UNCLASSIFIED

AD NUMBER
AD873310
LIMITATION CHANGES
TO: **Approved for public release; distribution is unlimited.**
FROM: **Distribution authorized to DoD only; Administrative/Operational Use; AUG 1970. Other requests shall be referred to Air Force Materials Lab., Wright-Patterson AFB, OH 45433.**
AUTHORITY
AFML ltr 7 Dec 1972

THIS PAGE IS UNCLASSIFIED

AD873310

AFML-TR-69-111
Part II

HIGH TEMPERATURE ELECTRICAL WIRE COATINGS

Norman Bilow
Kenneth L. Rose

Hughes Aircraft Company

TECHNICAL REPORT

August 1970

Each transmittal of this document outside of the Department of Defense must have prior approval of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright Patterson AFB, Ohio 45433.

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made ONLY with specific prior approval of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

NOTICE

When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

Copies of this report should not be returned unless return is required by security considerations, contractual obligations, or notice on a specific document.

AFML-TR-69-111
Part II

HIGH TEMPERATURE ELECTRICAL WIRE COATINGS

Norman Bilow
Kenneth L. Rose

Hughes Aircraft Company

TECHNICAL REPORT

August 1970

Each transmittal of this document outside of the Department of Defense must have prior approval of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright Patterson AFB, Ohio 45433.

This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made ONLY with specific prior approval of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio 45433.

AIR FORCE MATERIALS LABORATORY
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO

FOREWORD

This report was prepared by Hughes Aircraft Company, Culver City, California 90230, under Air Force Contract No. F33615-68-C-1322. The contract was initiated under Project No. 7340 "Nonmetallic and Composite Materials," Task No. 734007 "Coatings for Energy Utilization, Control and Protective Functions." The program was administered under the direction of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division, Air Force Materials Laboratory, with Dr. William Lehn serving as Project Engineer.

The principal investigator on the program was Dr. Norman Bilow, Head of the Polymer and Physical Chemistry Section of the Materials Technology Department. Technical assistance was received from Mr. Kenneth L. Rose and Mr. Arturo A. Castillo. Mr. Bert Gerpheide served as a consultant on electrical insulation, and Dr. R. I. Akawie and Dr. L. J. Miller consulted on Polymer Chemistry.

This is the second annual summary report on the contract and describes work conducted between 1 February 1969 and 30 April 1970. It was submitted by the authors in May 1970.

All experimental wire wrapping studies were conducted at Microdot Corporation under the direction of Mr. Richard Holzhauer. Assisting Mr. Holzhauer were Mr. Charles Milner and Mr. O. Addington.

This technical report has been reviewed and is approved.

WARREN P. JOHNSON, Chief
Elastomers and Coatings Branch
Nonmetallic Materials Division
Air Force Materials Laboratory

ABSTRACT

The purpose of this program was to develop high temperature resistant electrical insulation coatings for aerospace or hook-up wire for use on advanced high speed Air Force systems. Perfluoroalkylene-triazine, polybenzothiazole, polyterphenyleneoxide, silicone XR-4083, P13N polyimide, polyimidazoquinazoline and two types of "pyrrone" polymers were investigated as potential electrical wire coatings. All of these materials have good electrical properties at ambient temperature although zinc oxide filled polybenzothiazole has a high dissipation factor and the insulation resistance of perfluoroalkylenetriazine drops somewhat more rapidly with increasing temperature than several of the other polymers. Polyterphenyleneoxide had exceptionally good electrical properties but was too brittle to use as a wire insulation. Polyimidazoquinazoline was also too brittle. All of these polymers were evaluated as adhesives for Kapton polyimide tape in an effort to utilize them in wrapped insulation. In this respect the perfluoroalkylenetriazine, polyterphenyleneoxide and silicone XR-4083 were especially poor, particularly at temperatures over 260°C (500°F). Materials which failed to provide adequate adhesion for Kapton polyimide tape were evaluated as overcoats for "pyrrone" bonded Kapton insulation. A large portion of the research effort was devoted to the study of HR-100 "pyrrone" and P13N polymide because these materials provided excellent adhesion for Kapton as well as excellent long term thermal stability and good dielectric properties. It was determined that "pyrrone" (HR-100)-Kapton or P13N-Kapton combinations can provide useful wrapped insulations for multifilament connector wire which is to see temperatures of up to 371°C (700°F).

CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS (Continued)

LIST OF ILLUSTRATIONS (Continued)

LIST OF ILLUSTRATIONS (Continued)

LIST OF TABLES

LIST OF TABLES (Continued)

SECTION I
INTRODUCTION

The operation of aircraft at speeds in excess of Mach 3 has resulted in markedly increased temperatures to which aircraft hook-up wires are exposed. Temperatures around 550°F are commonly experienced on contemporary aircraft, while temperatures up to 800°F and possibly higher will be experienced in future hypersonic military aircraft. These temperatures are considerably in excess of those that existing aircraft hook-up wires can safely withstand under sustained conditions.

The high temperature hook-up wire currently specified for military aircraft has a thin (3-mil maximum), tough insulation and is procured against specification MIL-W-81381 "Wire, Electric, Polyimide Insulated, Copper and Copper Alloy." The insulation on this wire is constructed from tapes that are composites of polyimide and FEP Teflon fluorocarbon polymer. This wire has a continuous service temperature rating of only 392°F. When subjected to temperatures slightly above 400°F (204°C), the stresses generated exceed the yield strength of the FEP fluorocarbon sealant. As a result the insulation loses its sealant properties, and a general reduction in mechanical properties is observed. Thus, one way to improve the thermal threshold of hook-up wire would be to utilize a sealant that could be substituted for the FEP Teflon which has a service temperature equal to or better than that of the polyimide substrate film alone.

Many high temperature polymers that warranted evaluation as sealants in this program have been under development at laboratories throughout the country. Polyimides, silicone carborane (Dexsil) elastomers, P13N polyimide, polyphenylenes, perfluoroalkylenetriazines, "pyrrones," BBB resins, polybenzothiazole, poly(terphenylene oxide), and polyimidazoquinazoline were selected as primary candidate sealants because of the high probability that they would meet the high temperature insulation requirements. Furthermore, several of these polymers were available in research quantities and consequently their evaluation was feasible within the scope of this program. Other rarer prospective polymers also exist and warrant screening in subsequent studies.

PRECEDING PAGE BLANK-NOT FILMED

SECTION II
POLYMER EVALUATION

1. PERFLUOROALKYLENETRIAZINE

Perfluoroalkylenetriazine was evaluated as an insulation both by itself and in a perfluoroalkylenetriazine overcoat for "pyrrone" (HR-100)-Kapton coated wire. The perfluoroalkylenetriazine overcoated "pyrrone"-Kapton overcoating is discussed on pages 15, 18 - 20, and 87 - 96.

For this study a solution of terephthalonitrile N, N'-dioxide (0.26 gram) and perfluoroalkylenetriazine (20 grams) in a 1:1 mixture of bis(trifluoromethyl) benzene and ethyl acetate was prepared. The solution was used to coat a 20 gage tinned copper wire. The wires were then air dried and cured for 4 hours at 50°C. Insulation thickness was 5-6 mils. The insulation resistance* at 500 Vdc was measured between 75°F and 600°F and the results are shown in Table I. This data is graphically illustrated in Figure 1. From this

Table I. Insulation Resistance of Perfluoroalkylenetriazine

Temperature		Insulation Resistance, Ohms*	
°F	°C	Unsoaked Wire	After 16 Hour Soak in Wetted Water
75**	23	--	2×10^{9}
75	23	8×10^{12}	3×10^{12}
200	93	3×10^{9}	Shorted
300	149	5 x 10[illegible]	
400	204	3×10^{8}	
500	260	3×10^{8}	
600	316	Shorted	

*30 inch wires were used throughout this program.
**Sample tested in water.

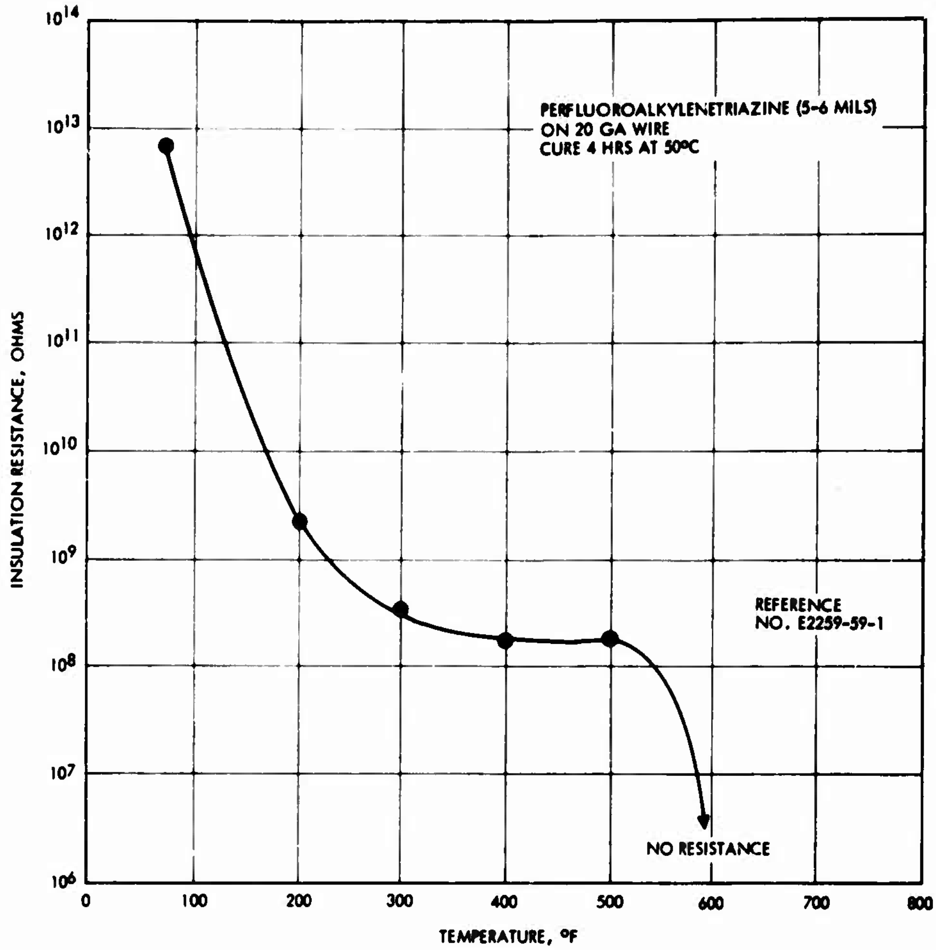


Figure 1. Insulation resistance of perfluoroalkylenetriazine insulated wire

graph it is quite apparent that, although perfluoroalkylenetriazine has excellent insulation resistance at ambient temperature, its much lower insulation resistance at 93°C (200°F) or above, and its total failure between 500° and 600°F indicated that this polymer was unsatisfactory insulation for use at temperatures above 500-600°F. Subsequent tests, however, showed that total failure need not occur below 600°F.

