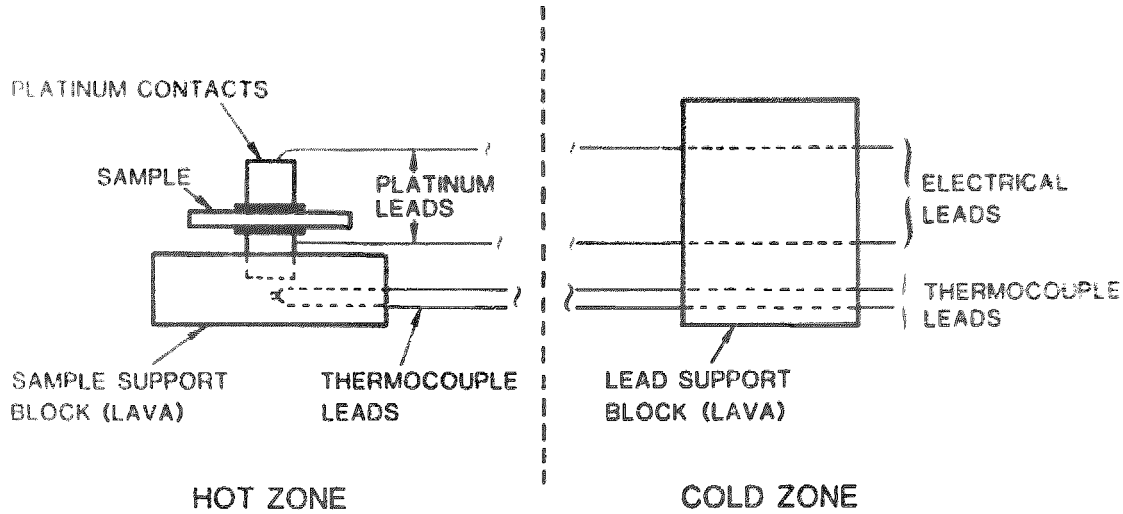


Figure 2. Sample holder configuration.

Electrical contact was made to the electroded samples using platinum cylinders as shown in Fig. 2. The bottom cylinder served to support the sample above the insulating support block. Platinum wires were used as electrical leads. These wires were encased in separate alumina tubes passing from the high temperature zone of the furnace to the low temperature region where they were supported by another insulating block (lava or boron nitride). This configuration minimized leakage currents since current paths between the electrodes were not in the hot zone of the furnace. Leakage currents were below 10^{-9} amps at 1000 V for temperatures below 400°C. At higher temperatures the leakage currents increased somewhat but were below 10^{-7} amps (at 1000 V) at 700°C. This is three to four orders of magnitude less than conduction obtained from the samples at these temperatures.

The temperature was measured using a thermocouple mounted in the sample support block as indicated in Fig. 2. The temperature lag between the sample support block (where the recording thermocouple was located) and the sample was measured (at the 10°C/minute heating rate) and was found to be within 10°C over the temperature range examined.

For the conductivity experiments, the sample holder assembly (Fig. 2) was positioned in an open-ended quartz tube which facilitated loading and positioning in the furnace assembly. The furance assembly was made by Marshall Furnace Co. and contained a tubular chamber which could be sealed to control the atmosphere. Separate thermocouples were used for

the furnace control and the sample temperature. In these experiments the furnace assembly was flushed with nitrogen and then sealed at a gauge pressure of ∿10 psi. The possibility of leakage currents through the vapors emitted from the samples during decomposition was also explored and was found to be insignificant. After a series of tests were completed, some of the chemicals in the heated gas condensed after cooling on parts of the sample holder. After a number of tests this caused some conduction but generally in the 10^{-8} A range or less. By baking out the sample holders and the quartz tube at 600-700°C after tests, this leakage was reduced to below 10^{-9} A.

The electrical measurement system schematic is shown in Fig. 3. A 100 K ohm resistor was placed in series with the high potential lead to limit short circuit currents. An automatic data acquisition system was used. The system was programmable so that several voltages could be sequentially applied to the specimen at selected time intervals. Voltage levels of 100, 300, 600 and 1000 V were used and were applied for 2-6 seconds at each voltage setting as the temperature was increasing at a rate of 10°C per minute. Temperature and current readings were taken at each voltage setting.

Possible effects associated with the time duration of the applied voltage were examined. An experiment was conducted in which the voltage was applied continuously at 1000 V. The results were compared with those from a sister sample cut from the same disk which was tested in the same system following the normal time interval step sequence. The results showed that

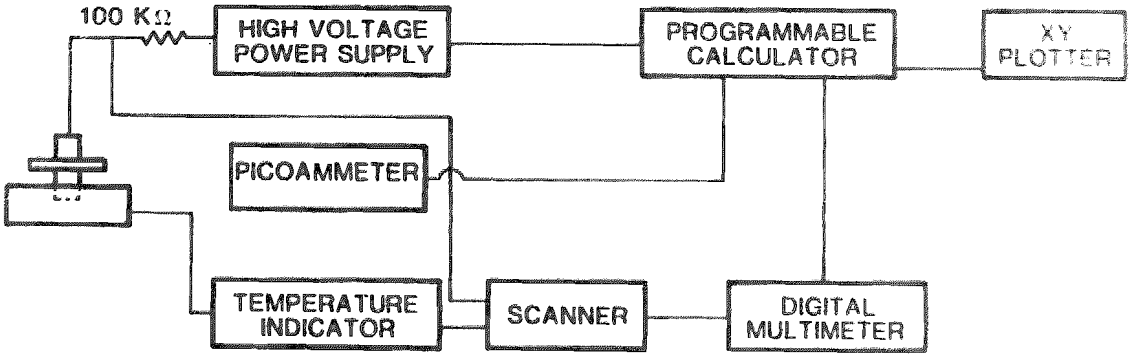


Figure 3. Electrical measurement system schematic showing the major components: Programmable Calculator (Monroe Model 1880), XY Plotter (Hewlett Packard Model 9872A), Operational Power Supply (Kepco Model OPS 2000B), Digital Picoammeter (Keithley Model 445), Digital Multimeter (Keithley Model 172A), Scanner (Keithley Model 702), Digital Temperature Indicator (Omega Model 199).

the length of time for voltage application did not significantly effect the measurements. This has relevance to the determination of possible polarization effects which is discussed in later sections.

The electrical conductivity (reciprocal of resistivity) was determined from

$$\sigma = k \frac{\ell}{A} \frac{I}{V}$$

where ℓ is the sample thickness, A is the electrode area, I is the sample current, V is the applied voltage, and k is a geometric correction for fringing fields. For the geometry of Fig. 1a, k was determined experimentally to have a value of $\sim$0.72 (note that k = 1 for the geometry of Fig. 1c).

The accuracy of the conductivity temperature dependence data, estimated from run-to-run reproducibility for a given material (sister samples), was found to be within a factor of two of the measured conductivity value.

B. Thermal Analysis Techniques

Thermal property measurements were made using high temperature mass spectroscopy and thermal gravimetric analysis. The mass spectrometer was a Bendix Model 3015 Time-of-Flight Mass Spectrometer which was equipped with a high temperature Knudsen inlet system. The spectra were obtained in vacuum at fixed temperatures with an electron beam energy of 65 eV. Time at temperature was $\sim$30 minutes. A heating rate of $\sim$10°C/minute was employed between temperature settings. Data were taken to $\sim$400°C. No changes in the spectra were observed

between ∿350 to 400°C except for a continuous increase in total pressure and ion current.

Two thermogravimetric analyses techniques were used. In one method the samples were heated in both air and flowing nitrogen for one hour at 100°C intervals up to 700°C. The heating and cooling rates were ∿25°C/minute. Percent weight loss was determined following each temperature cycle. The second method involved measuring weight loss while heating samples at 10°C/minute in nitrogen. A DuPont Model 990 Thermal Analyzer with a Model 951 Thermogravimetric Analyzer were used for these measurements. The precision associated with these techniques was estimated to be ±0.1 and ±0.5 wt.%, respectively.

IV. ELECTRICAL RESULTS

A. Phenolic Materials

Typical current versus temperature data obtained at four different applied voltages on as-molded phenolic FM-5135 and MXB-71 samples are shown in Figs. 4 and 5, respectively. The results show two peaks in the conductance at ∿250 and ∿400°C. The peak at ∿400°C is more pronounded in MXB-71 than in the FM-5135 material. Analysis of the current-voltage data shows ohmic (or nearly ohmic) conduction characteristics, in that the sample current scales nearly linearly with applied voltage. Deviations from ohmic behavior occur principally in the temperature regions below ∿270°C for FM-5135 and below ∿300°C for MXB-71. Non-ohmic behavior is most pronounced in the low temperature insulating state (<100°C) and in the region of the conductivity peak at 200-300°C. In these regions conductivity

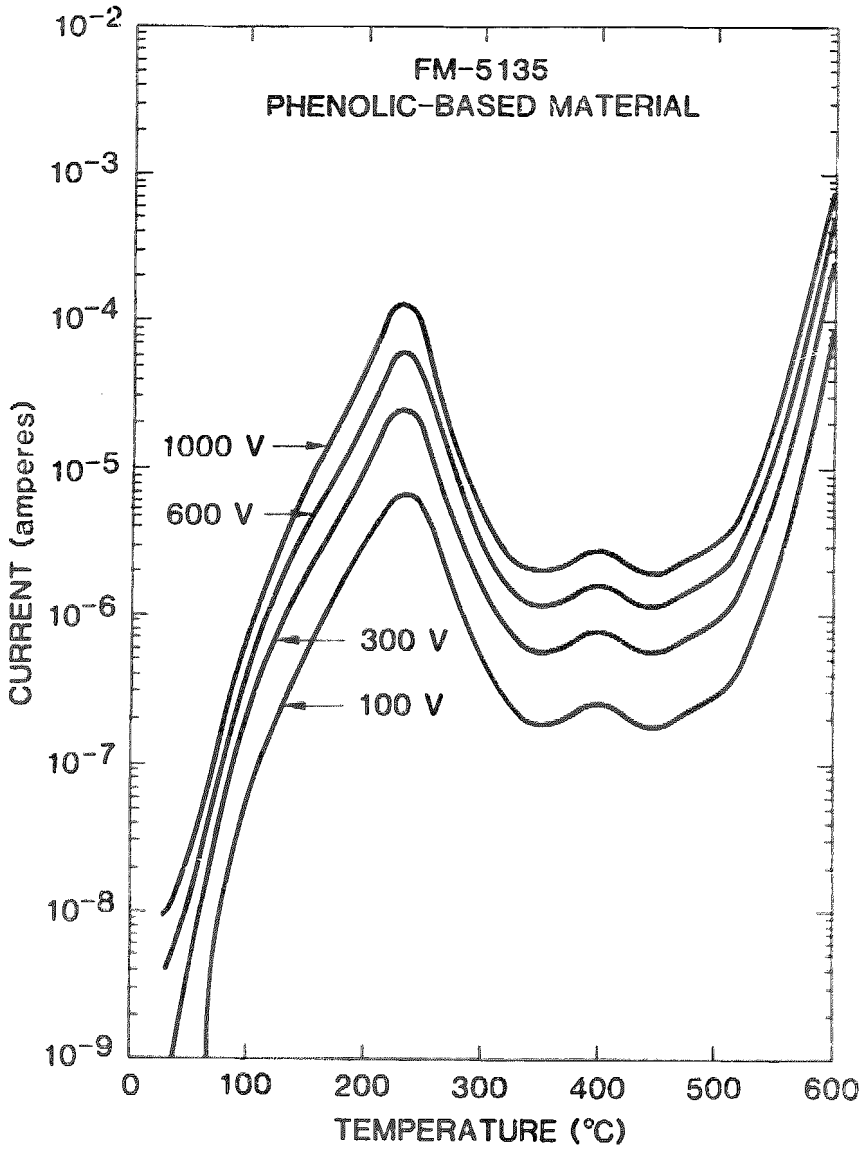


Figure 4. Typical sample current versus temperature for FM-5135 phenolic as a function of voltage. This material had aged (in the ambient environment) for 2-3 years prior to these experiments. Results show conductivity peaks at ∿250 and ∿400°C. Analysis of current-voltage data indicate deviations from ohmic conduction behavior in the region ≲270°C.

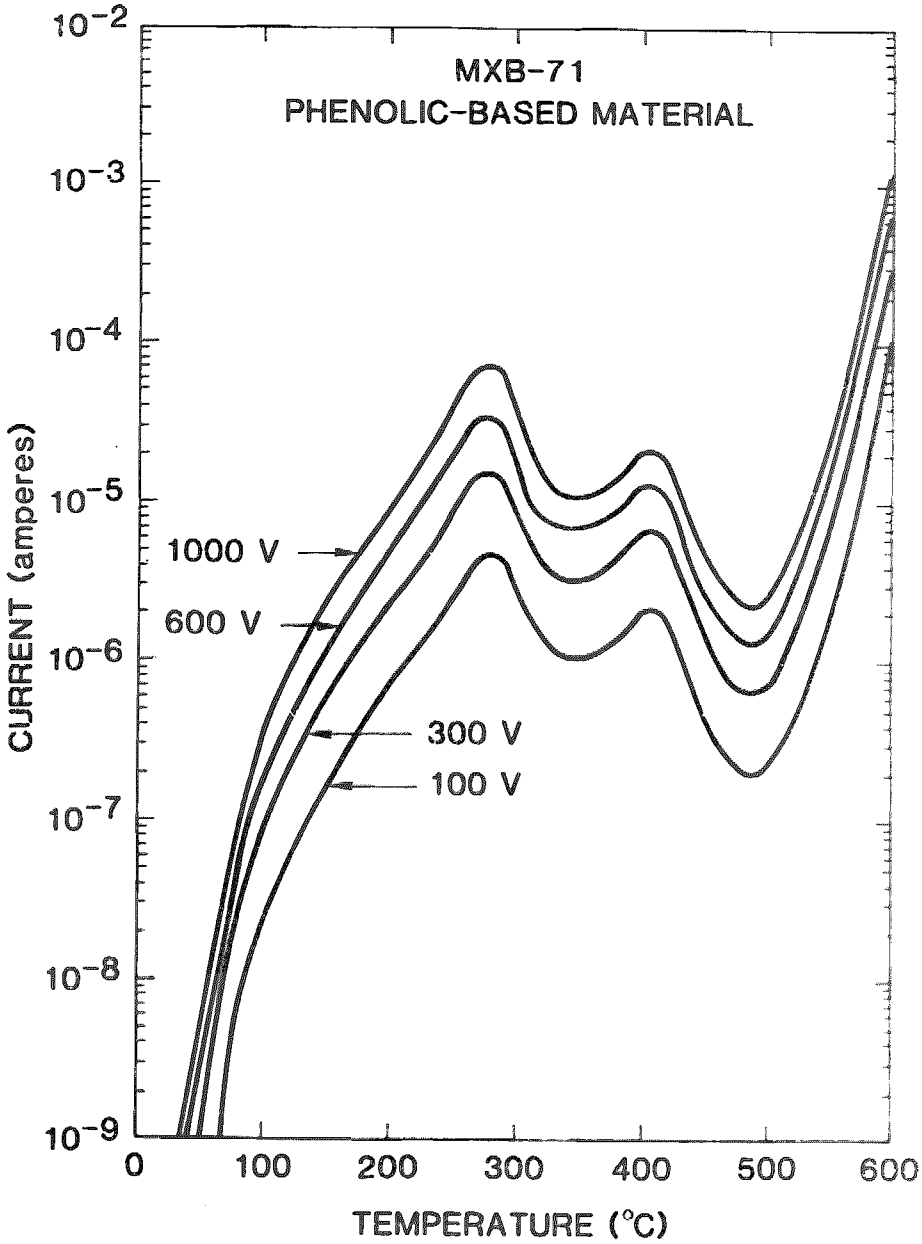


Figure 5. Sample current versus temperature for MXB-71 phenolic as a function of voltage. This material had aged in the ambient environment for 2-3 years prior to these experiments. Results show conductivity peaks at ∿270 and ∿400°C. Analysis of current-voltage data indicate deviations from ohmic conduction behavior in the region ≲300°C.

values determined from the 100 and 1000 V data differed by a factor of at most 2. In plots of the conductivity data throughout this report, temperature regions where there are significant deviations from ohmic behavior are indicated by the dashed lines.

The conductivity temperature dependence was measured on a number of samples from various lots of material. Results showed good repeatability in conductivity characteristics from sample-to-sample within a given lot of material. Lot-to-lot variations are shown in Figs. 6 and 7 and are more pronounced in the FM-5135 phenolic than in MXB-71. The extent to which these differences are associated with starting materials (formulation or age), processing conditions, or simple age is not known.

The effects of postcuring in air on the electrical conductivity are shown in Figs. 8-10. The data in Figs. 8 and 9 are for FM-5135 material from lots with different as-molded conductivity characteristics. The data in Fig. 10 are for MXB-71 material. Results show that in all of these materials postcures of 143°C for 16 hours followed by 200°C for one hour reduce the conductivity in the low temperature region ($\lesssim$250°C), reduce somewhat the magnitude of the conductivity peak at $\sim$250°C, and shift the peak to a higher temperature. Postcures to 250°C for one hour further reduce the conductivity in low temperature regions and eliminates (or significantly reduces) the conductivity peak at $\sim$250°C. Note that ohmic conduction was observed throughout a greater temperature range for the material

Table 1

Thermogravimetric Analysis Results

Data in parentheses are from experiments with temperature increasing at continuous rate of 10°C/min.

Material	Atmosphere	Percent Weight Loss						
		100°C	200°C	300°C	400°C	500°C	600°C	700°C
Phenolics								
FM-5135	N_2	0.7(0.3)	1.4(0.9)	2.2(1.3)	4.1(2.2)	9.9(5.0)	13.0(10.4)	14.0(12.9)
	air	0.7	1.3	2.5	18.8	38.1	38.1	38.1
MXB-71	N_2	0.7	1.3(0.7)	2.1(1.3)	8.1(2.3)	14.1(7.7)	16.7(12.7)	17.7(15.2)
	air	0.6	1.0	2.3	17.2	38.2	38.3	38.3
Silicones								
DC-302	N_2	0.1	0.2	0.4(0.7)	1.5(1.2)	4.5(3.7)	8.1(8.8)	8.8(14.3)
	air	0.05	0.3	0.9	2.1	6.0	12.4	12.5
BRP-50201	N_2	0.00	0.7(0.2)	1.0(0.5)	1.9(1.0)	5.0(2.0)	8.8(6.0)	9.0(9.0)
	air	0.06	0.4	0.8	1.3	3.8	13.6	14.7
CS-4202	N_2	0.04	0.1(0.2)	0.3(0.6)	1.3(1.4)	3.6(3.0)	6.6(7.0)	7.2(8.0)
	air	0.00	0.09	0.5	1.7	5.2	11.2	11.2

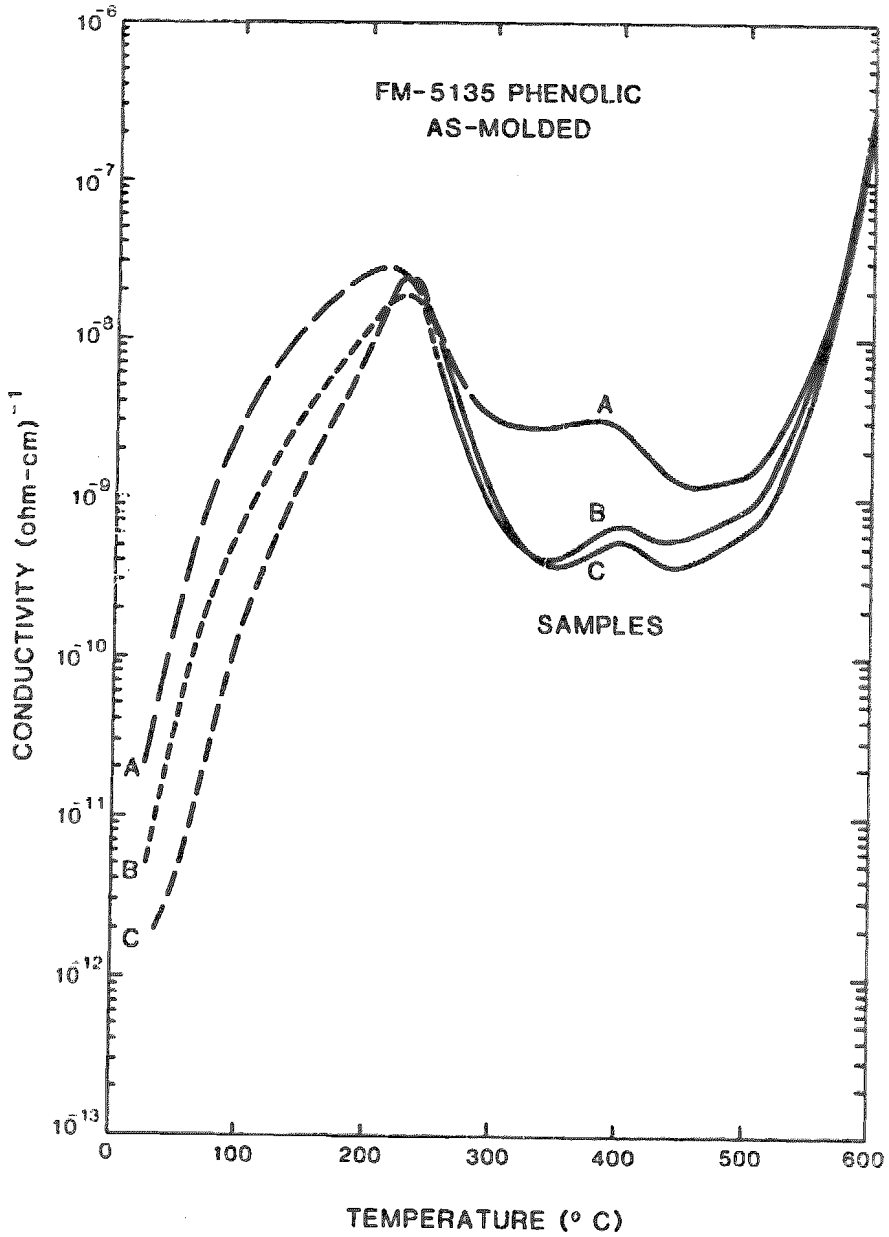


Figure 6. Conductivity temperature dependence for FM-5135 phenolic showing lot-to-lot variations. Sample A: starting material ∿5 months old, time since molding ∿1 month. Sample B: starting material ∿2 years old, time since molding ∿1 month. Sample C: starting material age unknown, time since molding 2-3 years. Conductivity differences may be due to starting material (formulation or age), processing conditions or sample age. Dashed portion of curves indicates regions of non-ohmic behavior.

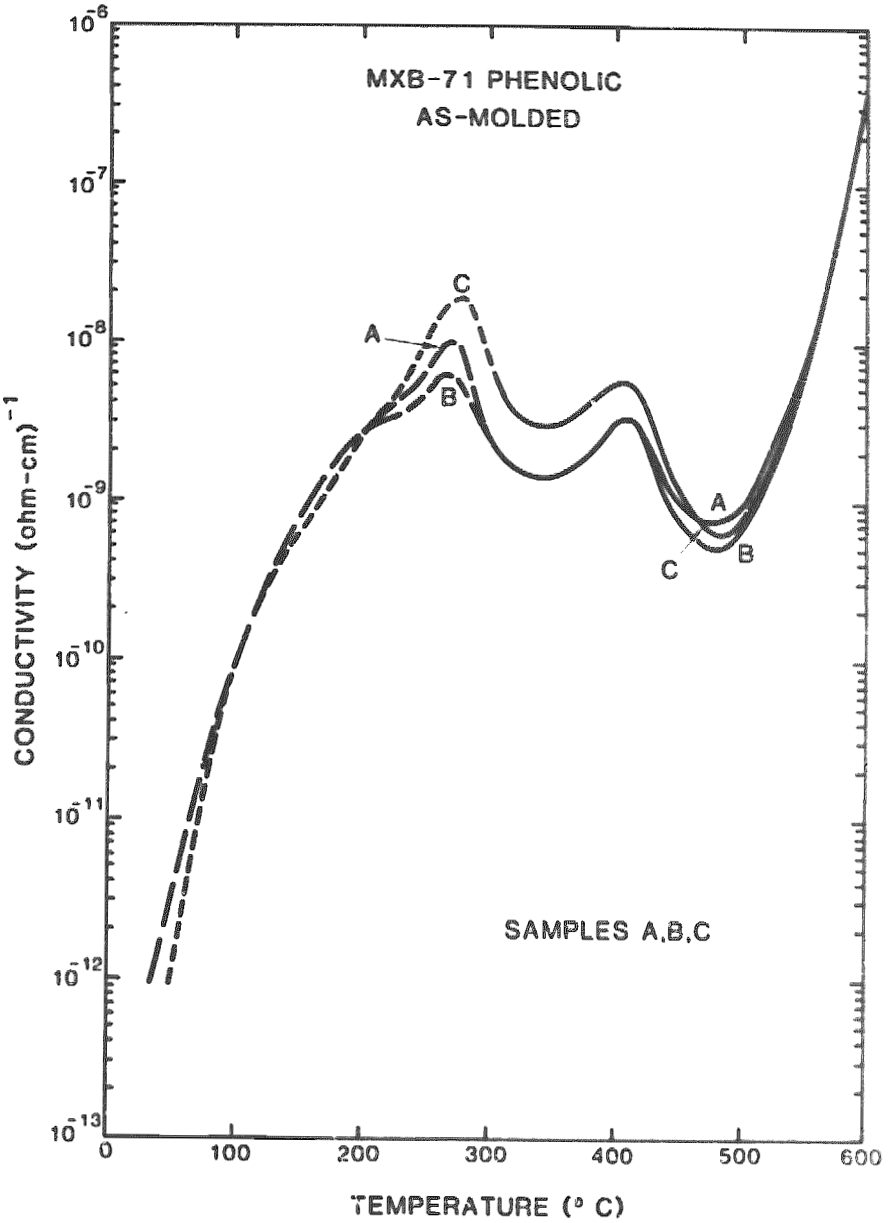


Figure 7. Conductivity temperature dependence for MXB-71 phenolic showing lot-to-lot variations. Sample A: starting material ∿5 months old, time since molding ∿1 month. Sample B: starting material ∿3 years old, time since molding ∿1 month. Sample C: starting material age unknown, time since molding 2-3 years. Conductivity differences may be due to starting material (formulation or age), processing conditions, or sample age. Dashed portion of curves indicates regions of non-ohmic behavior.

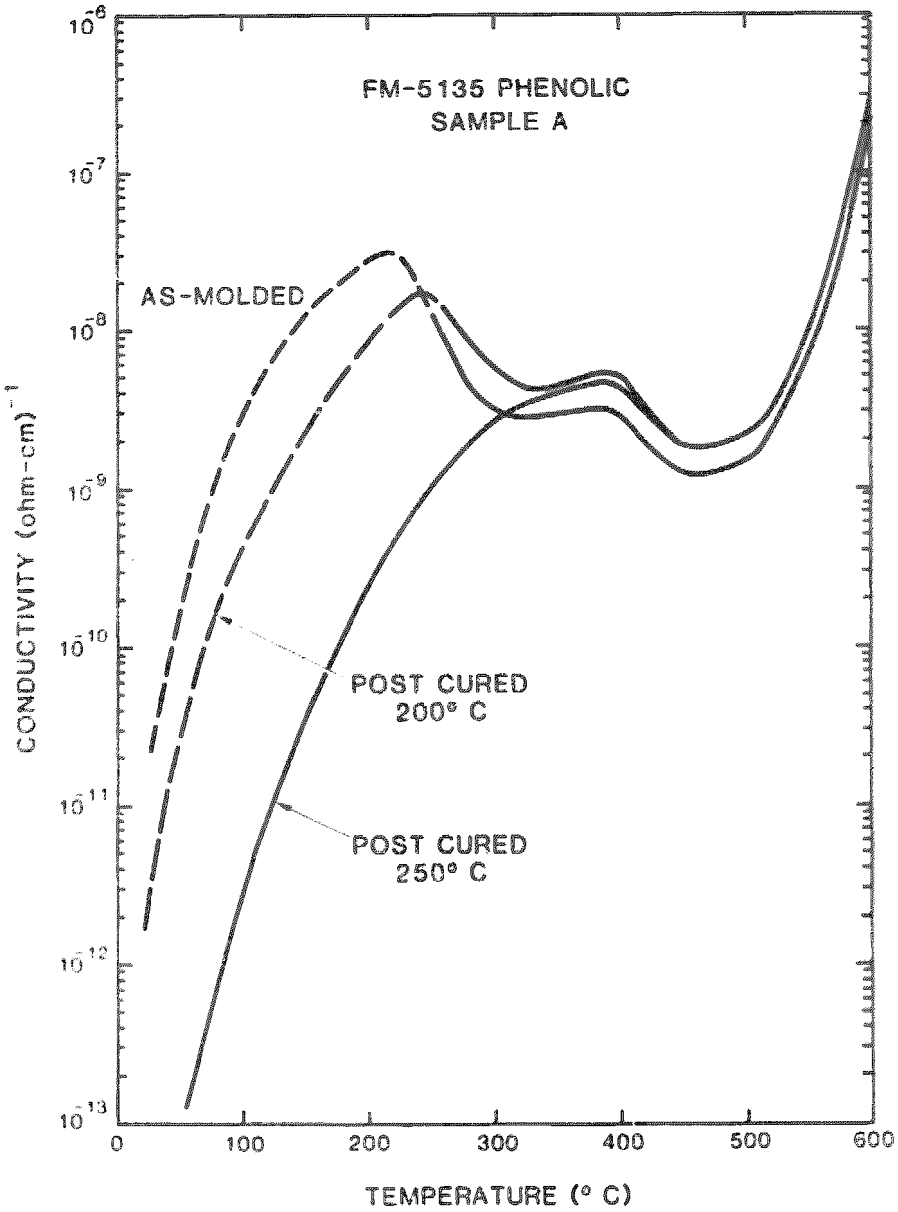


Figure 8. Conductivity dependence on postcuring (in air) for FM-5135 phenolic. Data are for samples prepared with starting material ∿5 months of age and measured ∿1 month after molding (same material as Sample A in Fig. 6). Postcure conditions are given in test. Dashed portion of curves denotes non-ohmic regions.