Before it could be rejected, however, it was necessary to ascertain if the cure conditions were really adequate. Consequently, several similar wires containing 1.3 percent curing agent were prepared with a 5 mil insulation thickness and these were cured as follows:

16 hours at 50°C in air

16 hours at 100°C in argon

16 hours at 150°C in argon

A second perfluoroalkylenetriazine solution was prepared containing 20 grams of the perfluoroalkylenetriazine and 0.52 gram (2.6 percent) terephthalonitrile N, N'-dioxide. Coated wires were again prepared having 5 mils of insulation and these were cured under the same three cure conditions. Insulation resistance measurements were then made over the temperature range of 23-427°C (75-800°F) and these data are presented in Table II.

It was quite evident from these subsequent tests that the 4 hour 50°C cure initially used was not sufficient to provide cured polymer with optimum electrical properties. The perfluoroalkylenetriazine containing 1.3 percent terephthalonitrile N, N'-dioxide shorted between 204 -260°C (400 -500°F) when it had the 16 hour cure at 50°C (122°F) but survived to 260 -316°C (500 -600°F) with the 16 hour 150°C (302°F) cure.

A somewhat similar situation existed with the triazines which contained 2.6 percent terephthalonitrile N, N-dioxide as the curing agent. In this case the polymer cured for 16 hours at 50°C (122°F) survived to 260 -316°C (500 -600°F) and both polymers cured for 16 hours at 100°C (212°F) or 16 hours at 150°C (302°F) survived to over 700°F.

Thus, it is apparent that as far as insulation resistance is concerned the perfluoroalkylenetriazine can withstand temperatures to 371°C (700°F), at which point its insulation resistance was about 10^9 ohms*, for short time intervals.

Polymer with 2.6 percent curing agent exhibited optimum properties when cured at 100°C (212°F) for 16 hours whereas that with 1.3 percent curing agent required a cure at 150°C (302°F) for 16 hours.

---

*30 inch long wires.

Table II. Insulation Resistance of Perfluoroalkylenetriazine Insulated Wire

Temperature		Insulation Resistance*, Ohms		
°F	°C	16 Hour 50°C Cure	16 Hour 100°C Cure	16 Hour 150°C Cure
1.3 percent Terephthalonitrile N,N'-Dioxide				
75	24	1×10^{13}	1×10^{13}	2×10^{13}
200	93	2×10^{10}	3×10^{10}	5×10^{10}
300	149	2×10^{9}	2×10^{9}	2×10^{9}
400	204	1×10^{8}	2×10^{8}	2×10^{8}
500	260	Shorted	1×10^{8}	8×10^{7}
600	316		Shorted	7×10^{7}
700	371			1×10^{9}
800	427			Shorted
2.6 percent Terephthalonitrile N,N'-Dioxide				
75	24	8×10^{12}	1×10^{13}	2×10^{13}
200	93	1×10^{10}	2×10^{10}	7×10^{10}
300	149	8×10^{8}	1×10^{9}	3×10^{9}
400	204	2×10^{8}	3×10^{8}	3×10^{8}
500	260	1×10^{8}	1×10^{8}	1×10^{8}
600	316	Shorted	6×10^{7}	8×10^{7}
700	371		8×10^{8}	8×10^{8}
800	427		Shorted	Shorted

*30 inch lengths.

Results of these insulation resistance measurements are illustrated graphically in Figures 2 and 3. All three of the wires which survived 600°F showed an increase in insulation resistance before failing. No explanation for this phenomenon is obvious although it may be an effect of swelling.

Moisture resistance of the perfluoroalkylenetriazine was appraised by investigating the effect of a 95 percent relative humidity 28 day 85°C (185°F) exposure on perfluoroalkylenetriazine overcoated "pyrrone" (HR-100)-Kapton wires. Results of these tests are discussed in the section on wire evaluation (pages 87 - 95).

Thermal soak tests were also conducted on samples of perfluoroalkylenetriazine overcoated "pyrrone"(HR-100)-Kapton insulated wires. For these tests the wires were tightly wrapped in aluminum foil and placed in an oven and insulation resistance measurements were made periodically. The soak temperature was 260°C (500°F) and the time was 120 hours. Results of the test are shown in Table III.

Table III. Insulation Resistance of Perfluoroalkylenetriazine Overcoated "Pyrrone"-Kapton Insulated Wire as a Function of Thermal Aging

Temperature		Time, hours	Insulation Resistance, Ohms 30 inch lengths Perfluoroalkylenetriazine Overcoated "Pyrrone"-Kapton #56-3
°C	°F		
24	75	--	2×10^{12}
260	500	4	1×10^{10}
260	500	24	1×10^{10}
260	500	76	2×10^{10}
260	500	120	3×10^{10}
24	75*	--	2×10^{13}

*The second ambient temperature test was run upon completion of the thermal soak.

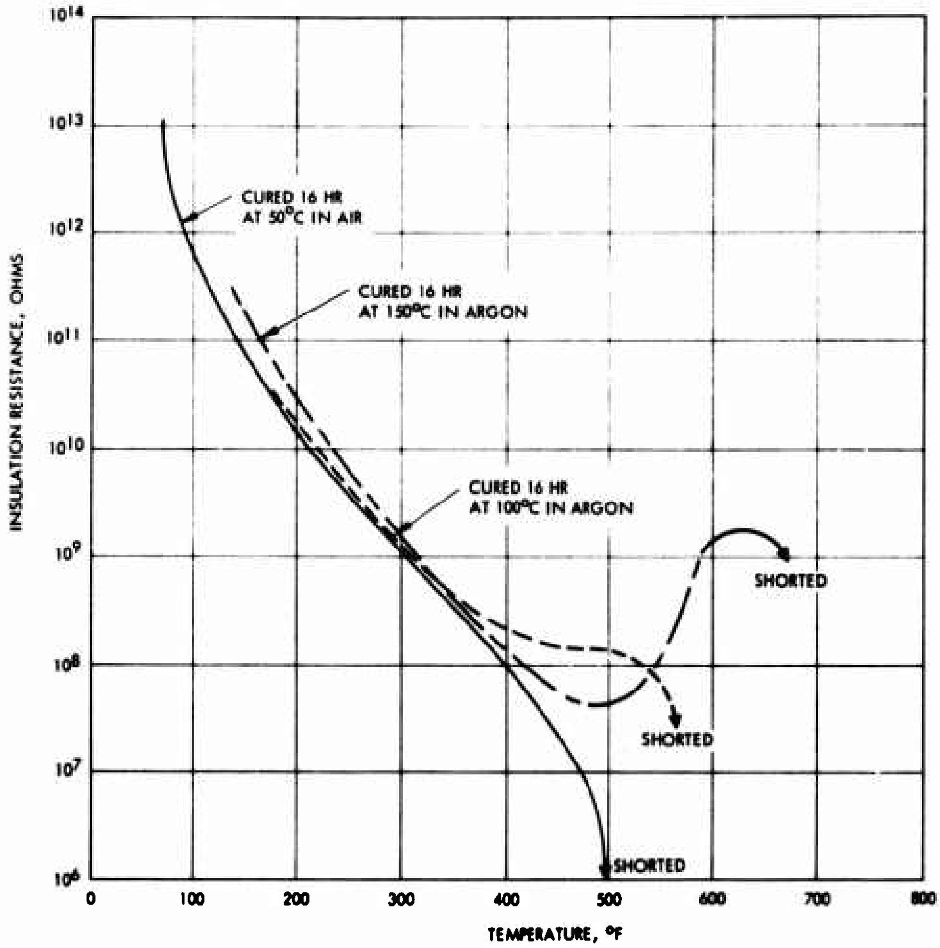


Figure 2. Insulation resistance measurements on perfluoroalkylenetriazine coated wires — 1.3 percent terephthalonitrile N, N'-dioxide

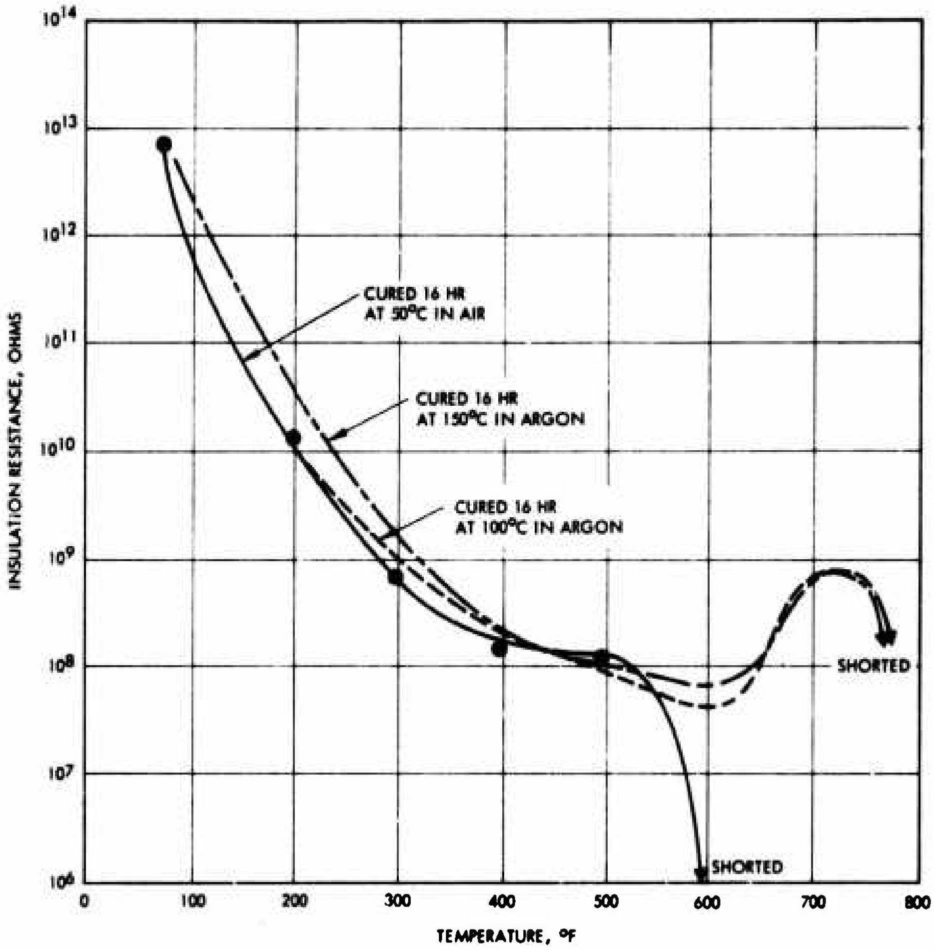


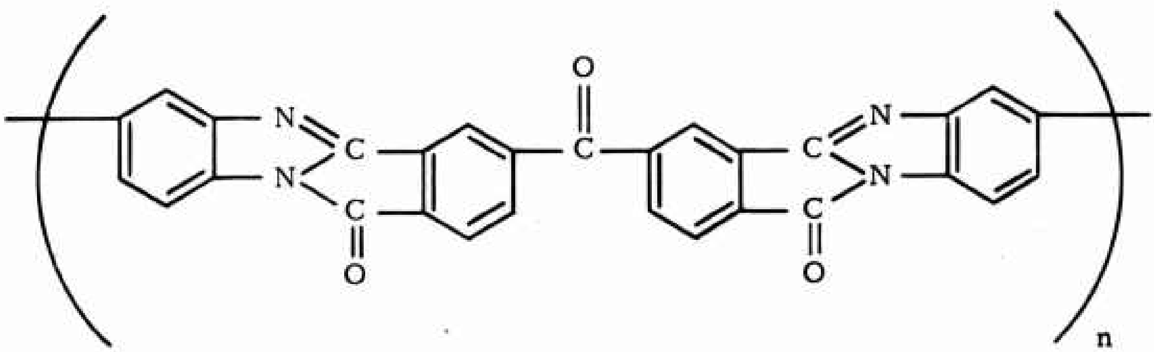
Figure 3. Insulation resistance measurements on perfluoroalkylenetriazine coated wires — 2.6 percent terephthalonitrile N, N' -dioxide

It was quite evident from these data that the triazine had good stability at 260°C (500°F).