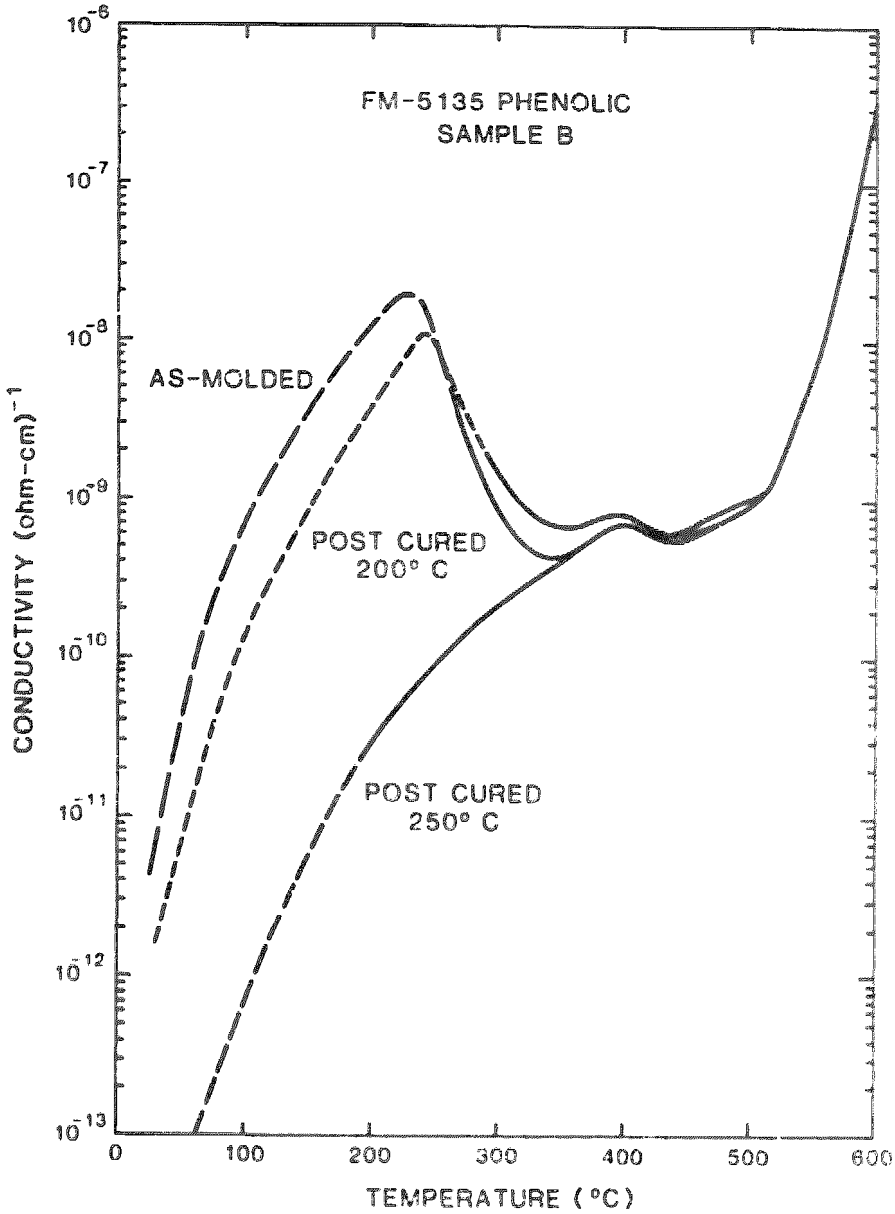


Figure 9. Conductivity dependence on postcuring (in air) for FM-5135 phenolic. Data are for samples prepared with starting material over 2 years old and measured ~1 month after molding (same material as Sample B in Fig. 6). Postcure conditions are given in text. Dashed portion of curves denotes non-ohmic region.

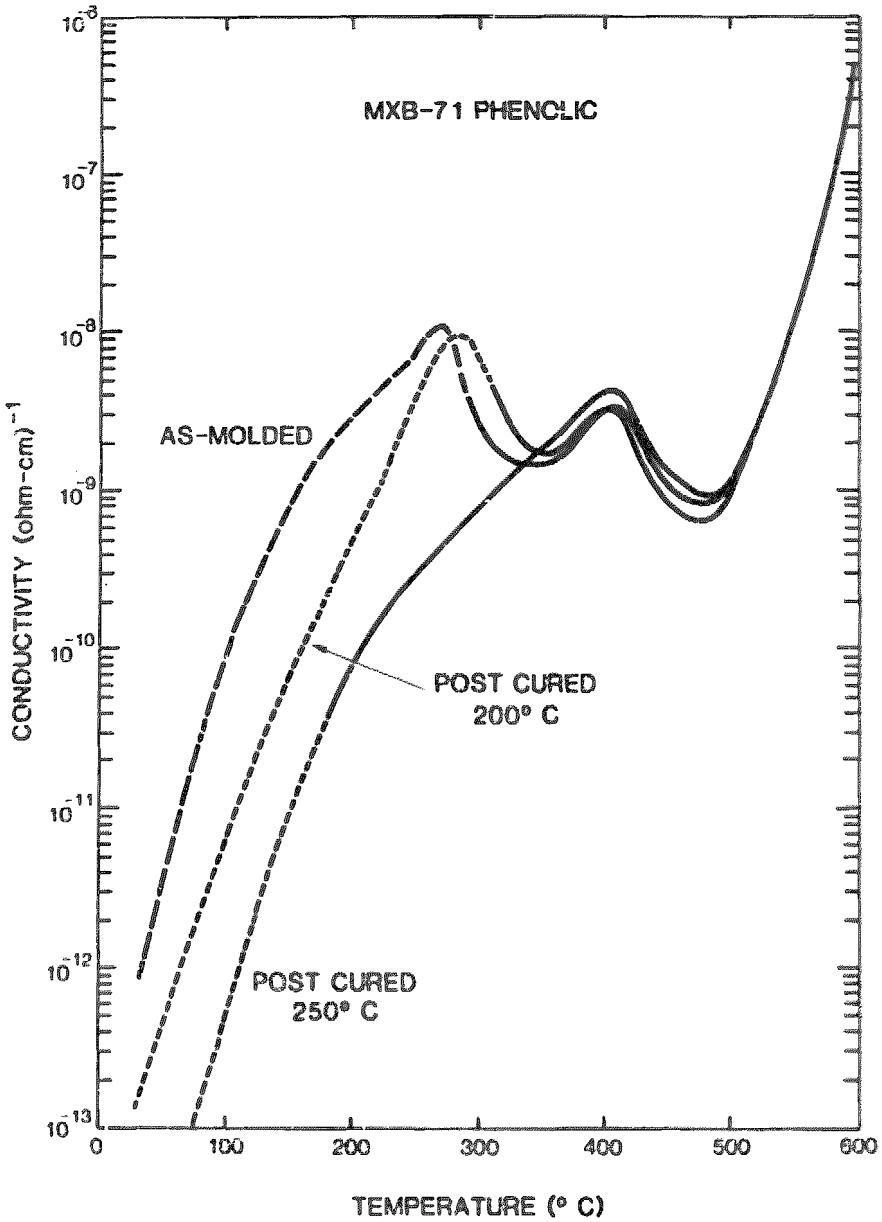


Figure 10. Conductivity dependence on postcuring (in air) for MXB-71 phenolic. Data are typical for these materials (i.e., variations from lot-to-lot were not as pronounced as for FM-5135, see Figs. 8 and 9). Postcure conditions are given in text. Dashed portion of curves denotes non-ohmic region.

postcured to 250°C (i.e. after elimination of the conductivity peak at ∿250°C).

Visible changes in the coloration of the materials were observed as a result of postcuring and cycling to high temperature. Postcures to 200°C for one hour did not change the appearance of the phenolics in comparison with the as-molded material. Postcures to 250°C for one hour caused a notable darkening of the material. For rapid heating and cooling rates some delamination occurred. Consequently, for postcuring to 250°C heating rates were used which were comparable to those utilized in the electrical experiments where no delamination occurred.

Upon temperature cycling to 600°C, the material further pyrolyzed, became porous, and developed a blackish appearance. The electrical contacts, made using silver paint, did remain intact after cycling to 600°C. There were small regions within the electrode which appeared porous. This could have resulted from the decomposition process.

The temperature dependence of the conductivity of a pyrolyzed FM-5135 sample was measured after it was cycled to ∿610°C in nitrogen. The results are shown in Fig. 11. The data are compared with those obtained on the sample in the as-molded state. Results show that conductivity peaks are completely eliminated after cycling to ∿610°C, but the resulting material is fairly conductive. Upon heating to higher temperatures, the material continued to pyrolyze (carbonized) and the conductivity increased further. The most pronounced changes (increases) were in the lower

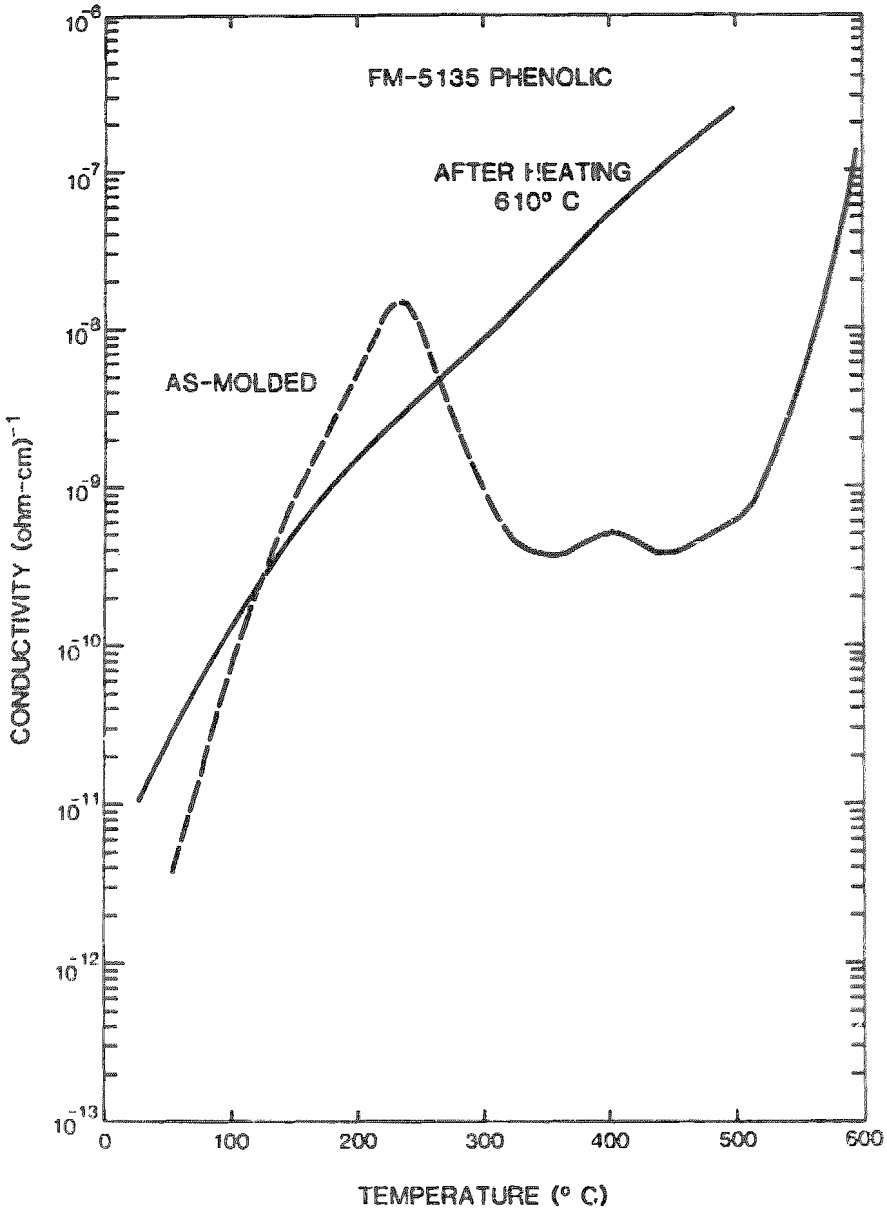


Figure 11. Conductivity temperature dependence of FM-5135 phenolic: as molded and after temperature cycling to ∿610°C. Dashed portion of curve denotes non-ohmic region.

temperature region. The carbonized material exhibited ohmic conductance characteristics.

Surface conduction did not appear to be significant (or pronounced) in these materials. Measurements made using the three-terminal guard ring technique of Fig. 1b (outer guard ring grounded) on pyrolyzed material (after heating to ∿600°C) showed that the measured conductivity was within 10% of that measured using the standard technique (no guard ring, Fig. 1a). Further evidence that surface conduction was not significant was seen in the results of four-terminal experiments discussed in Appendix A.

Polarization effects were also examined. As mentioned in Section III, the length of time for application of voltage did not significantly effect results during a heating cycle. Furthermore, the voltage polarity was also reversed several times during some of the conductivity runs, producing only minor changes on the conductance (within ∿10%). In addition to these results, polarization experiments were conducted at a fixed temperature of 160°C. The time dependence of the sample current was measured after application of voltage (1000 V). The voltage polarity was also reversed during these experiments. The initial experiments were on materials which had not been postcured. The results were difficult to interpret since time dependent effects related to curing as well as possible polarization phenomena were observed. Experiments at 160°C on materials which had been postcured to 200°C indicated that some polarization phenomena may occur,

but again the affect on conductance was not greater than ∿10%. Similar polarization experiments were not conducted at higher temperatures because of complicating effects associated with material decomposition. No significant polarization was observed in the carbonized material. These results suggest that conduction is principally electronic in character in the carbonized material and in the postcured material below ∿200°C.

B. Silicone Materials

The temperature dependence of the sample current as a function of applied voltage for the silicone materials is shown in Figs. 12-14. Analyses of these data show nearly ohmic conduction, with deviations from ohmic behavior being not as pronounced as for the phenolics. Some non-ohmic behavior occurs throughout much of the temperature range for these materials in the as-prepared state and after cycling to high temperature. Generally conductivity increases of ∿50% were observed over the voltage range of 100 to 1000 V. More significant deviations from non-ohmic behavior (similar to those for the phenolics) occurred in the low temperature insulating state and at high temperatures $\gtrsim$450°C. These regions are denoted by the dashed portions of the conductivity curves which are given in Figs. 15-17. The conductivity temperature dependence is considerably different for these materials than for the phenolics. Specifically, the conductivity is much reduced in the lower temperature region and the conductivity peaks are less pronounced.

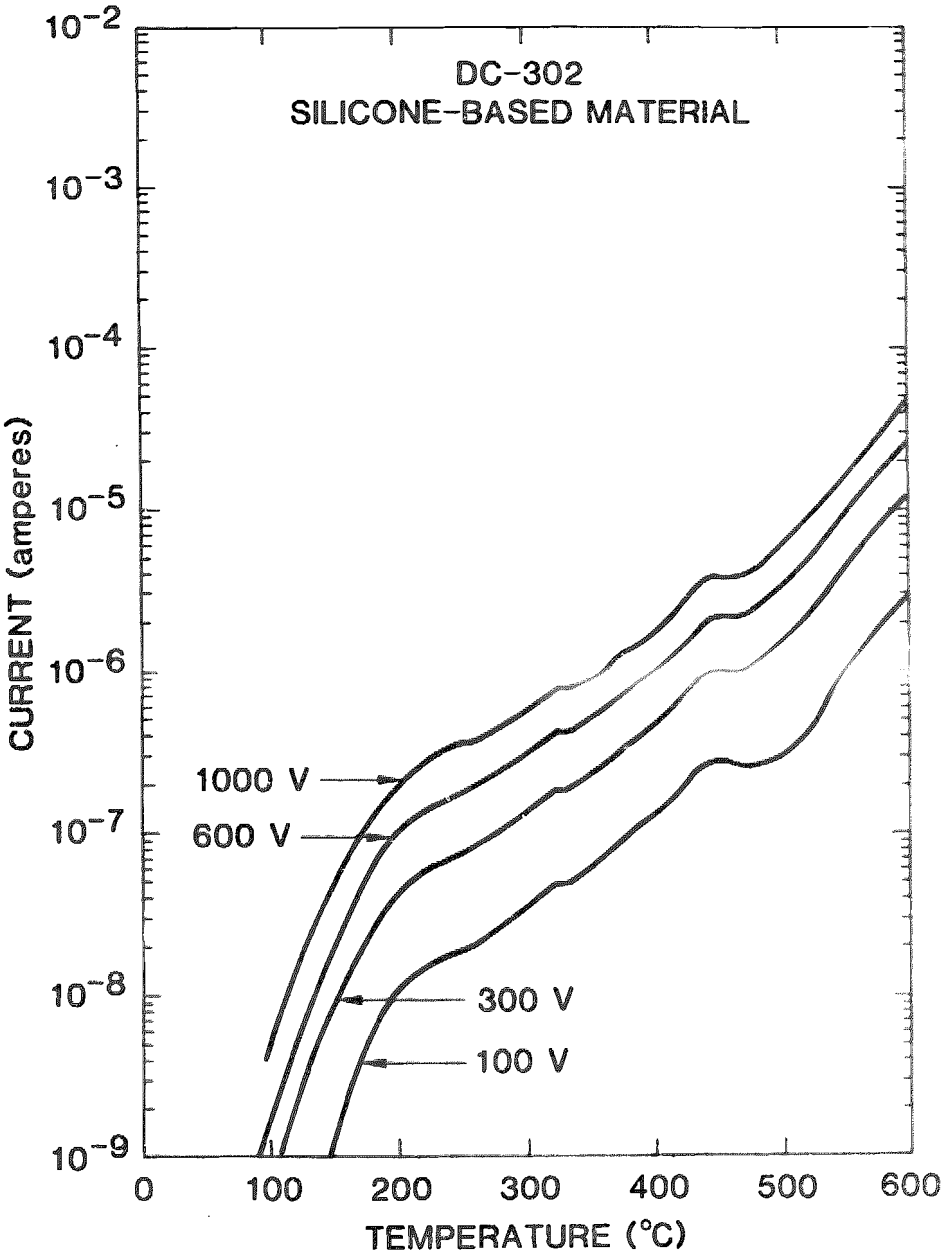


Figure 12. Sample current temperature dependence as a function of applied voltage for a typical DC-302 silicone sample.

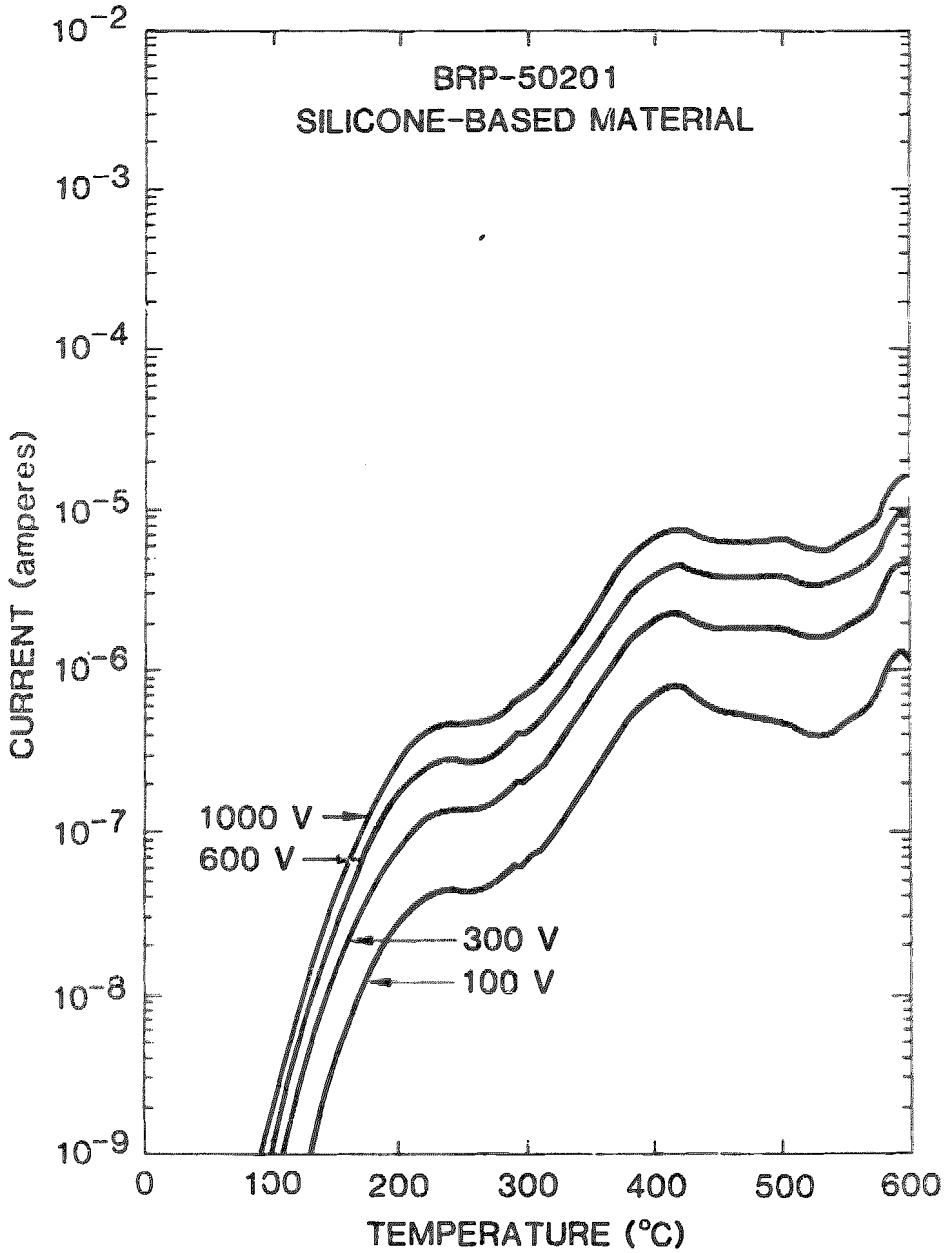


Figure 13. Sample current temperature dependence as a function of applied voltage for a typical BRP-50201 silicone sample.

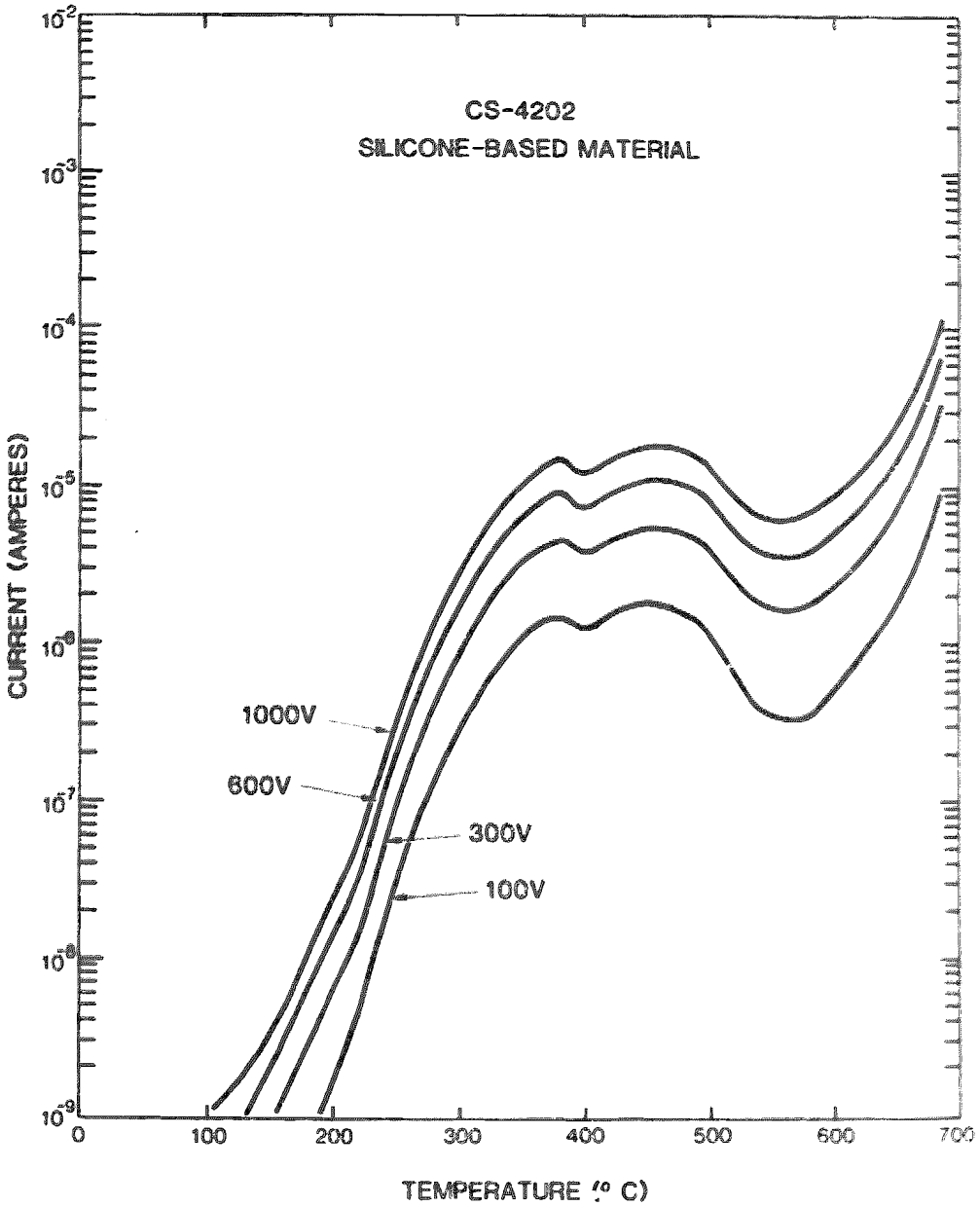


Figure 14. Sample current temperature dependence as a function of applied voltage for a typical silicone-glass composite, CS-4202, sample.

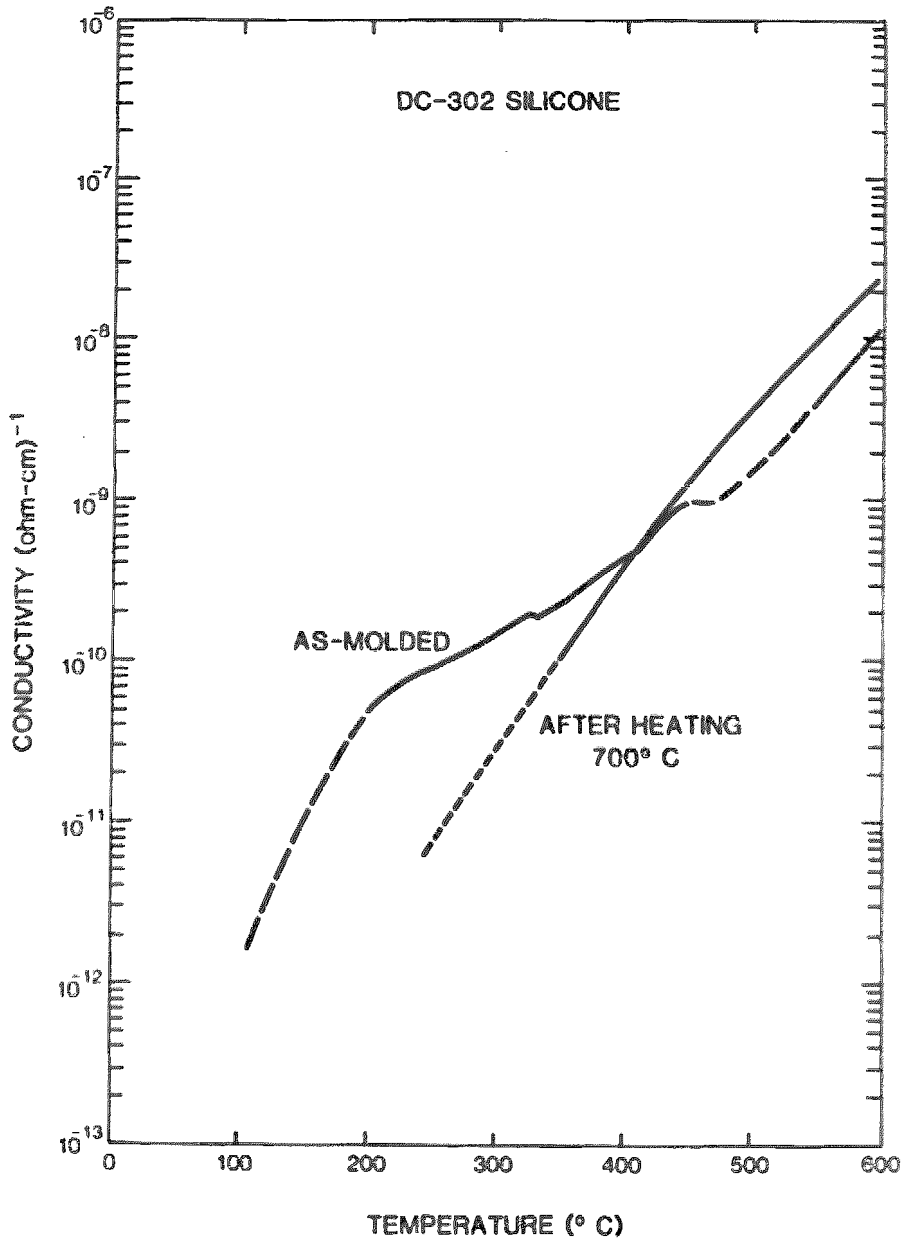


Figure 15. Conductivity temperature dependence for DC-302 silicone material: as-molded and after temperature cycling to 700°C. Dashed portion of curves denotes non-ohmic regions.