The polymer would have to exhibit much higher temperature stability than this before it could be rated as a good prospective insulation for the purposes of this program.

2. "PYRRONE" (HUGHES HR-100)

"Pyrrone" resins generally are condensation products of aromatic tetracarboxylic acids or their anhydrides with aromatic tetraamines. However, the specific "pyrrone" Hughes HR-100 used in studies described herein was derived from the tetraethyl ester of benzophenone-tetracarboxylic acid and 3, 3'-diaminobenzidine. Results obtained with this polymer are not representative of those which would have been obtained from "pyrrone" resins derived from the free tetracarboxylic acids or the corresponding dianhydrides. The conventional "pyrrones" have grossly different solubility and processing characteristics. The theoretical structure of the polymer is shown below; however, elemental analyses demonstrate that HR-100 does not undergo complete ring closure.



Prior Hughes work on "pyrrone" wire insulation was described in the first annual summary report on this contract*.

---

*N. Bilow and K. L. Rose, High Temperature Electrical Wire Coatings, AFML-TR-69-111, Part I, May 1969.

An isothermal weight loss study was made on the HR-100 prepolymer to determine its rate of advancement in argon at 350°C (662°F) and the results are compared in Figure 4 to those obtained at 250°C (482°F) and 300°C (572°F). At this temperature (350°C) the polymer appears to reach its full cure in approximately 3 hours, whereas at 300°C about 4 hours are required. On the basis of these tests the polymer was generally cured for 3-4 hours at 316°C (600°F) in subsequent work.

It is to be noted, however, that the composition of the cured polymer varies with cure temperature, as evidenced by the elemental analyses, and that its empirical formula deviates considerably from theory.

	Percent C	Percent H	Percent N	Percent O
At 300°C	73.69	3.49	11.32	10.69
At 350°C	74.91	4.12	11.59	9.24
Calc. for $C_{29}H_{12}O_3N_4$	75.00	2.60	12.06	10.33

Five hundred feet of 3 mil thick Kapton polyimide tape (3/8 inch wide) was coated with "pyrrone" (HR-100) from a dioxane solution and was B-staged by passing the tape through a 2-foot long oven, under argon, at such a rate as to provide a 5-minute heating period. The oven temperature (at the center) was 280°C (556°F).

Silver plated multifilament copper wire (20 gage) was then wrapped with the coated tape. Wrapping was conducted at the Microdot Corp. using a tape temperature* of 315°-371°C (600-700°F). The tape was cured at 371°C (700°F) for 15 minutes and postcured under argon for 4 hours at 315°C (600°F).

*This is an apparent temperature measured with a thermocouple placed near but not on the tape. The actual tape temperature is unknown but is assumed to be within 50-100°F of the observed value.

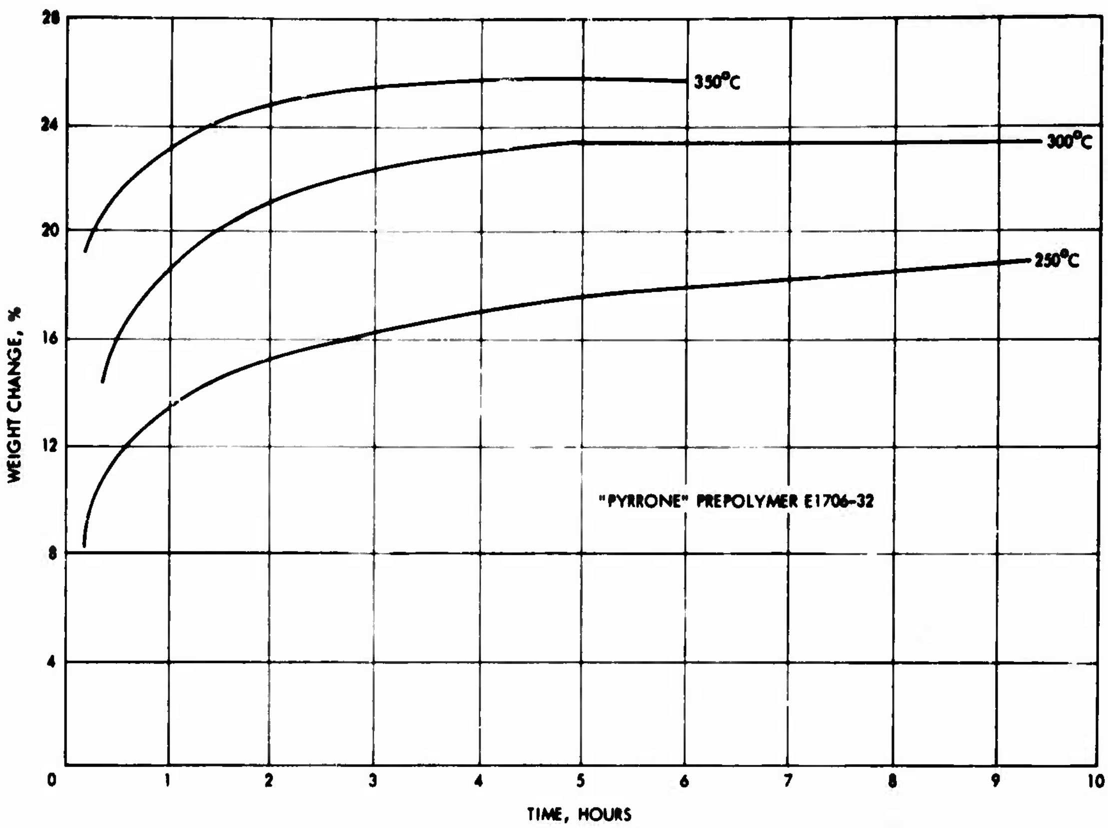


Figure 4. "Pyrrone" prepolymer weight loss as a function of cure temperature

Insulation resistance measurements showed that this wire was essentially the same as a smaller sample previously prepared. Results are shown in Table IV.

These results are presented graphically in Figure 5. Also shown in Figure 5, for comparison, is the insulation resistance data on the "pyrrone" (HR-100)-Kapton wire prepared a month previously. The wires appear to be equivalent.

Similar tests were conducted on FEP-sealed Kapton wire from Haveg Corporation and the results of these tests are illustrated in Figure 6.

Although the Haveg wire had better water resistance at ambient temperature, the abrupt drop in insulation resistance of the dry wire between 149-204°C (300-400°F) indicated that its glass transition temperature had been exceeded. Loss of its sealant properties as a

Table IV. Results of Insulation Resistance Measurements

Temperature		Insulation Resistance, Ohms 30 inch lengths
°F	°C	
75	23	6×10^{12}
200	93	1×10^{11}
300	149	1×10^{11}
400	204	3×10^{10}
500	260	7×10^{9}
600	316	2×10^{8}

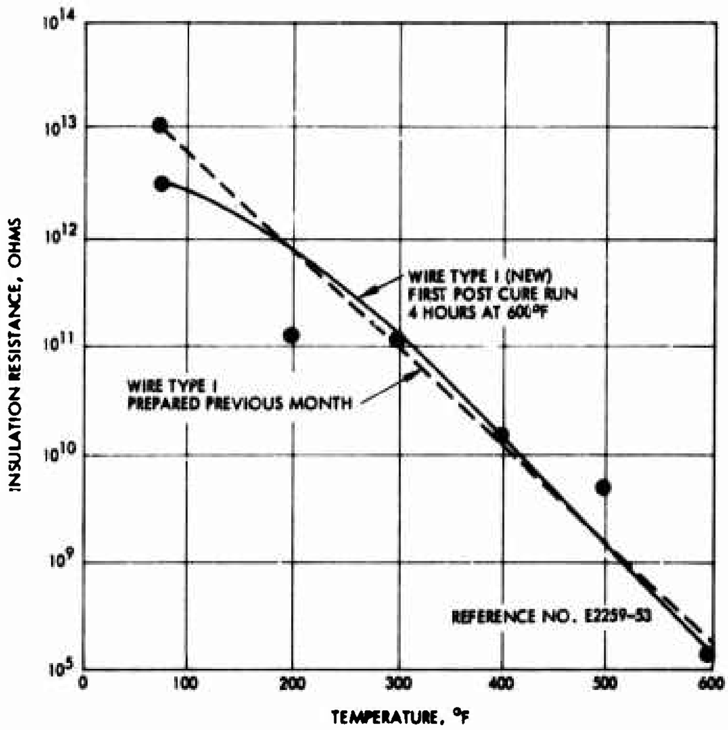


Figure 5. Insulation resistance of "pyrrone"-Kapton insulated wire

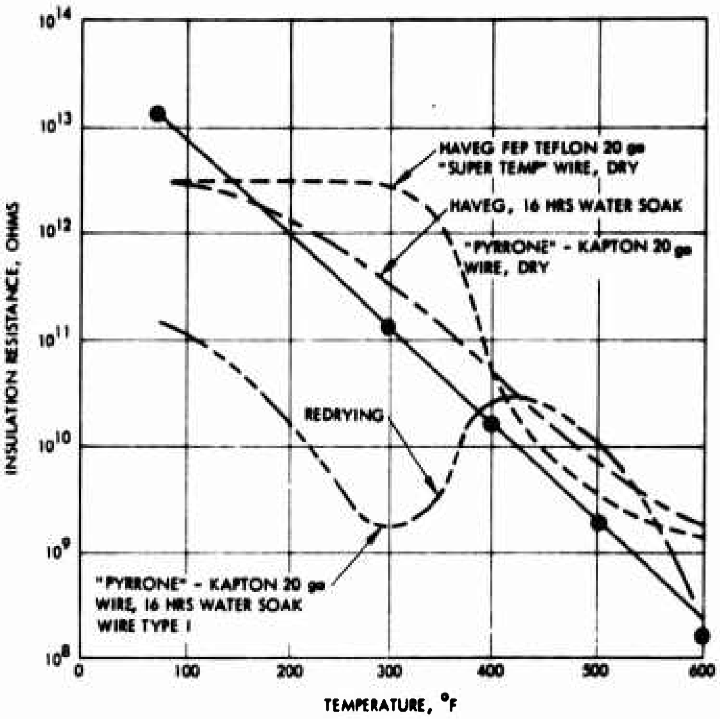


Figure 6. Effect of temperature on insulation resistance

result of this transition was expected, and in subsequent work discussed in the evaluation section of this report it will be noted that analogous FEP Teflon Kapton wires from Milo-Carolina Corp. peel and stick together at 288°C (550°F) and above. Furthermore, although its insulation resistance was slightly better than the Type I "pyrrone"-Kapton insulated wire between 204-316°C (400-600°F), adhesive failure occurs and very deleteriously affects its physical properties. In contrast the linear drop in insulation resistance of the "pyrrone"-Kapton wire (Type I) demonstrated that "pyrrone" resin had not exceeded its glass transition temperature. This supported the observation that adhesive failure was not observed up to 427°C (800°F). It is also important to note that the water-soaked "pyrrone"-Kapton wire (I) regained its properties when redried above 149°C (300°F).