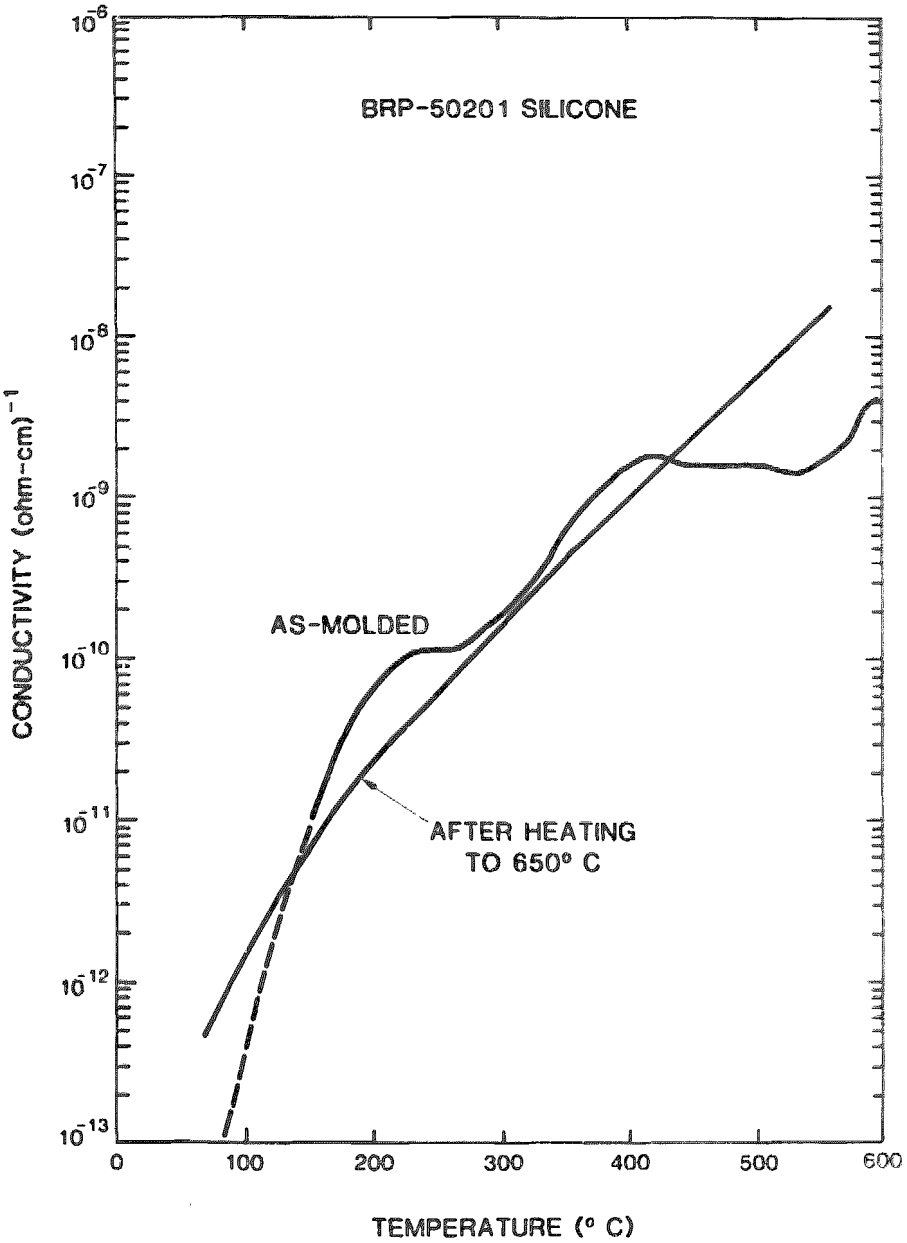


Figure 16. Conductivity temperature dependence of BRP-50201 silicone material: as-molded and after temperature cycling to 650°C. Dashed portion of curves denotes non-ohmic regions.

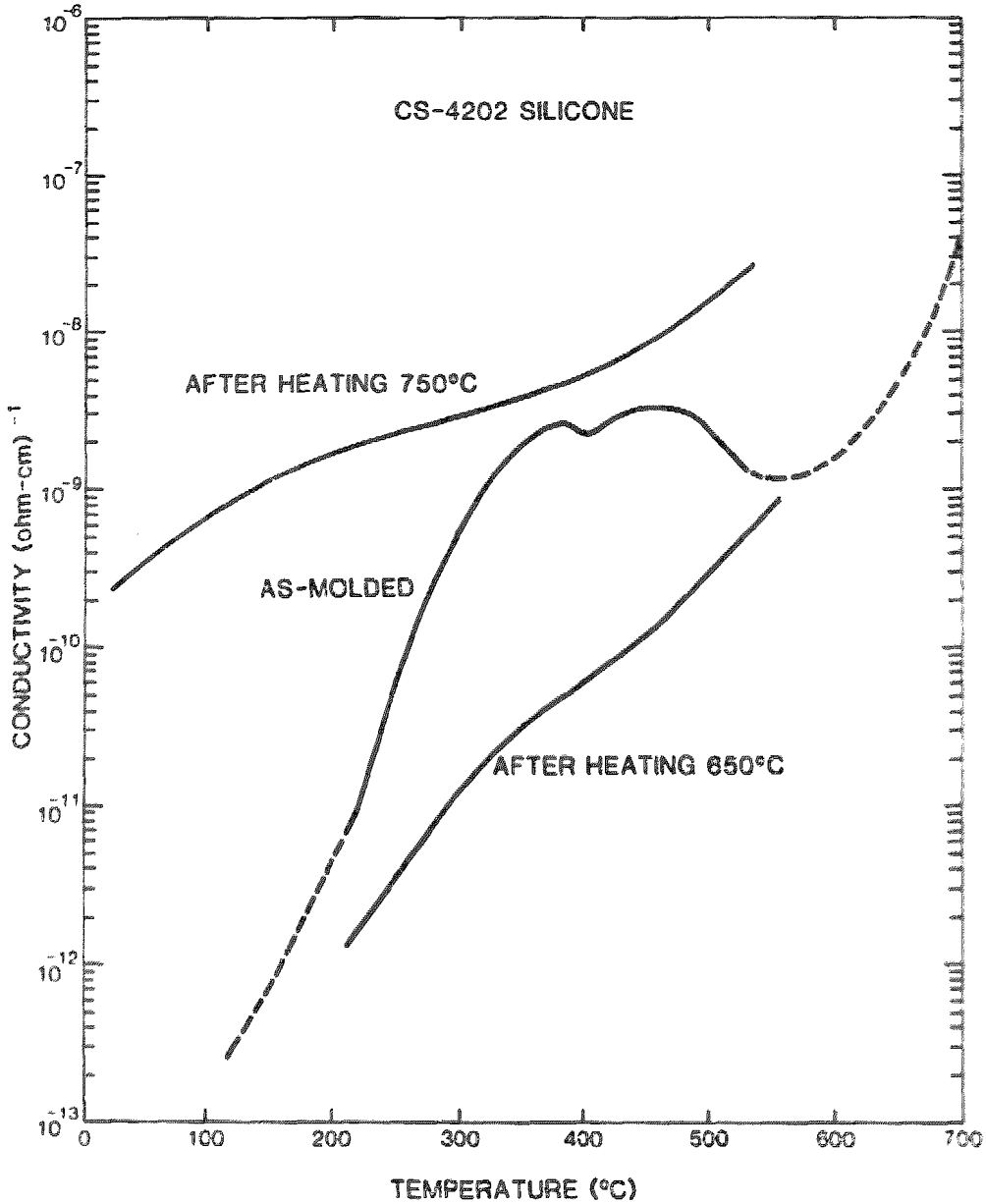


Figure 17. Conductivity temperature dependence for silicone glass composite, CS-4202 material: as-prepared and after temperature cycling to ∿650°C and to ∿750°C. Dashed portion of curves denotes non-ohmic regions.

The conductivity data for DC-302 (Fig. 15) show slight irregularities (peaks) in the as-molded material at temperatures of ∿320°C and ∿450°C. After cycling to ∿700°C the low-temperature conductivity is further reduced and conductivity peaks are eliminated. Although the conduction above ∿450°C is slightly higher than for the as-molded materials, this material remains a fairly good insulator. Further pyrolysis, however, is expected to increase the conductivity.

The conductivity temperature dependence for BRP-50201 in the as-molded state and after temperature cycling to ∿650°C is shown in Fig. 16. In the as-molded material, conductivity peaks appear at ∿250°C and ∿430°C. These peaks are slightly more pronounced than those for the as-molded DC-302. The peaks are removed after temperature cycling to ∿650°C. As for DC-302, this material remains fairly insulating, at least in the low temperature range after cyclying to ∿650°C.

Data for the silicone glass composite, CS-4202, in the as-prepared state and after temperature cycling to ∿650°C and ∿750°C are shown in Fig. 17. In the as-prepared material the value of the conductivity below ∿250°C is the lowest observed for the as-prepared materials. However, there are two conductivity peaks at ∿380°C and ∿450°C. Data above 600°C show conductivity increases with temperature which are similar to those for the phenolics above ∿500°C (see Fig. 18). After cycling to 650°C, the conductivity is significantly reduced indicating that the material in this state is a good insulator. After cycling to ∿750°C, however, the conductivity is

significantly enhanced, with resulting conductivity values much greater than for the as-prepared material. In this state the material is probably carbonized.

The conductivity data for an as-prepared silicone (CS-4202) and phenolic (MXB-71) material are plotted in Fig. 18. Comparing these results helps illustrate some of the differences between these two classes of materials. Specifically, the silicones have lower conductances in the low temperature region (below 300-350°C) and exhibit less pronounced conductivity peaks and deviations from ohmic behavior than the phenolics. Other data have shown that as the material pyrolyzes, with heating to ∿500°C for the phenolics and ∿650°C for the silicones, the conductivity peaks are eliminated and the low temperature conductivity is reduced (i.e., the materials are better insulators). At higher temperatures, however, the material carbonizes and a conductive material is formed. For the silicones this occurs at temperatures 100-150°C higher than for the phenolics.

V. THERMAL RESULTS

Thermal analysis experiments were performed on the same materials used in the electrical conductivity experiments. The thermogravimetric results are presented in terms of weight loss from heating in both air and nitrogen environments and are given in Table 1. The two different techniques employed gave similar results. The differences can be explained by measurement precision or time at temperature.

The observed masses and relative intensities obtained from the mass spectroscopy experiments are given in Appendix B.

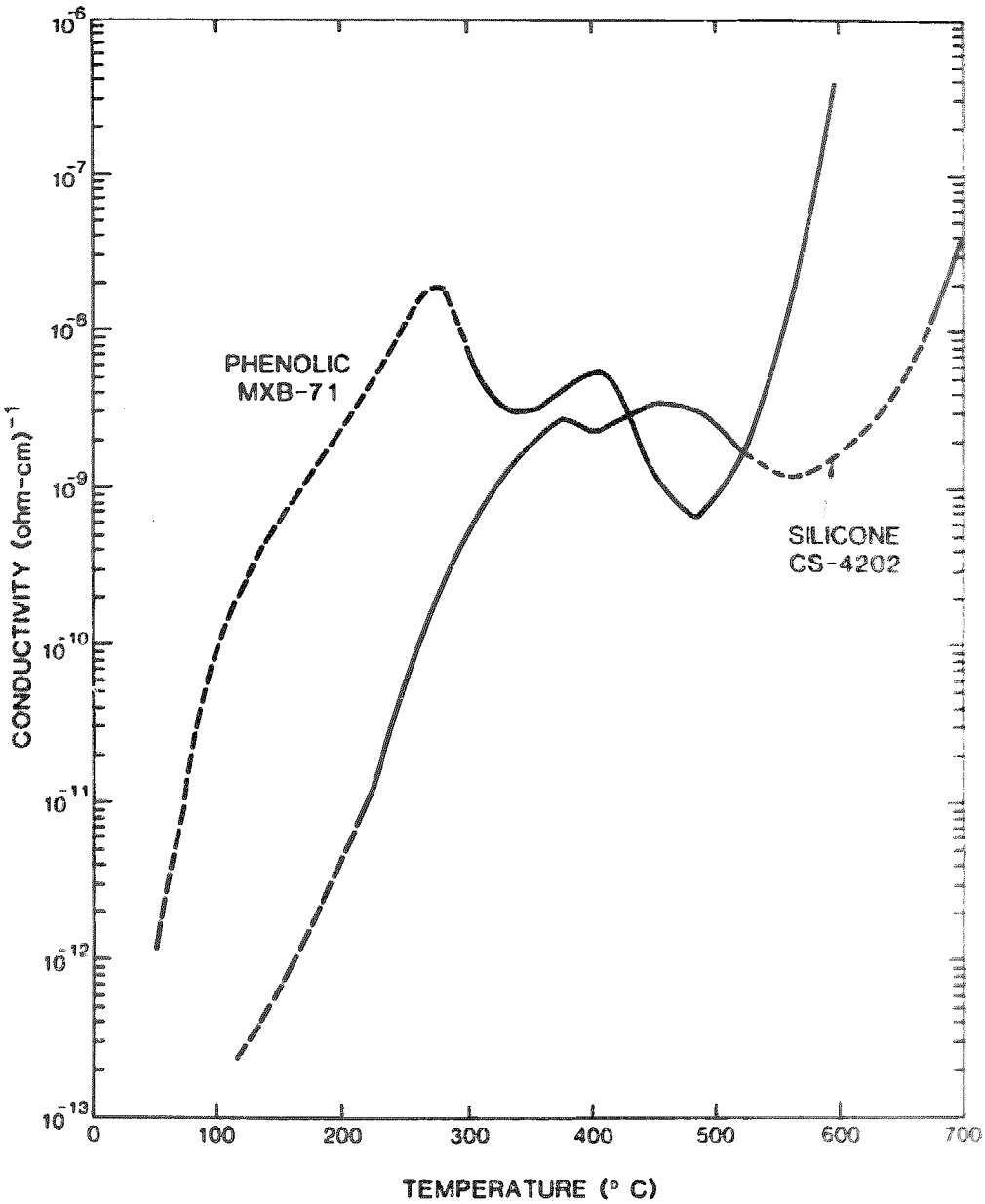


Figure 18. Conductivity temperature dependence for as-prepared phenolic (MXB-71) and silicone (CS-4202) materials. Data are compiled from results in previous figures and show that the silicone materials are better high temperature insulators than the phenolics.