"Pyrrone"-Kapton insulated wires (Type I) evaluated in this study were post-cured under argon in the apparatus illustrated in Figure 7.

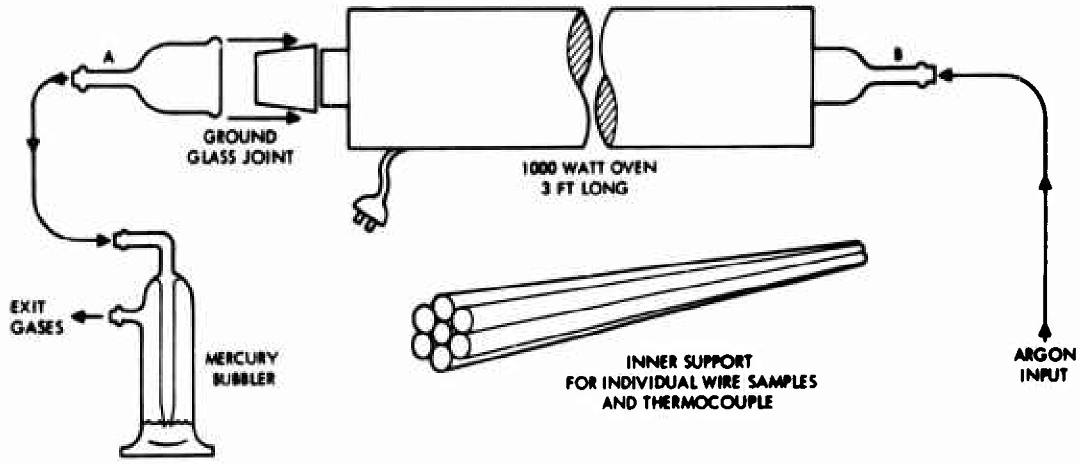


Figure 7. Wire postcure oven

a. Teflon Overcoated "Pyrrone"-Kapton Insulated Wires

Thirty-foot sections of the postcured wire (Type I - batch 2) were wrapped with 7 mil, 1/4 inch wide, Teflon TFE tape using a 50 percent overlap (Type IA) and the Teflon was sintered at 538°C (1000°F) for a period of about 1/2 minute. After sintering, the TFE layer was 4 mils thick. A second section of wire was double-wrapped with TFE also using 50 percent overlap (Type IB). This double wrap was 14 mils thick before sintering and 10 mils afterwards. Sintering was conducted at 1000°F for 1.1 and 2.1 minutes.

One 3-foot section of each of the overcoated wires was soaked in water for 16 hours and its insulation resistance was then measured. Wires were then removed from the water and tested again at elevated temperature. Results of these tests are shown in Table V below as well as in Figures 8, 9 and 10.

b. Perfluoroalkylenetriazine Overcoated "Pyrrone"-Kapton Insulated Wire

Other 30 inch lengths of postcured "pyrrone"-Kapton wrapped wire (Type I, No. 38A) were given coatings of perfluoroalkylenetriazine containing 1 percent terephthalonitrile N, N'-dioxide as a curing agent.

Table V. Insulation Resistance of Teflon Overcoated "Pyrrone"-Kapton Insulated Wire

Temperature,		Insulation Resistance,** Ohms		
		Type IA (No. 54-1) 4 mil, 1.1 min. sinter	Type IA (No. 54-2) 3.5 mil, 2.1 min. sinter	Type IB (No. 54-3) 10 mil, 1.0 min. sinter
°F	°C			
75*	23	7×10^{11}	1×10^{8}	6×10^{9}
75	23	3×10^{11}	2×10^{11}	3×10^{12}
200	93	5×10^{9}	1×10^{10}	2×10^{13}
300	149	2×10^{12}	3×10^{12}	1×10^{13}
400	204	3×10^{11}	1×10^{12}	6×10^{12}
500	260	1×10^{11}	2×10^{11}	2×10^{11}
600	316	7×10^{10}	1×10^{11}	6×10^{10}
700	371	3×10^{10}	8×10^{10}	8×10^{9}
800	427	3×10^{9}	3×10^{9}	8×10^{8}

*tests in water. **30 inch lengths

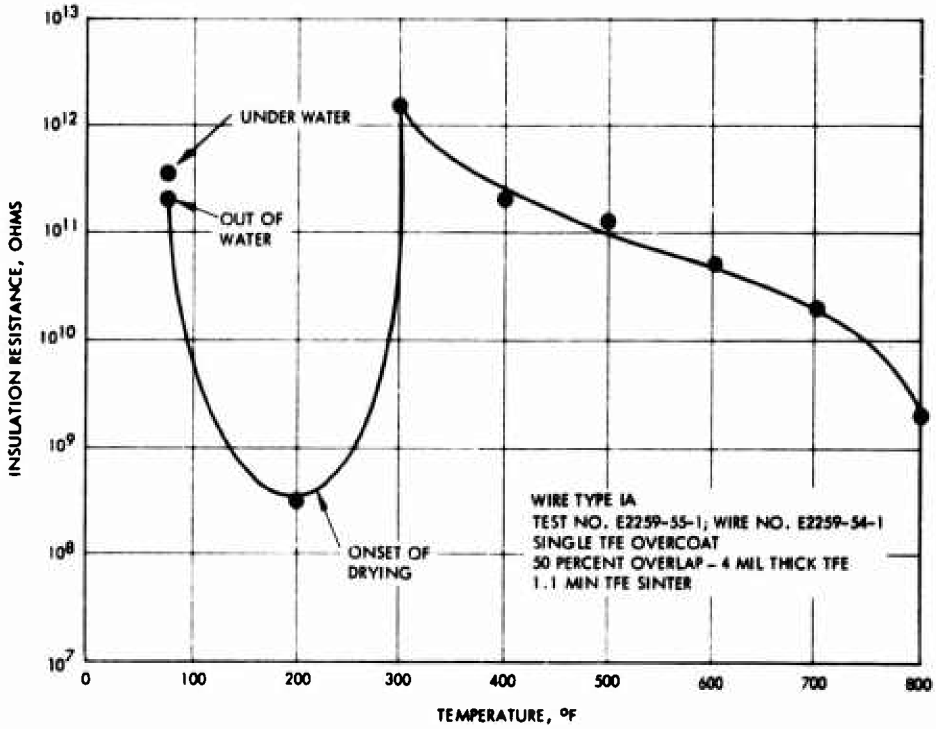


Figure 8. Insulation resistance of water-soaked Teflon TFE overcoated "pyrrone"-Kapton insulated wire — single overcoat - 1 minute sinter

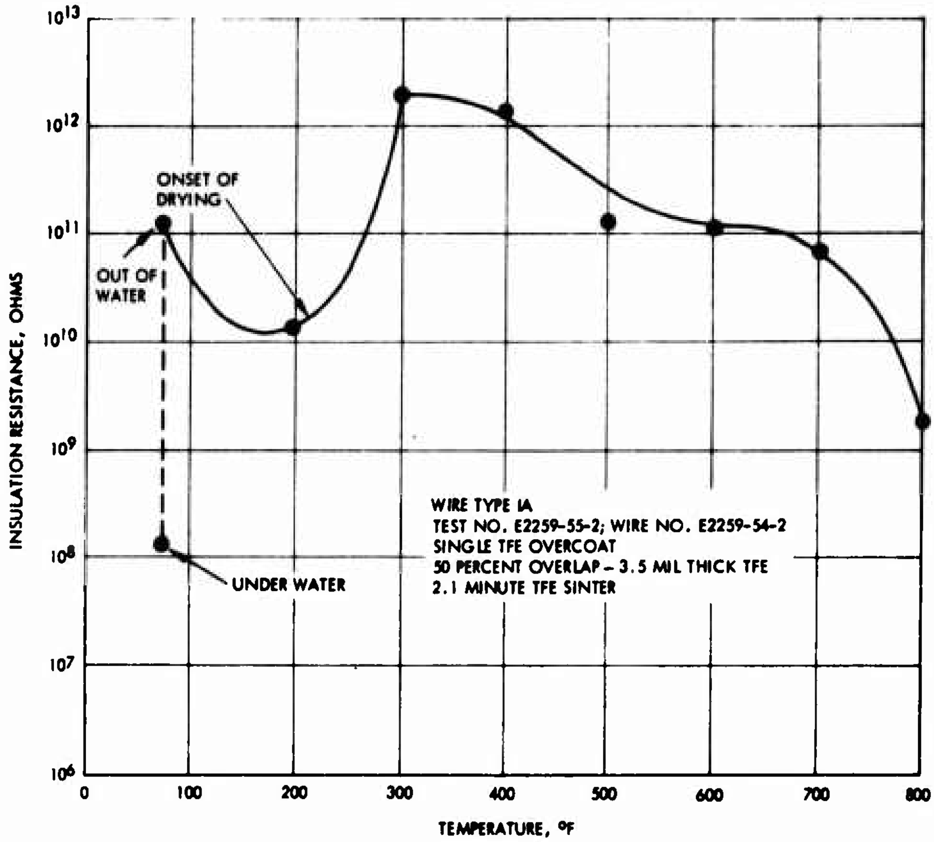


Figure 9. Insulation resistance of water-soaked Teflon TFE overcoated "pyrrone"-Kapton insulated wire - single overcoat - 2 minute sinter

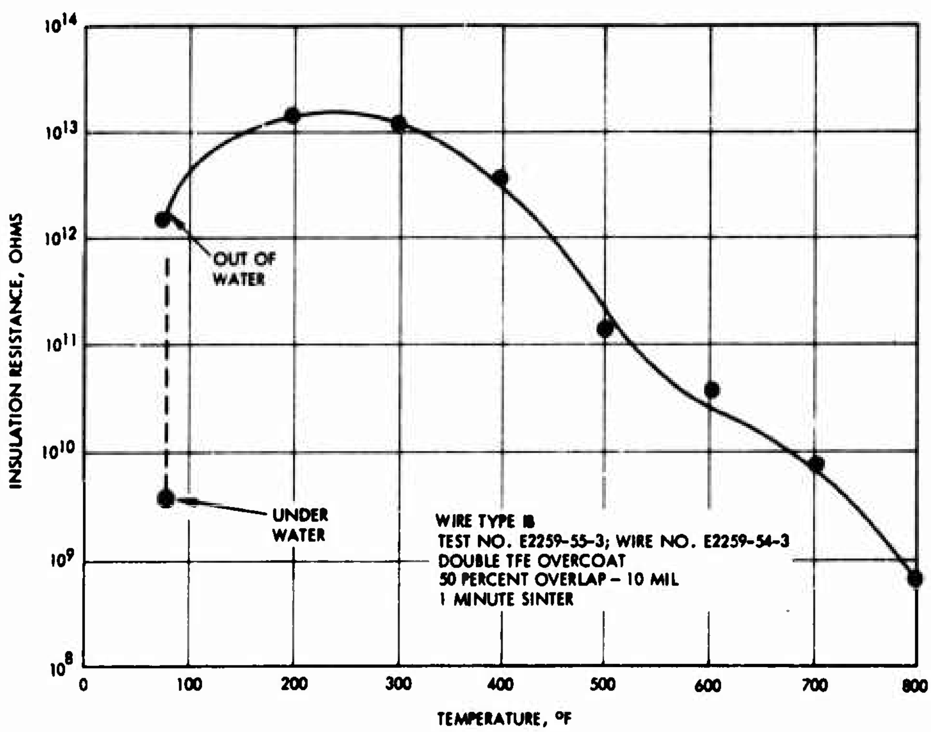


Figure 10. Insulation resistance of water-soaked Teflon TFE overcoated "pyrrone"-Kapton insulated wire - double overcoat - 1 minute sinter

The perfluoroalkylenetriazine polymer (20 grams) was dissolved in bis(trifluoromethyl)benzene for the dip application. Coated wires were air-dried for 2-1/2 hours, then cured for 2 hours at 150°C (302°F). One wire was soaked in water for 16 hours before measuring its insulation resistance and one wire was tested dry. The results of these tests are shown in Figure 11.

Another perfluoroalkylenetriazine overcoated "pyrrone"-Kapton insulated wire (Type ID) was divided into six 3-foot sections and after air drying overnight wire pairs were cured as follows:

Schedule a. 4 hours at 50°C

Schedule b. 4 hours at 100°C

Schedule c. 4 hours at 150°C

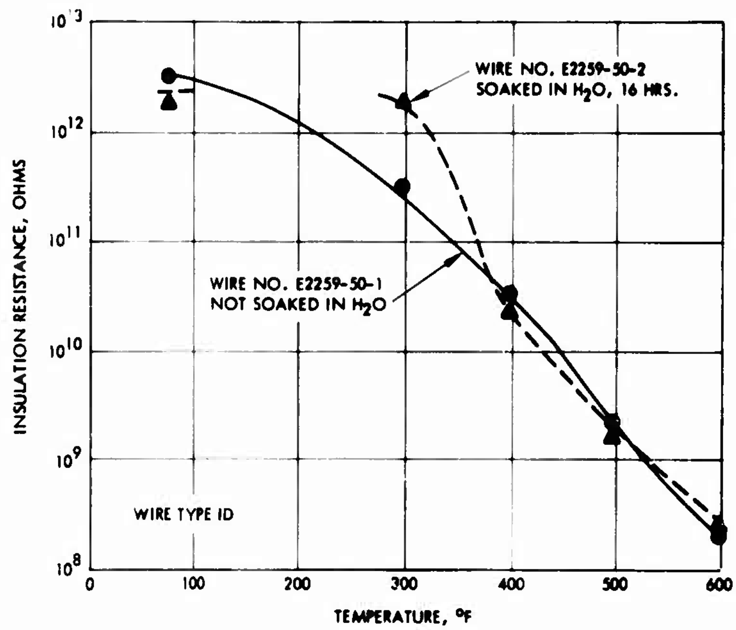


Figure 11. Insulation resistance of perfluoroalkylenetriazine overcoated "pyrrone"-Kapton insulated wire

The perfluoroalkylenetriazine coating was 0.75 mil thick. One wire of each of these pairs was soaked in anionic wetted water for 16 hours and subsequently tested for its insulation resistance. Results are shown in Table VI as well as in Figure 12.

Table VI. Insulation Resistance of Perfluoroalkylenetriazine Overcoated "Pyrrone"-Kapton Insulated Wires

Temperature,		Insulation Resistance, Ohms		
		Perfluoroalkylenetriazine Cure Schedule		
°F	°C	4 hours, 150°C	4 hours, 100°C	4 hours, 50°C
75*	23*	2×10^{12}	1×10^{7}	2×10^{8}
75	23	2×10^{12}	2×10^{8}	7×10^{6}
200	93	1×10^{10}	5×10^{7}	6×10^{6}
300	149	1×10^{11}	6×10^{10}	2×10^{10}
400	204	6×10^{10}	5×10^{10}	4×10^{10}
500	260	1×10^{9}	1×10^{9}	8×10^{8}
600	316	1×10^{8}	1×10^{8}	8×10^{7}
700	371	8×10^{6}	1×10^{7}	1×10^{7}

*Tests conducted under water. All wires 30 inches long.
These results are illustrated in Figure 12.

One pound of tetraethyl ester based "pyrrone" (HR-100, batches E 1706-32-1 and 2), having 1.5 equivalent percent of free carboxyl groups and an inherent viscosity of 0.52, was advanced at 200°C for 45 minutes under nitrogen, having a flow rate of 95 cc/min, until its inherent viscosity was 0.56. After cooling, the polymer was pulverized and dissolved in dioxane to yield a 28 percent solids content lacquer. This lacquer was used to coat 1 mil and 2 mil thick Kapton tapes in order to prepare insulated wires thinner than previously prepared. The prior wires had been made from 3 mil thick tape. The prepolymer lacquer was prepared by dissolving 208 g of the advanced resin (batch E2259-83A) in dioxane.

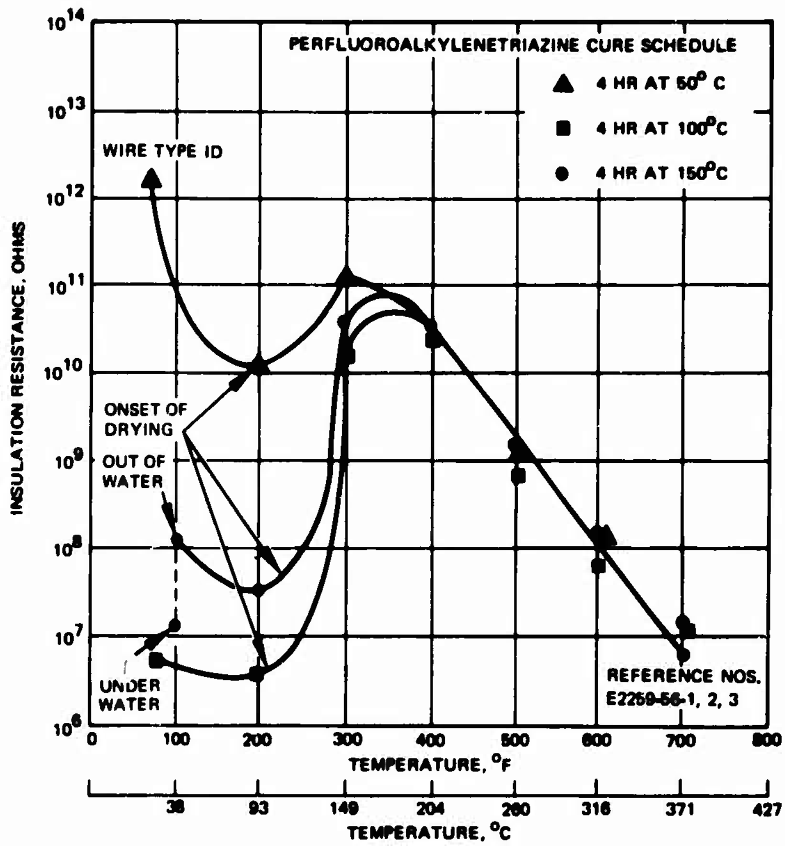


Figure 12. Insulation resistance of perfluoroalkylenetriazine coated "pyrrone"-Kapton insulated wire

These conditions are similar to those used previously; however, the coating device was modified with a new gear reduction train, new take-up wheels and guides to provide a more uniform feed, more uniform take-up, and a tighter wrap.

Two rolls of Kapton polyimide tape (1 mil x 3/8 inch wide) were coated using the 28 percent solids content lacquer. A total of 877 feet of tape was produced using the following conditions:

1. Solvent removal (drying) at 85°C for 49 seconds
2. B-stage oven temperature 270°C
3. Tape speed 1 ft/75 seconds
4. B-stage time 2.5 minutes
5. Argon flow rate 2 cu. ft/hr.

The coated tapes had 0.75 mil of "pyrrone" resin.

These processing conditions differ slightly from those used previously. When we first started to prepare this batch of tape it appeared that the resin did not B-stage properly; however, it was soon discovered that the argon flow rate was only one-tenth that used previously and consequently the tapes experienced less cooling and were actually being B-staged at a higher temperature. As soon as the problem was recognized it was possible to reproduce the tapes again.

One roll (719 feet) of 2 mil x 3/8 inch Kapton tape was also coated with the HR-100 lacquer. Identical coating conditions were used. The wire was wrapped on the take-up spool at a tension of 2 pounds.

Stranded 20 ga. 19/32 inch silver-plated copper wire was subsequently insulated by wrapping it at Microdot Corporation, using the 2 mil Kapton tape, at a wrapping temperature of 420°C (788°F). A 50 percent overlap was used. Heat was applied at the wrapping point for the first 400 feet of wire, but was moved 6 inches downstream for the remainder of the wire. The wire was thrice passed through a 10-foot curing oven at 371°C (700°F) for a total cure period of 3 minutes. The final insulation thickness was 7 mils. One hundred fifty foot lengths of wire were then post-cured in argon at 316°C (600°F) for 4 hours.

Similar wire was insulated with the HR-100-coated 1 mil Kapton tapes. This wire was made with 2 plies of HR-100-Kapton wrapped in opposite directions. Here also the overlap was 50 percent. The wrap temperature at the first head was 400°C (725°F) and at the second head was 420°C (788°F). The wire was passed through the curing oven 3 times at 371°C (700°F) at a rate of 5 feet/minute, giving a total exposure time of 6 minutes. The ultimate insulation thickness was 5.5 mils. The wire was considerably darker than one made earlier in the program and this indicated that the cure temperature may have been higher than previously used, even though the recorded oven temperature was not higher. It should be noted that a different curing oven had been used this time. The wire looked very good, even though dark.

Insulation resistance measurements were made on these new wires at 500 VDC with tests conducted in a molten metal bath.* After the tests were completed the wires were soaked for four hours in anionic "wetted" water and tested again. Results of these tests are shown in Table VII.

Wire Type I' was the single wrapped wire made from 2-mil Kapton. The HR-100 resin was fused by heating the tape downstream from the wrapping point.