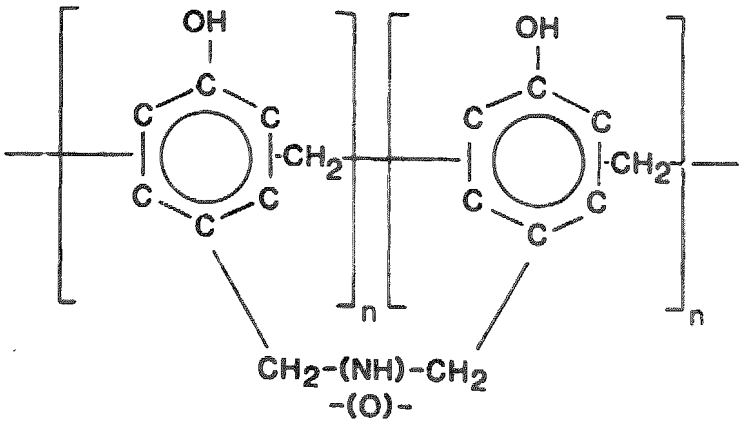
The spectra presented were chosen to illustrate significant changes with temperature. Other spectra were obtained but are not presented. In all of the mass spectra taken, the low temperature spectra ($\lesssim$100°C) are dominated by background peaks particularly at masses 16 to 18, 28 and 32 due to the presence of H_2O, N_2, and O_2 in the spectrometer. The intensities of the peaks at higher temperature are one or two orders of magnitude greater than background.

The weight loss data on the phenolic FM-5135 show significant mass losses above 100°C with values approaching 10-14 wt.% at 600-700°C in nitrogen. A larger loss occurs in air. The mass spectra for FM-5135 (Table B-1, Appendix B) contains both water (masses 16 to 18) and ammonia (masses 14 to 17) below 317°C. Both phenyl groups (mass 77) and possibly some alkylamine fragments (masses around 45) are also present. At higher temperatures the peaks from 30 to 43 could be explained by the presence of methyl and dimethylamine fragments. The peaks at 76-78, 90, 93, and 106 indicate the presence of a cresol. A proposed crosslinked phenolic structure is shown in Table 2. Several possible molecular fragments are given in Tables 2 and 3.

The MXB-71 phenolic also exhibits significant, mass losses above 100°C, with the loss in air greater than that in N_2. The mass spectra of MXB-71 (Table B-2, Appendix B) are dominated by water below 300°C. Also important are both phenyl groups and possibly some alkylamine fragments which can explain the large peaks around 31 and 45. The presence of

TABLE 2

Proposed Phenolic Structure



PROPOSED FRAGMENT		MASS
NH_3	(AMMONIA)	17
CH_3NH_2	(METHYLAMINE)	31
C_3H_2		38
CHNC, C_3H_3		39
CH_2NC		40
CH_2NCH		41
CH_2NCH_3		43
CH_3NHCH_3	(DIMETHYLAMINE)	45
C_6H_5	(PHENYL)	77
C_6H_6	(BENZENE)	78
$C_6H_5CH_2$	(BENZYL)	90
C_6H_5O	(PHENOXY)	93
$C_6H_4CH_2O$	(CRESOL)	106

TABLE 3

Proposed Mass Spectrometer Fragments for FM-5135 Phenolic

Fragment	Relative Intensity		
	78°C	317°C	350°C
Water	35	80	100
Ammonia	100	80	57
Alkylamines	18	13	60
Phenyl	26	4	28
Phenol	-	-	51
Cresol	-	-	100

TABLE 4

Proposed Mass Spectrometer Fragments for MXB-71 Phenolic

	Relative Intensity		
Fragment	110°C	225°C	300°C
Water	100	51	64
Alkylamines	11	100	64
Phenyl	11	2	45
Phenol	-	2	45
Cresol	-	2	100

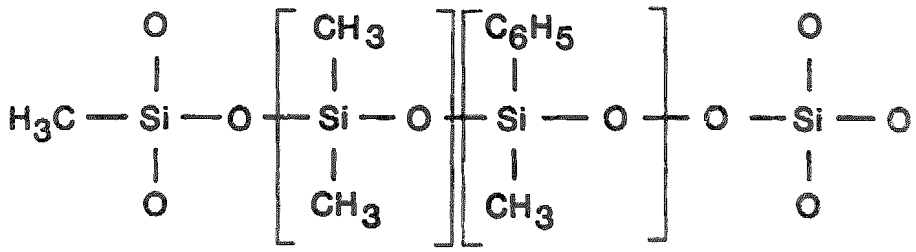
amine groups is also indicated by the intensities from masses 14 to 17. At higher temperatures the presence of peaks at masses 78, 90, 93 and 106 indicate the presence of a cresol (see Tables 2 and 4). Peaks observed at masses 38 and 39 could be explained by fragmentation of the phenyl groups. This was confirmed by taking a spectrum at a reduced electron beam energy of 30 eV. The intensities of the peaks at masses 38 and 39 decreased relative to the intensities of the mass peaks at 106 and 121. This indicates that the species at masses 106 and 121 are parent ions and the ones at 38 and 39 are fragments.

The thermal analysis for silicone-based DC-302 shows very similar behavior in air and flowing N_2 with a slightly larger mass loss in air. The weight loss is less than for the phenolics, especially below ∿500°C. The mass spectrometric results (Table B-3, Appendix B) indicate that below 300°C the major loss is due to water (mass 18).

Since toluene was used as a solvent in these resins it is possible that the mass at 91 is due in part to $C_7H_7^+$ [18]. The mass at 77 is due to a phenyl group. The masses at 91 and 107 are probably due to $SiO_3CH_3^+$ and $SiO_4CH_3^+$ fragments. The peaks from 26 to 29 are an indication of ethyl groups. The peaks around 40 to 45 could be due to methyl-silicone groups and possible SiO^+ at 44. At higher temperatures the phenyl peak dominates the spectra (with phenyl fragments at masses 38, 39, and 49-55). Heavier unidentified molecules with masses up to 153 are also present at high temperatures. Tables 5 and 6 contain a proposed silicone resin structure and several possible fragments.

TABLE 5

Proposed Structure for Silicone Resins



PROPOSED FRAGMENT	MASS
$SiCH_3$	43
SiO	44
C_6H_5	77
SiO_3CH_3	91
SiO_4CH_3	107

TABLE 6

Proposed Mass Spectrometer Fragments for DC-302 Silicone

Fragment	Relative Intensity		
	117°C	230°C	360°C
Water	100	100	15
$SiCH_3$	28	25	84
SiO	31	17	43
Phenyl	11	29	100
Toluene) SiO_3CH_3)	33	41	15
SiO_4CH_3	13	1	1

TABLE 7

Proposed Mass Spectrometer Fragments for BRP-50201 Silicone

Fragment	Relative Intensity	
	110°C	350°C
Water	6	2
$SiCH_3$	11	2
SiO	-	1
Phenyl	3	100
Toluene) SiO_3CH_3)	100	3
SiO_4CH_3	3	1

TABLE 8

Proposed Mass Spectrometer Fragments for CS-4202 Silicone

Fragment	Relative Intensity	
	202°C	375°C
Water	12	1
$SiCH_3$	14	16
SiO	8	-
Phenyl	37	100
Toluene) SiO_3CH_3)	-	3
SiO_4CH_3	-	2

The BRP-50201 silicone material also loses weight very slowly until above 500°C with the largest loss in air between 500-600°C. The mass spectra (Table B-4, Appendix B) at low temperatures show very little water loss (see also Table 7). The largest peak, which occurs at 91, is probably due to $SiO_3CH_3^+$ or the toluene solvent [20]. The high temperature spectra are dominated by the phenyl peak at mass 77 and fragments at 38, 39, and 50-52. The peaks at masses 40 to 44 could be due to methyl-silicone groups. The spectra for BRP-50201 indicate the presence of large molecules with masses up to 280.

The silicone-glass composite, CS-4202, lost only minor amounts of material up to ∿500°C. As with other samples, more material was lost in air than in N_2. The mass spectra (Table B-5, Appendix B) indicates that only minor amounts of water were lost (see also Table 8). The peak at masses 38, 39, 50-52 and 77 indicates the presence of phenyl groups. The peaks for masses 40 to 43 could be due to methyl-silicone entities.

Several comparisons can be made between these materials. The silicone-based materials (aklyl-phenyl-silicone composites) had much better thermal stability than the phenolics (with phenolic and amine based constituents). The silicone-glass composite CS-4202 was the most stable material because it lost the least amount of weight at high temperatures. All the materials lost more weight in air when compared to the weight loss in N_2. The MXB-71 and FM-5135 phenolics were the least stable in air and N_2. The mass spectra of MXB-71 and FM-5135 were similar. Both materials probably contain amines and phenols.

The FM-5135 spectra between 200-300°C were dominated by the loss of NH_3. All of the materials except the BRP-50201 and CS-4202 silicones lost a large amount of water. At the higher temperatures all the spectra indicated the presence of heavier molecules but the spectra for BRP-50201 had molecules with molecular weights that were 100 to 150 mass units larger than any of the other materials.

VI. CORRELATION OF ELECTRICAL AND THERMAL RESULTS

Certain correlations can be made between the electrical and thermal characteristics. For the phenolics, FM-5135 and MXB-71, the mass loss which occurs between 200 and 300°C (water and/or ammonia and amines) correlates with the conductivity peak at 250-270°C (see Figs. 19 and 20). As the temperature is increased and volatilization occurs, the material exhibits a decrease in the conductance (above 250-270°C) which produces a peak in the conductivity temperature dependence. As the temperature is further increased the loss of other mass species occurs (phenolic and amine groups and higher molecular weight species) and results in a second peak in the conductivity at ∿400°C. The position and magnitude of these peaks are expected to vary with heating rate. As the material pyrolyzes with heating to ∿500°C, the conductivity peaks are eliminated and the low temperature conductivity is reduced (i.e., the materials are better insulators). At higher temperatures, however, the material carbonizes and becomes conductive.

The silicone materials DC-302, BRP-50201, and CS-4202 have much better stability and are better insulators than the phenolics.

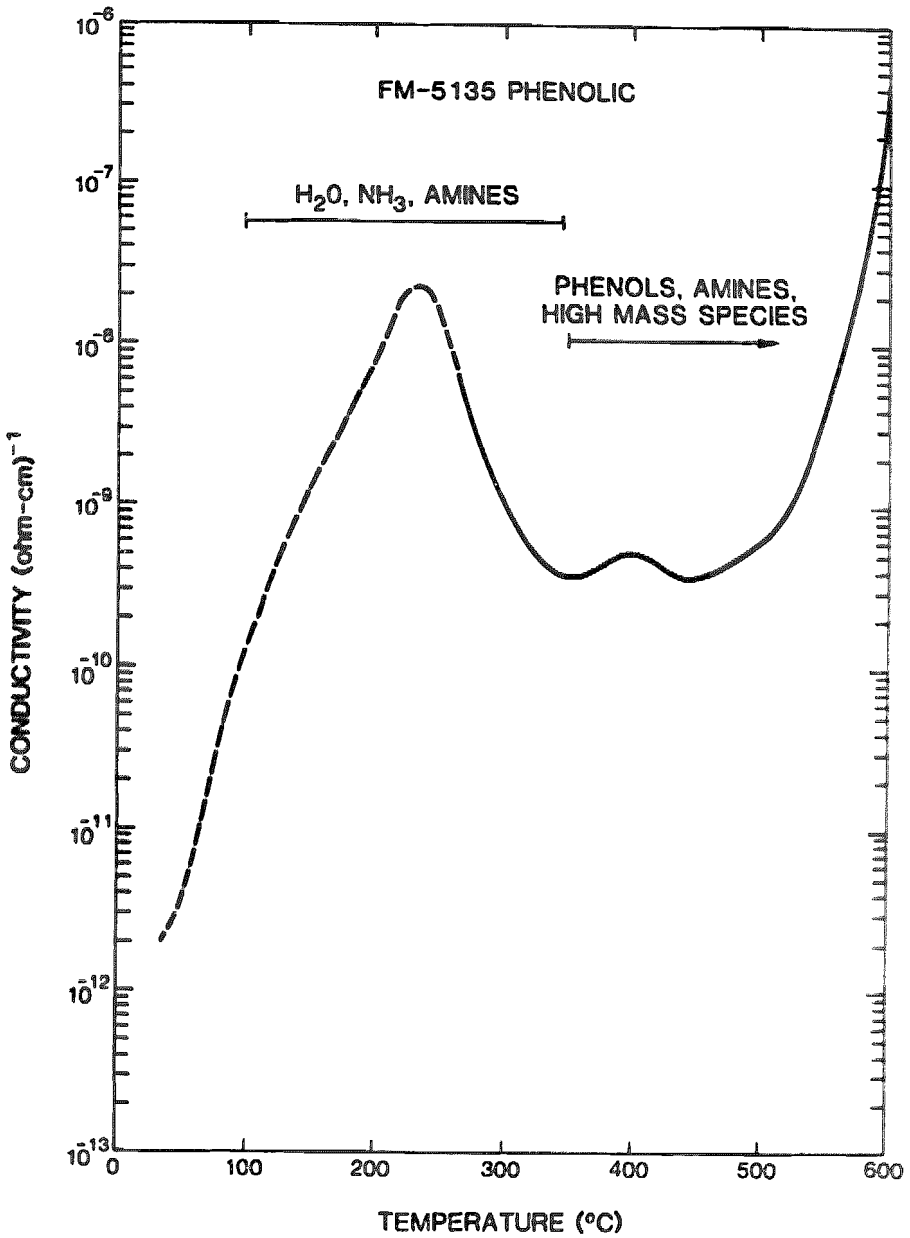


Figure 19. Correlation of temperature dependence of electrical conductivity peaks and major thermal decomposition products for FM-5135 phenolic.

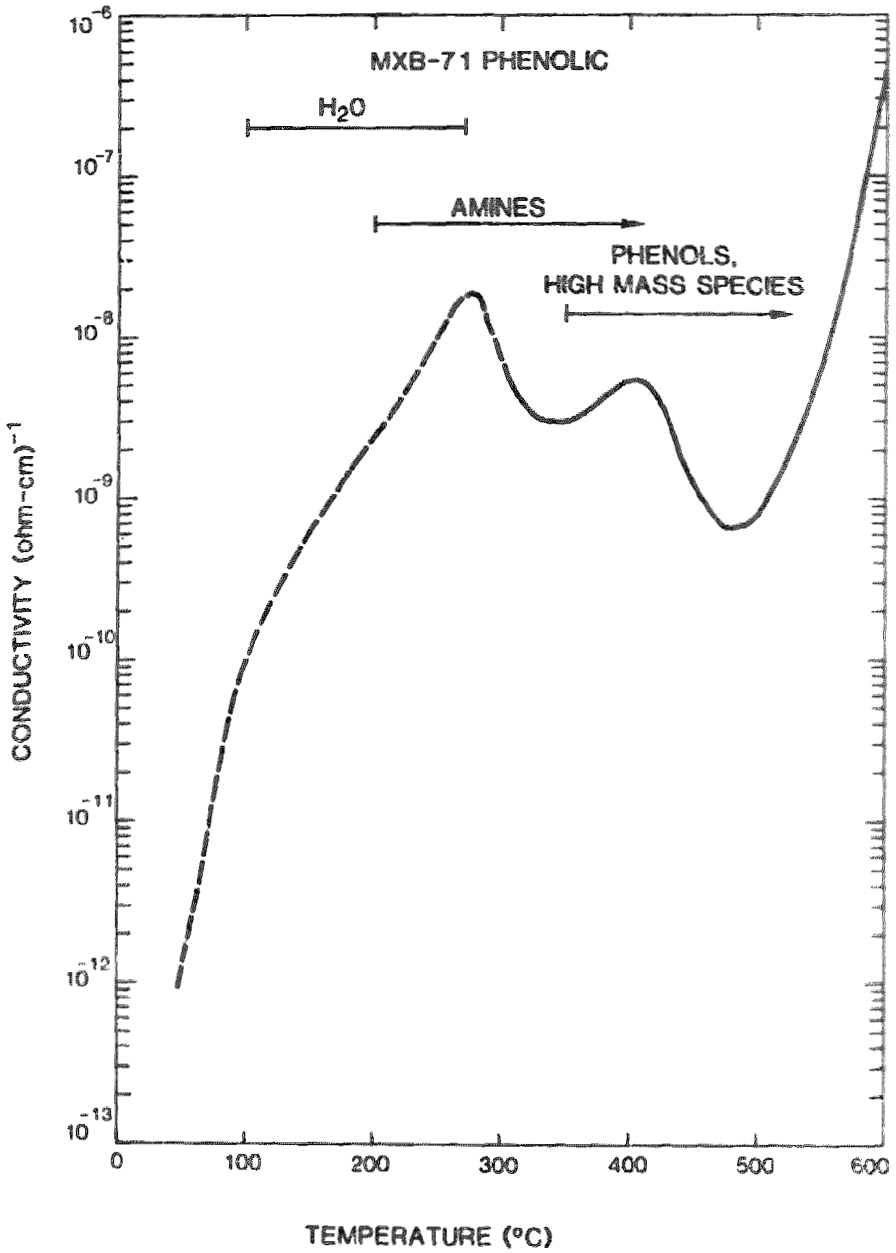


Figure 20. Correlation of temperature dependence of electrical conductivity peaks and major thermal decomposition products for MXB-71 phenolic.

They exhibit less mass loss, have lower conductivities in the low temperature region (below 300-350°C), and exhibit less pronounced conductivity peaks and deviations from ohmic behavior (see Figs. 21-23). The CS-4202 silicone is the most stable of the materials examined and is the best insulator below ~250°C. At the higher temperatures, the silicones lose both alkyl-silicone and phenyl groups as well as high molecular weight polymers. As for the phenolics, the changes in conductivity correlate with outgassing and/or decomposition of the material. Conductivity peaks are eliminated after pyrolysis upon temperature cycling or postcuring. The conductivity decreases with pyrolysis upon heating the silicones to 600-650°C (phenolics to ~500°C). Upon heating to higher temperatures the material carbonizes and the conductivity increases in a manner which is relatively independent of the starting material or previous postcure conditions [5,9].

Deviations from ohmic behavior in all of these materials appear to be most pronounced when the material is not in a state of equilibrium. At low temperature in the insulating state nonohmic behavior may be due to outgassing or other material or contact properties. At higher temperatures, however, the nonohmic behavior appears to be associated with non-equilibrium effects related to decomposition and/or outgassing.

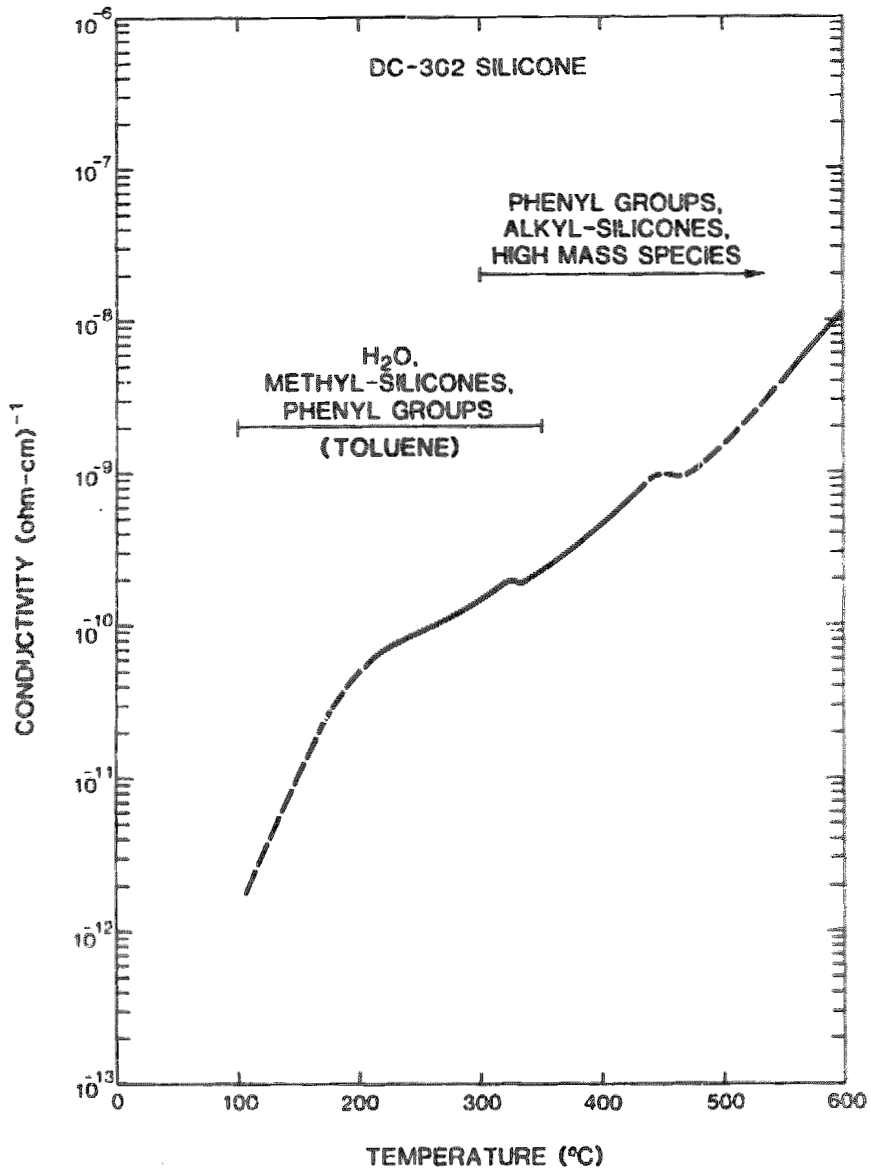


Figure 21. Correlation of temperature dependence of electrical conductivity peaks and major thermal decomposition products for DC-302 silicone material.

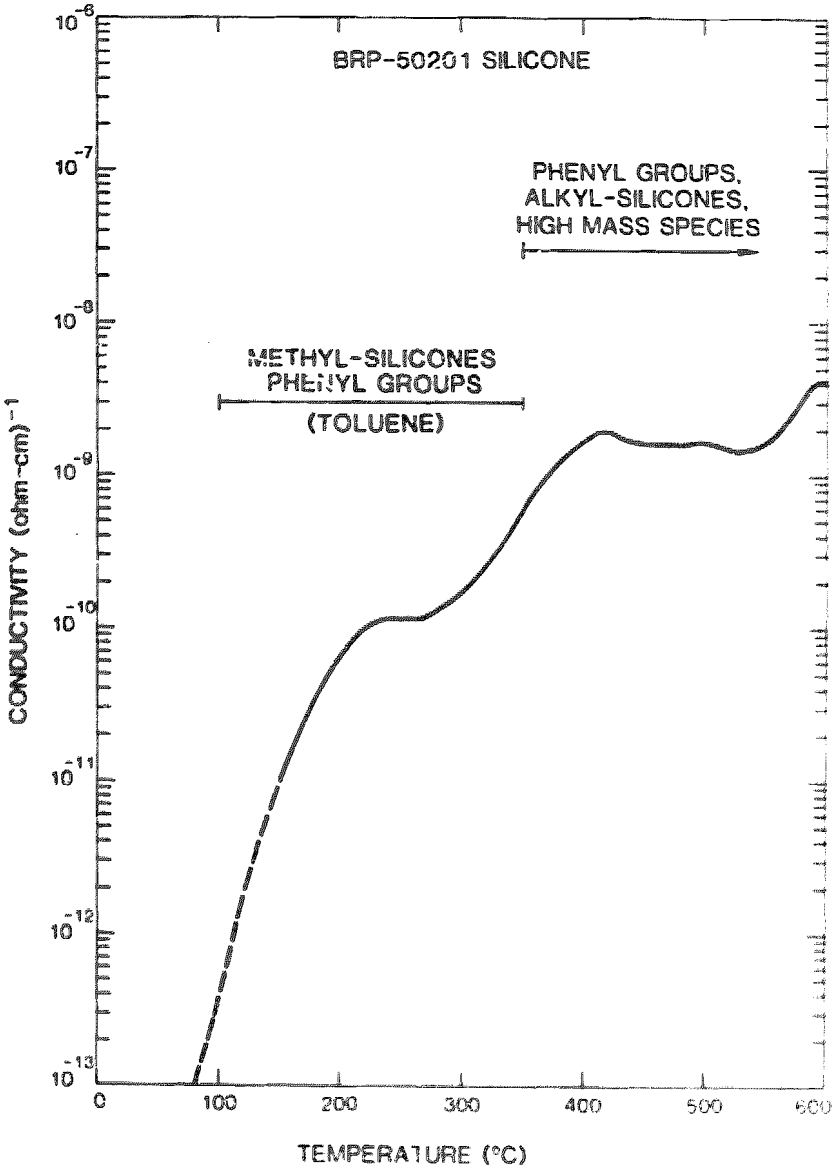


Figure 22. Correlation of temperature dependence of electrical conductivity peaks and major thermal decomposition products for BRP-50201 silicone material.

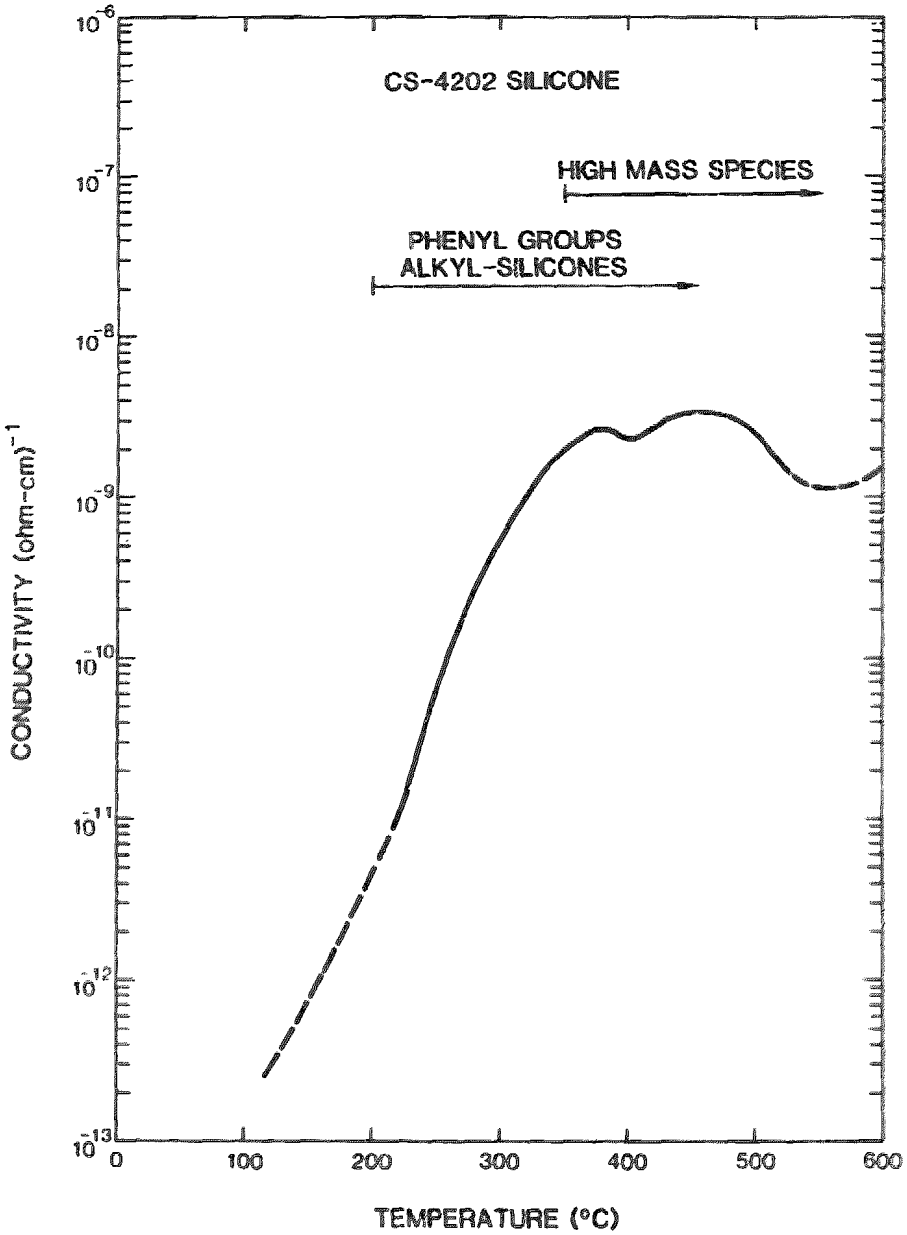


Figure 23. Correlation of electrical conductivity peaks and thermal decomposition products for the silicone-glass composite, CS-4202.

VII. CONCLUSIONS

These experiments show a direct correlation between thermal stability and temperature dependent electrical conduction characteristics in silicone- and phenolic-based materials. Peaks in the conductivity and deviations from ohmic conduction correlate with decomposition and the loss of volatiles from the material. The excellent correlation between thermal and electrical properties suggests that electrical conductivity can be used as a thermal analysis tool in characterizing these materials.

These studies have also shown that the conductance is relatively independent of electrode material, surface conduction, polarization effects, and voltage polarity and magnitude. This suggests that in these experiments a bulk property of the material (conductivity) has been measured. Furthermore, the nature of the conductivity appears to be principally electronic in character especially in the post-cured and carbonized material. It seems reasonable, however, to expect some ionic character to the conductivity especially during outgassing and/or decomposition.

The application of these materials to firesets must include consideration of structural as well as electrical characteristics. Whereas postcuring can improve the electrical insulating characteristics at low temperature, it must be recognized that such postcures may adversely affect the structural properties. All of these factors must be taken into account in selecting materials, processing parameters, and postcure conditions for high temperature applications.

VIII. ACKNOWLEDGEMENTS

O. (Buzz) Milton is credited with having initiated these studies and for having provided the instrumentation and preliminary data. G. L. Cessac, E. A. Salazar and K. R. Hirschbuehler provided the test materials and supplied information and guidance regarding materials properties. The molded materials were obtained from Bendix, Kansas City. E. A. Salazar and G. L. Cessac coordinated the overall effort between Bendix, Kansas City and Sandia National Laboratories in Albuquerque. J. H. Lair and F. F. Francis assisted in all phases of the experimental work. J. C. Lanoue and C. S. Casaus conducted some of the thermogravimetric analysis measurements. C. E. Robinson assisted with some of the thermal measurements. C. Arnold and L. A. Harrah made valuable contributions to understanding the chemistry of decomposition.