Wire Type II was the double wrapped wire with each layer being HR-100 on 1-mil Kapton.

Wire Type I was the single wrapped wire with HR-100 on 2-mil Kapton; however, in contrast to wire Type I', this wire was fused by heat at the wrapping point.

Table VII. Results of Insulation Resistance Measurements

Temperature		Insulation Resistance,** Ohms		
°F	°C	Type I'	Type II	Type I
258	125	3×10^{12}	7×10^{11}	3×10^{12}
302	150	4×10^{11}	1×10^{11}	5×10^{11}
350	177	3×10^{10}	1×10^{10}	3×10^{10}
424	225	4×10^{8}	2×10^{8}	6×10^{8}
500	260	3×10^{7}	2×10^{7}	4×10^{7}
600	316	3×10^{6}	7×10^{6}	7×10^{6}
-	-	-	-	-
75*	23	4×10^{12}	5×10^{12}	2×10^{12}
*Tested under water.		**30 inch lengths.		

*Cerro-Tru, distributed by Peck Lewis Corporation, Los Angeles, California; 58 percent Bi, 42 percent Sn, m.p. 281°F (139°C).

Results of these tests indicated that the three types of wire were virtually equivalent electrically. These results are illustrated in Figure 13.

Portions of the postcured HR-100 2 mil Kapton wire (I) and the double wrapped HR-100 1 mil Kapton insulated wires (II) were returned to Microdot Corp. and overwrapped with one layer of Teflon (TFE) and subsequently sintered at 1000°F. The 2 mil by 3/8 inch Teflon tape had a 50 percent overlap. The wire speed through the sintering oven was 10 ft/min. resulting in a 1.5 minute sinter. This overwrap did not sinter as well as subsequently prepared wires, probably due to insufficient tension, and its lower quality was evident in subsequent electrical tests conducted after long term thermal aging. Thirty-second sintering was definitely too short a period of time.

It should be noted here that the HR-100-Kapton insulated wires made previously were all fabricated from 3-mil Kapton tape and consequently were thicker than the new wires.

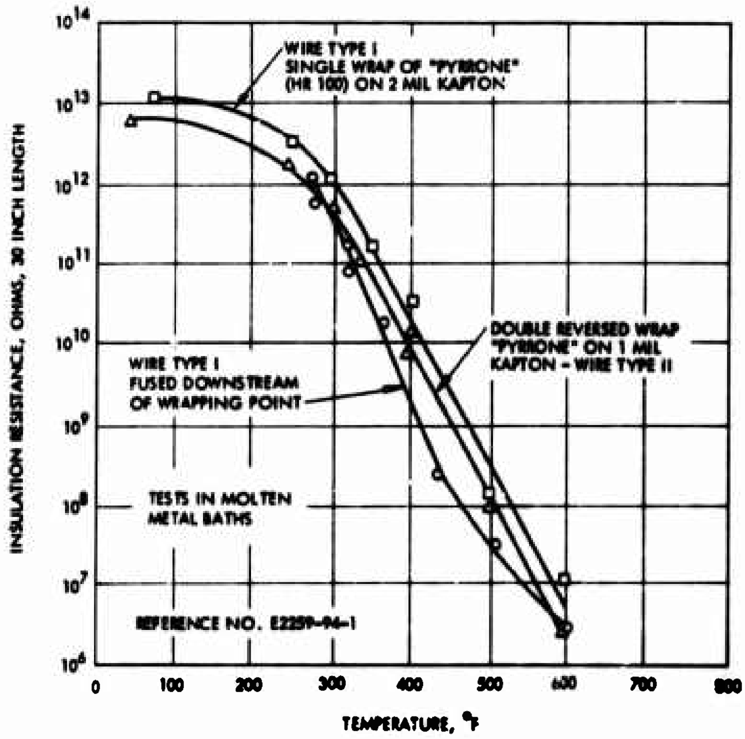


Figure 13. Insulation resistance of several "pyrrone"-Kapton insulated wires

Thermal aging studies, in air, were carried out on the various wires and are described on the following page.

For comparison a Teflon FEP-Kapton wire from Milo Carolina Wire and Cable Co. /(MIL-W-81381/1-20) was also tested and was the first to fail. A series of much longer and more significant thermal aging studies is described in the wire evaluation section of this report. Tests described on the following page are early results. Much longer term tests on the most advanced wires are discussed in that section.

Specifically the HR-100-Kapton wires tested were:

1. Single wrapped (50 percent overlap) HR-100 on 3-mil Kapton tape (3/8 inch wide) and overwrapped with 4-mil Teflon TFE (1/4 inch wide, 50 percent overlap). (Type IA)
2. Single wrapped (50 percent overlap) HR-100 on 3-mil Kapton tape (3/8 inch wide) and twice overwrapped with 3-mil Teflon TFE (1/2 inch wide, 50 percent overlap). (Type IB)

Insulation resistance measurements were made on wires immersed in anionic wetted water after a 4-hour soak period. Wires were dried and tested after 4 days at 316°C (600°F) and after 16 days at 316°C (600°F). The results are in Table VIII.

Table VIII. Insulation Resistance of Two "Pyrrone"-Kapton-Teflon Wires as a Function of Thermal Aging

Wire Type	Reference Number	Insulation Resistance,** Ohms		
		In Water Before Heat Exposure	After 96 Hours at 600°F	After 384 Hours at 600°F
IA	E2259-82-1a	1×10^{7}	2×10^{12}	2×10^{6}
	1b	1×10^{7}	2×10^{12}	3×10^{6}
IB	2a	4×10^{12}	2×10^{6}	0.9×10^{5}
	2b	5×10^{12}	3×10^{12}	6×10^{5}*
	Milo Carolina Wire	1×10^{10}	shorted	shorted

*Most of this wire was in excellent condition, but a small area showed evidence of breakdown.
**30 inch lengths.

Other life cycle tests were also conducted. For these tests both double layer Teflon TFE overwrapped HR-100-Kapton wires (Type IB) and single layer Teflon TFE overwrapped HR-100-Kapton wires (Type IA) were evaluated. Wires were heated at $600^{\circ}F$ ($316^{\circ}C$) in air and at various intervals they were removed from the oven, cooled, and tested under wetted water. The best of the four wire samples had an insulation resistance of 10^{11} ohms after 167 hours at $600^{\circ}C$. Other test data are shown in Table IX and are graphically illustrated in Figure 14.

Table IX. Insulation Resistance as a Function of Thermal Aging

Wire Type	Insulation Resistance, Ohms, 30 Inch Lengths								
	0 hour	27 hours	51 hours	72 hours	97 hours	120 hours	143 hours	167 hours	190 hours
IB	6×10^{13}	2×10^{13}	8×10^{12}	6×10^{12}	4×10^{12}	5×10^{12}	3×10^{12}	4×10^{8}	-
IB	6×10^{13}	2×10^{13}	1×10^{13}	6×10^{12}	7×10^{12}	5×10^{12}	2×10^{8}	6×10^{7}	-
IA	7×10^{13}	5×10^{12}	5×10^{12}	4×10^{12}	3×10^{12}	1×10^{8}	2×10^{8}	6×10^{7}	-
IA	7×10^{13}	2×10^{13}	1×10^{13}	6×10^{12}	2×10^{12}	3×10^{12}	2×10^{12}	4×10^{11}	Shorted

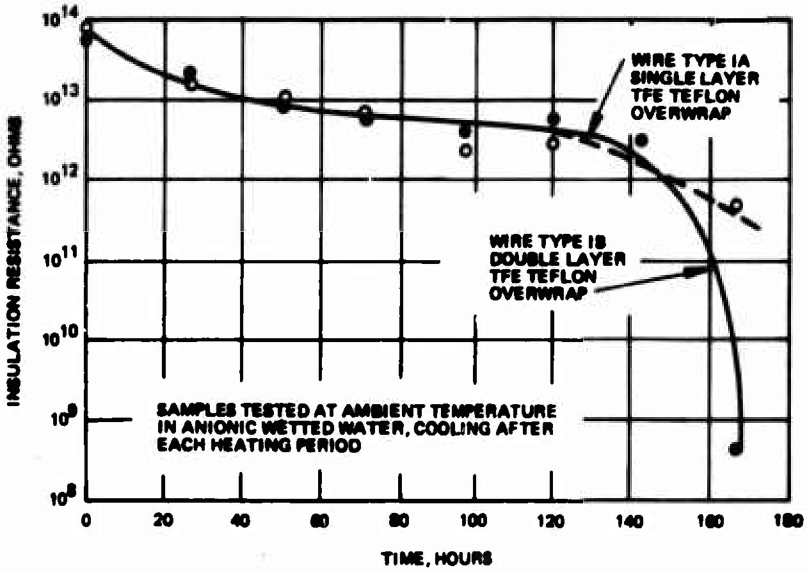


Figure 14. Insulation resistance of "pyrrone"-Kapton insulated wires as a function of thermal aging in air at $600^{\circ}F$

No significant electrical breakdown was observed in any of the wires during the first 97 hours. After 120 hours one wire showed significant breakdown, after 143 hours two wires had degraded significantly, and after 167 hours three wires had degraded.

The TFE overwraps showed fine cracks before the wires deteriorated electrically and this is probably due to stresses produced as the TFE continues to sinter. Tests on the more advanced wires are discussed later in this report.

Samples of the single wrapped HR-100 2-mil Kapton wire (I) and the double wrapped HR-100 1 mil Kapton wire (II) were also double overwrapped with Teflon TFE using 50 percent overlaps. In these cases, the first TFE layer was a white 2 mil by 3/8 inch TFE tape and the second TFE layer was a virtually colorless 2 mil tape. This time sintering was conducted at 1000°F for 45 seconds (20 ft/min. wire speed).

Two other types of wire were made from the single wrapped "pyrrone"-2 mil Kapton and the double wrapped "pyrrone"-1 mil Kapton by overcoating with a double wrap of TFE-FEP tape, again using a 50 percent overlap. This tape was 2 mils by 1/2 inch wide. These wires were sintered at 600°F in a 10-foot oven at a tape speed of 15 ft/min. (40 seconds total time). Insulation resistance measurements of these overcoated wires as a function of temperature are graphically illustrated in Figures 15 and 16.

Measurements of insulation resistance as a function of temperature were made on two "pyrrone" (HR-100)-Kapton (1 mil) wires. These include both a double wrapped and a triple wrapped wire. Results of these tests are shown in Table X and a graph of the data is shown in Figure 17. These data represent the best HR-100 Kapton wires fabricated in this program.

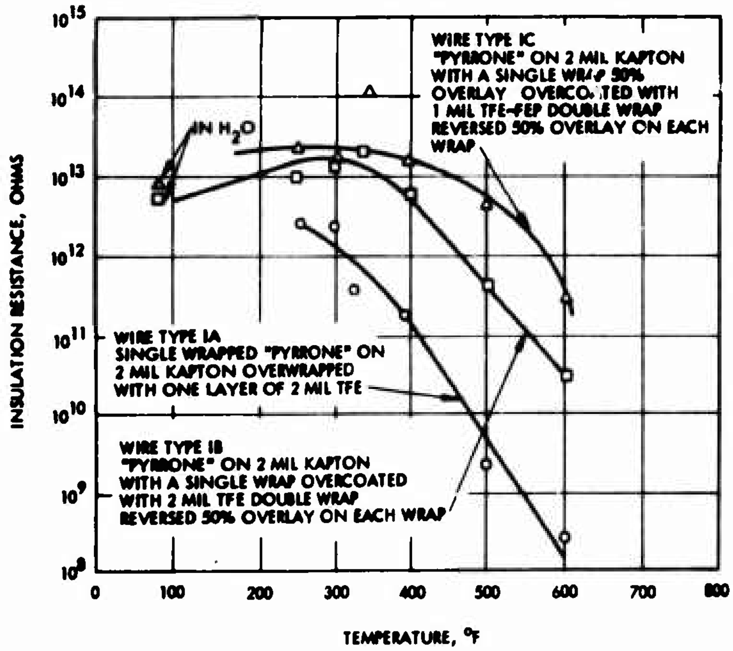


Figure 15. Insulation resistance of Teflon overcoated "pyrrone"-Kapton insulated wires as a function of temperature

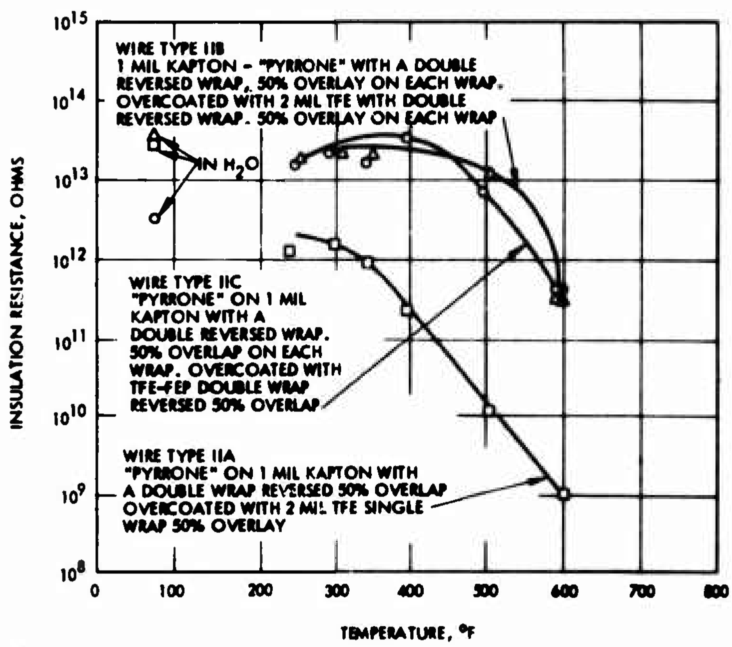


Figure 16. Insulation resistance of Teflon overcoated "pyrrone"-Kapton insulated wires as a function of temperature

Table X. Insulation Resistance as a Function of Temperature of "Pyrrone" (HR-100)-Kapton (1 mil) Insulated Wires

Temperature		Insulation Resistance, Ohms at 500 Vdc, 30 Inch Lengths	
^{o}F	^{o}C	Type II	Type VII
75*	24*	3×10^{12}	5×10^{12}
250	121	7×10^{12}	1×10^{13}
300	149	2×10^{12}	3×10^{12}
350	177	3×10^{11}	4×10^{11}
400	204	3×10^{10}	2×10^{10}
450	232	4×10^{9}	4×10^{9}
500	260	2×10^{8}	2×10^{8}
600	316	1×10^{7}	1×10^{7}

*Ambient temperature tests were conducted in anionic wetted water, all other tests were made in molten Cerro-Tru.

Code: II Hughes HR-100 on 1 mil Kapton polyimide - double wrap
VII Hughes HR-100 on 1 mil Kapton polyimide - triple wrap

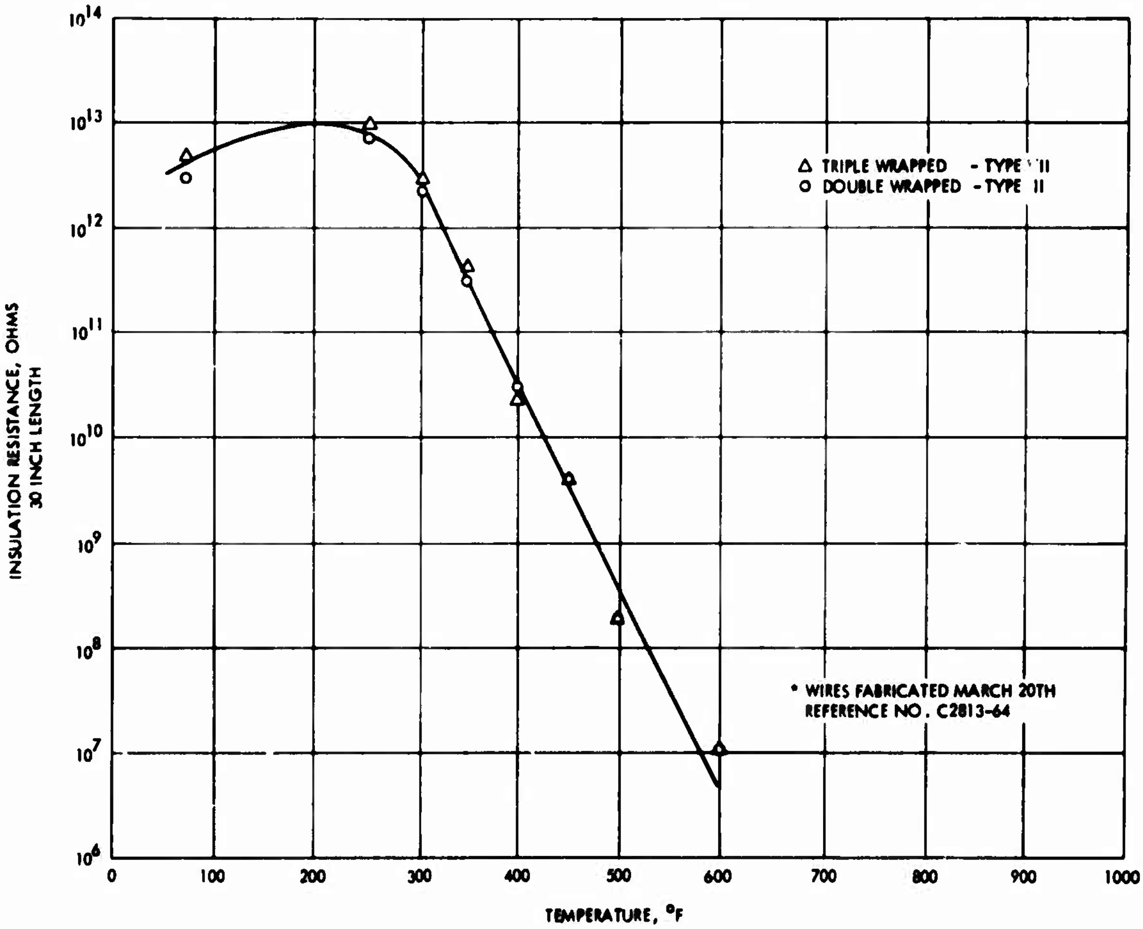
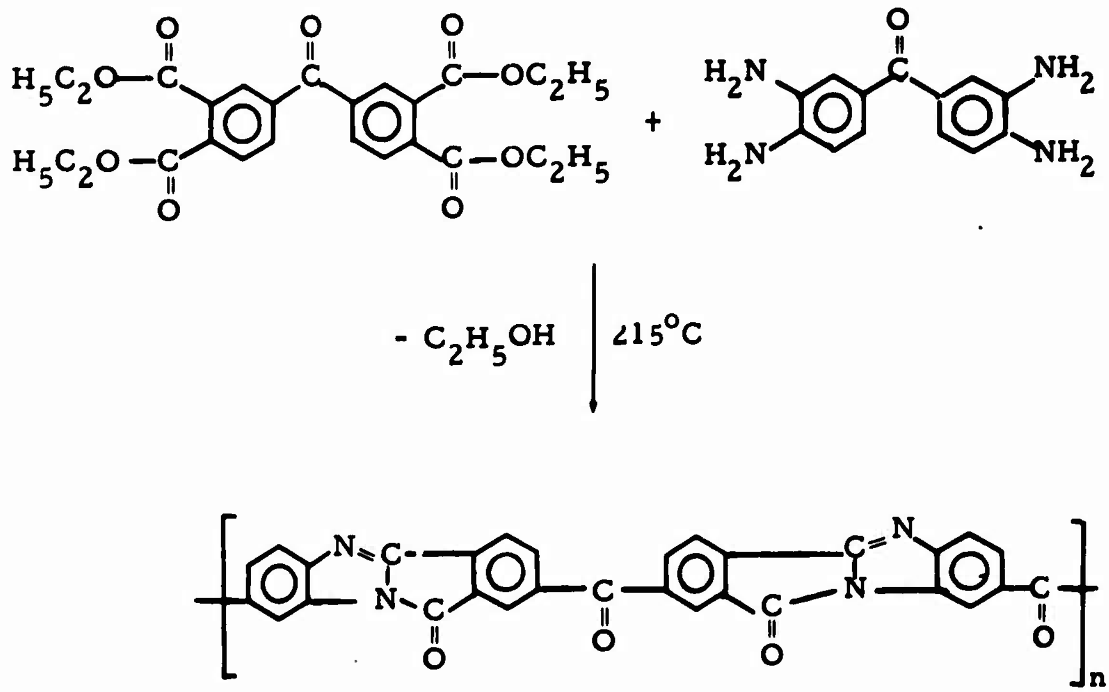


Figure 17. Insulation resistance of double and triple wrapped HR-100-Kapton wires as a function of temperature

3. "PYRRONE" (HUGHES HR-300)

Hughes HR-300 is a "pyrrone" type polymer derived from the condensation of 3, 3', 4, 4'-tetraaminobenzophenone with tetraethyl 3, 3', 4, 4'-benzophenonetetracarboxylate.

The synthesis is illustrated below.



Prepolymer preparation was carried out as follows (Experiment 1706-62). High purity tetraethyl ester (E1706-31, 47.1 g, 0.1 mole, 99.6 percent) was heated under argon to 180°C and solid tetraaminobenzophenone (24.2 g, 0.1 mole) was added while stirring continually. Since the amine did not melt immediately, the temperature was raised to 215°C. It took 20 minutes from the initial addition before all of the amine melted and dissolved. Stirring was continued for 30 minutes beyond this point. The reaction mixture was cooled rapidly in an ice bath and pulverized, in a mortar, to pass through a 40-mesh sieve. After drying in a vacuum oven overnight at 65°C, the inherent viscosity was measured and found to be 0.48 at 25°C at a concentration of 2.5 g per 100 cc of anhydrous dimethylformamide. It was soluble in dioxane and a 20 percent by weight solids content solution was prepared in this solvent.