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X. APPENDIX A

FOUR-TERMINAL CONDUCTIVITY EXPERIMENTS

Four-terminal conductivity experiments were pursued on the FM-5135 phenolic. The sample and electrode configuration is shown in Fig. A-1. The current electrodes were across the ends of the sample, whereas the voltage electrodes contacted only the surface regions surrounding the specimen near the center of the sample. The samples were cut from the same disks as used for the two-terminal experiments. Current flow was in the plane of the disk, in contrast to the two-terminal experiments where current flow was normal to this plane. In order to avoid electrical leakage through the sample holder, the sample and electrical leads were suspended within the sample chamber, as indicated in Fig. A-2. The sample chamber served as a thermal mass around the specimen in which a thermocouple was mounted. The sample and thermocouple temperature were the same. The sample leads were supported outside the hot zone of the furnace. The circuitry used for the conductivity measurements is shown in Fig. A-3. For these experiments the voltage across the sample was 300 V.

During the first cycle to high temperature the voltage drop across the voltage electrodes had an unexpectedly low value and was nonohmic. However, the current-voltage characteristics obtained using the contacts at the ends of the specimen were very similar to those for the two-terminal experiments of Fig. 1a in the text. An explanation for this behavior can be given in terms of differences in surface and

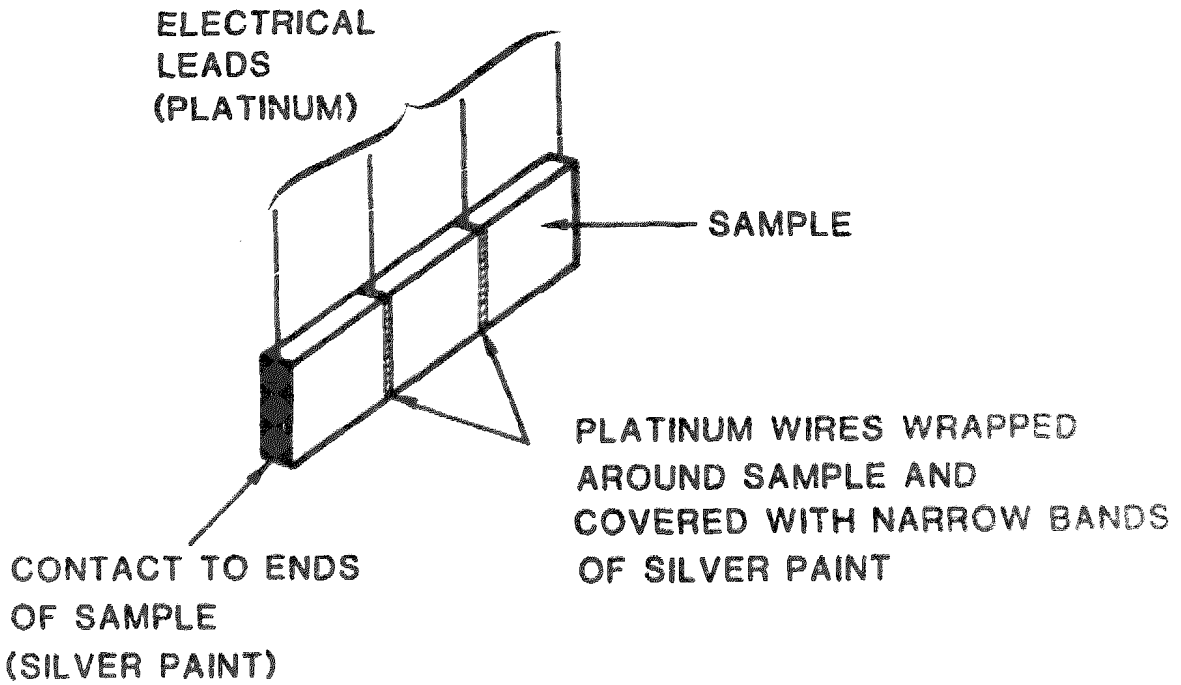

Figure A-1. Four-terminal electrical conductivity sample and electrode configuration.

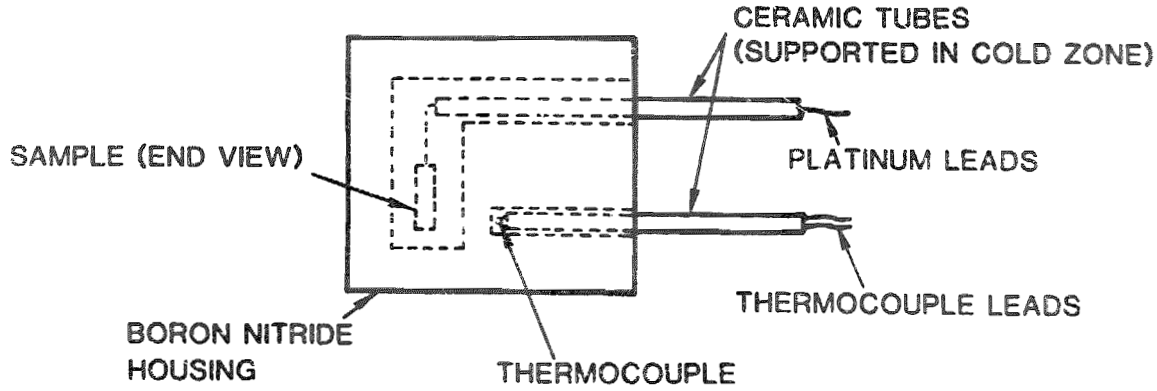

Figure A-2. Sample holder configuration for four-terminal electrical conductivity experiments.

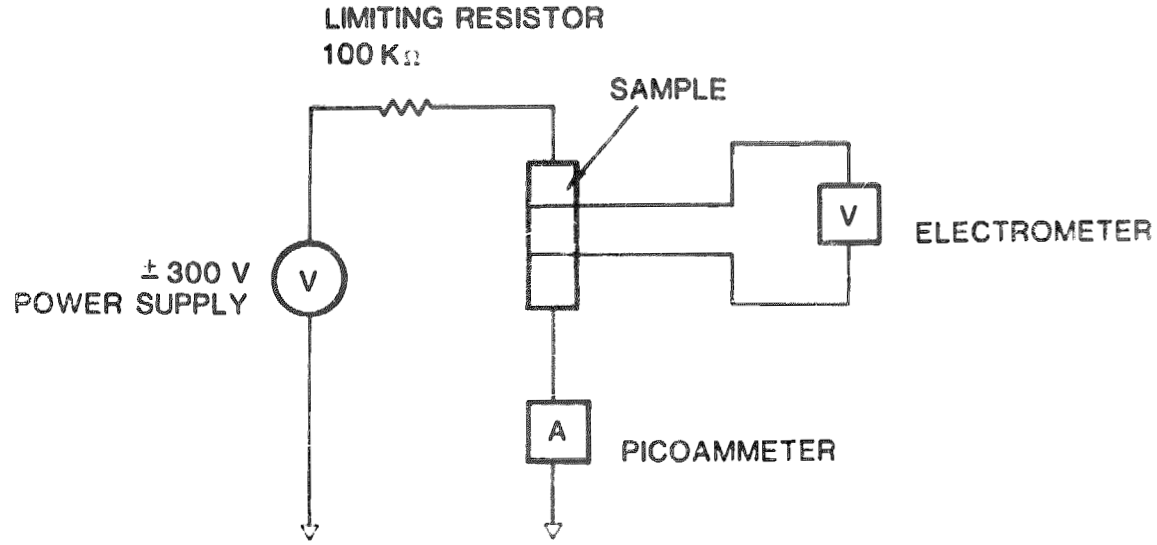

Figure A-3. Circuit schematic for four-terminal electrical conductivity measurements.

bulk conduction. Specifically, during decomposition the surface may become resistive at a more rapid rate than the bulk material. In this situation, the voltage contacts to the surface do not give an accurate measurement of a bulk property. This suggestion is consistent with results obtained upon postcuring where reductions in conduction are observed after volatile constituents are lost from the material. After the sample pyrolyzes upon cycling to high temperatures (∿600°C), the voltage measured using the voltage electrodes on the surface was as expected for a sample where the surface and the bulk of the material were in equilibrium. Although more experiments need to be conducted in order to fully understand these effects, it does appear that during decomposition that the surface of the material may become resistive more rapidly than that of the bulk material. This would imply that the electrode area of coverage and positioning on the sample is important in these experiments. In order to obtain the results observed in the two-terminal experiments of Fig. 1a, the loss of volatiles from the material under the electrodes during decomposition probably occurred at a rate slower than that from material in an unelectroded area. Thus, the electrode configuration may influence the rate dependence of the effects observed in these experiments.

XI. APPENDIX B

MASS SPECTRA RESULTS

Observed masses and relative intensities as a function of temperature from mass spectrometer experiments on phenolic and silicone materials.

Table B-1: FM-5135 Phenolic

78°C		317°C		350°C	
Mass	Int	Mass	Int	Mass	Int
12	8	12	3	12	9
13	2	13	1	13	2
14	100	14	6	14	5
15	9	15	11	15	14
16	39	16	71	16	33
17	11	17	100	17	82
18	35	18	80	18	100+
26	7	25	1	25	2
27	14	26	2	26	11
28	100+	27	7	27	30
32	63	28	31	28	88
37	4	29	8	29	28
38	14	31	7	30	4
39	11	32	2	37	12
40	18	37	1	38	37
41	8	38	3	39	16
42	11	39	1	40	23
43	18	41	3	41	12
44	2	42	3	42	21
49	6	43	5	43	39
50	9	44	7	49	14
51	6	45	12	50	21
52	4	49	1	51	14
54	9	50	1	52	16
55	8	51	1	53	7
56	7	54	1	54	12
57	4	55	1	55	9
62	2	56	1	56	11
66	3	77	4	62	14
69	5			64	18
73	2			65	21
77	26			76	28
				77	28
				78	18
				90	25
				93	51
				106	100
				121	44
				134	5

Table B-2: MXB-71 Phenolic

110°C		225°C		300°C	
Mass	Int	Mass	Int	Mass	Int
12	2	12	5	12	9
14	15	13	5	13	5
15	2	14	18	14	9
16	15	15	39	15	36
17	72	16	21	16	23
18	100+	17	89	17	32
26	2	18	100+	18	64
27	4	24	3	19	9
28	100	25	5	25	5
31	2	26	29	26	27
32	19	27	61	27	73
38	2	28	76	28	55
39	6	29	58	29	36
40	4	30	16	30	18
41	6	31	95	31	9
42	4	32	18	32	5
43	4	37	8	36	9
44	9	38	11	37	27
45	6	39	34	38	100
50	2	40	16	39	36
51	4	41	39	40	27
52	2	42	39	41	18
55	4	43	58	42	18
56	2	44	24	43	18
57	2	45	100	49	27
77	11	46	37	50	55
		57	5	51	27
		58	5	52	36
		59	3	53	18
		65	3	54	27
		77	3	62	27
		93	3	64	27
		106	3	65	36
				74	9
				76	45
				78	36
				90	27
				93	45
				103	9
				106	100
				112	9
				121	45
				135	9

Table B-3: DC-302 Silicone

117°C		230°C		360°C	
Mass	Int	Mass	Int	Mass	Int
12	8	12	3	12	2
13	1	13	1	13	1
14	51	14	9	14	5
15	6	15	12	15	17
16	27	16	10	16	7
17	47	17	28	17	5
18	100	18	100	18	15
25	1	26	6	19	1
26	5	27	18	25	2
27	17	28	59	26	24
28	100+	29	15	27	49
32	45	30	3	28	43
37	2	31	5	29	35
38	4	32	7	31	2
39	19	37	3	32	1
40	13	38	7	37	16
41	23	39	22	38	30
42	15	40	8	39	80
43	28	41	14	41	85
44	31	42	11	42	56
45	17	43	25	43	94
50	5	44	17	44	43
51	9	45	85	45	23
52	5	50	8	48	10
53	5	51	12	49	68
54	4	52	8	50	99
55	17	55	1	51	72
56	12	57	1	52	17
57	17	59	1	53	43
70	10	63	7	54	45
77	11	64	7	55	47
91	33	77	20	62	22
107	13	91	41	67	29
		107	1	69	22
				70	30
				71	27
				73	23
				77	100
				78	15
				79	16
				80	18
				81	20
				82	20
				91	15
				96	2
				103	3
				114	1
				117	2
				127	2
				141	1
				153	2

Table B-4: BRP-50201 Silicone

110°C		350°C	
Mass	Int	Mass	Int
12	1	12	1
14	4	13	1
15	1	14	1
16	2	15	7
17	2	16	3
18	6	17	1
26	1	18	2
27	6	25	1
28	36	26	10
29	8	27	11
30	1	28	9
31	1	29	3
32	5	30	1
37	1	31	1
38	3	32	1
39	11	36	1
40	5	37	7
41	11	38	13
42	5	39	27
43	11	40	8
45	3	41	4
46	2	42	3
50	4	43	2
51	7	44	1
52	3	49	5
55	8	50	32
56	8	51	40
57	11	52	32
60	1	54	3
61	3	55	2
62	7	56	1
64	9	62	9
65	3	66	3
68	5	73	9
69	5	74	7
70	6	77	100
77	3	91	3
82	4	95	1
84	4	104	1.5
88	3	116	1
91	100	127	0.5
96	4	131	0.5
104	3	140	0.5
110	3	153	0.5
118	2	176	0.5
125	2	190	1
132	1	198	0.5
139	2	206	3
141	1	269	0.5
151	1	280	1
153	1		
155	2		
192	1		
206	1		
250	1		

Table B-5: CS-4202 Silicone

202°C	
Mass	Int
12	3
13	1
14	12
15	3
16	7
17	5
18	12
26	3
27	10
28	100
29	19
32	14
37	2
38	3
39	12
40	7
41	19
42	10
43	14
44	8
45	8
50	5
51	7
52	5
53	3
55	14
56	8
57	10
62	2
64	1
67	3
69	7
70	5
71	3
72	2
73	2
77	37

375°C	
Mass	Int
12	1
13	1
14	2
15	6
16	2
17	0.5
18	1
25	1
26	10
27	14
28	13
29	2
36	1
37	9
38	15
39	36
40	11
41	21
42	16
49	5
50	34
51	46
52	36
62	10
66	8
73	10
77	100
81	3
90	3
103	2
114	1
117	1
128	1
142	0.5
154	1
168	0.5
178	0.5
186	1

Distribution:

B. L. Sadler, D814-1, BKC
C. H. Smith, D814-2, BKC

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2360 T. L. Workman
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2362 G. L. Cessac
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5815 F. F. Francis
8110 J. Barham
8120 W. E. Alzheimer
8150 J. L. Wirth
8160 R. D. Cozine
8310 D. M. Schuster
8320 R. L. Rinne

3141 L. Erickson (5)
3151 W. L. Garner (3)
3154-3 C. Dalin (25)
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