Kapton polymide tape (1 mil x 3/8 inch x 500 ft) was coated with the HR-300 lacquer and subsequently dried by passing it through an air oven at 200°C to provide 50 second exposure. The tape then passed through a B-stage oven at 260°C using an exposure period of 150 seconds. The atmosphere was predominantly argon flowing at a rate of 2 standard cu. ft. /hr. Tape speed was 1 foot/75 seconds. This resulted in a total coating thickness of 0.75-1.00 mil (0.37-0.50 mils per side).

Multifilament silver plated copper wire (19/32, 20 gage, 0.039 inch diameter) was wrapped with this tape at a temperature of 800 ±100°F. (This was indicated by a thermocouple placed near the wrapping point. The actual tape temperature is unknown.)

Two types of wire were produced. Type VIII was double wrapped, each layer being in the opposite direction and the overlay on each layer was about 50 percent. The other, Type IX, was triple wrapped with each layer being reversed and with a 50 percent overlap in each layer. Each layer added 6 mils to the diameter of the wire or 3 mils to the wall thickness. Thus, wire VIII had a 6 mil wall (12 mil total insulation) whereas IX had a 9 mil wall (18 mil total insulation).

Wrapped wires were passed through a 10 foot air curing oven at 600°F (316°C) at a rate of 20 ft. /min. giving a total exposure period of 30 seconds. They were subsequently postcured in nitrogen for 4 hours at 600°F (316°C).

It was too late in the program to evaluate these wires comprehensively. They were excellent in appearance and were lighter in color than the HR-100-Kapton type wires, thus indicating better oxidation resistance.

Insulation resistance as a function of temperature was determined for each of these wires and the results are shown in Table XI. These results are illustrated graphically in Figure 18 and are compared to results obtained on the HR-100-Kapton insulated wires.

Table XI. Insulation Resistance as a Function of Temperature of "Pyrrone" (HR-300)-Kapton Insulated Wires

Temperature		Insulation Resistance Ohms at 500 Vdc 30 Inch Lengths	
°F	°C	Type VIII	Type IX
75*	24*	3×10^{12}	4×10^{12}
250	121	4×10^{12}	2×10^{13}
300	149	5×10^{12}	1×10^{13}
350	177	1×10^{12}	7×10^{11}
400	204	1×10^{11}	2×10^{11}
450	232	2×10^{10}	3×10^{10}
500	260	2×10^{9}	2×10^{9}
600	316	4×10^{7}	5×10^{7}

*Ambient temperature tests were conducted in anionic wetted water whereas all other tests were conducted in molten Cerro-Tru Bi-Sn alloy.

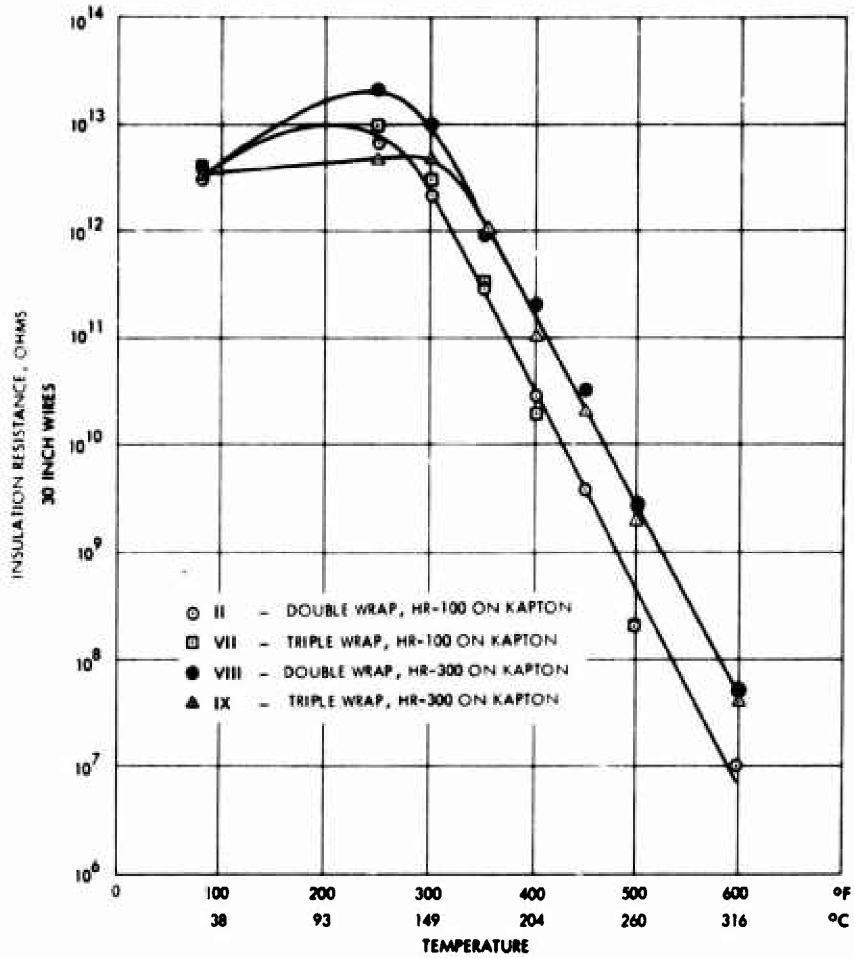


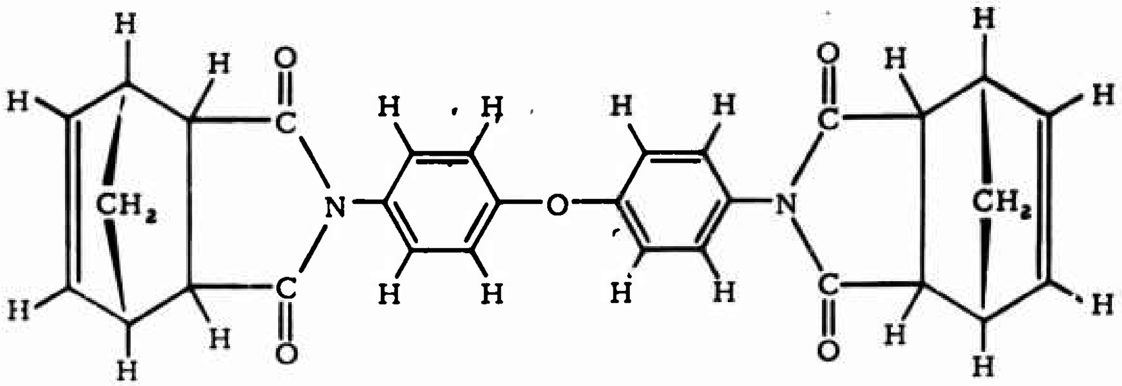
Figure 18. Insulation resistance as a function of temperature of HR-100 and HR-300 bonded Kapton insulated wires

It is interesting to note that the insulation resistance-temperature profile differs very little between VIII and IX even though wire IX has 33 percent more insulation. The same is true of II and VII.

Also shown in Figure 18 for comparison is an insulation resistance-temperature profile of the HR-100 based wires II and VII. Although the two curves are rather close, it is evident that the tetra-aminobenzophenone based polymer HR-300 (in Wires VIII and IX) is superior to the tetraaminobiphenyl based polymer HR-100. Thermal aging studies on the HR-300 wires are described on page 34.

4. POLYIMIDE P13N

Polyimide P13N is a product of TRW Systems Corporation. To our knowledge the prepolymer structure is that shown below.



Assuming this structure is correct, the polymer is derived from nadic anhydride [endo-bicyclo (2, 2, 1)-hept-5-ene-2, 3-dicarboxylic anhydride] and diaminodiphenyl ether. It would therefore not be a completely aromatic polymer. Nevertheless, it has a high degree of thermal stability near 316°C (600°F). A prepolymer of this structure would be expected to cure through vinyl type polymerization via the terminal vinyl groups in the cyclohexene rings.

In the first set of experiments the polyimide was sprayed, without further dilution, onto 3 by 6 inch sheets of 5-mil Kapton film. The specimens were air-dried for 60 minutes, then advanced (B-staged) under nitrogen at 200°C (392°F) for (a) 30 minutes, (b) 60 minutes, (c) 90 minutes, and (d) 120 minutes. Two-ply laminates subsequently were prepared and cured at 70 psi and 260°C (500°F) for 60 minutes, then postcured for 4 hours at 316°C (600°F) under argon. Prior to postcure the samples were completely free of voids and showed good adhesion. However, after postcure, the adhesive interlayers were full of voids.

In an effort to eliminate the void problem, a new series of laminates was prepared using different B-staging conditions. These conditions are shown in Table XII.

Table XII. B-Staging Conditions Used on P13N Polyimide Laminates

Sample (E2259-79)	Time, minutes	Temperature	
		°C	°F
1	100	220	428
2	120	220	428
3	140	220	428
4	160	220	428
5	100	230	446
6	120	230	446
7	140	230	446
8	160	230	446
9	100	240	464
10	120	240	464
11	140	240	464
12	160	240	464

These specimens were all B-staged under nitrogen. The first eight were cured at 70 psi and 260°C (500 F) for 1 hour, whereas samples 9 and 10 were cured at 150 psi and 260°C (500°F) for 1 hour. Specimens 11 and 12 were too far advanced to provide a bond when molded. Samples 1 and 2 showed good adhesion but many small voids. Samples 3 and 4 also showed good adhesion and had much lower void content. Samples 5 through 8 qualitatively had poorer adhesion and did not show good flow. Samples 9 and 10, even with the increased molding pressure, failed to flow sufficiently well to provide any degree of adhesion.

From these experiments it was apparent that a B-staging temperature of 230°C (446°F) for 2 hours was about the optimum. The conditions varied somewhat when the resin was used to prepare insulated wire, however.

In subsequent work, 16 sheets of sodium-etched Kapton film (3 inches x 6 inches x 3 mils) were spray coated with the P13N prepolymer lacquer. After a 30-minute air dry they were B-staged in nitrogen as shown in Table XIII. Subsequently, 2 ply laminates were prepared using a 60 minute cure at 150 psi and 316°C (600°F) and an 8-hour post-cure in argon at 316°C.

Table XIII. B-staging Conditions Used to Bond Kapton Film Spray Coated with P13N Prepolymer Lacquer

Sample 2259-85	B-Stage Conditions	
	Time, minutes	Temperature, °C
1	100	230
2	120	230
3	140	230
4	160	230
5	100	240
6	120	240
7	140	240
8	160	240

Samples B-staged at 240°C (464°F) all provided poor adhesive bonds, whereas samples B-staged at 230°C (446°F) bonded together very well. In T-peel tests conducted at 315°C (600°F) the specimens all failed in the substrate rather than in the adhesive interface. The latter samples had a sufficiently small number of voids in their interfaces to suggest that void-free P13N bonded Kapton insulated wires could be achieved.

After finding conditions which appeared satisfactory for preparing essentially void-free P13N adhesive interfaces, attempts were made to coat Kapton tape (2 mil x 3/8 inch wide) with sufficient uniformity to permit wrapped insulation to be made. In these coating studies the P13N lacquer appeared to wet the tape quite uniformly, but when the tape was passed through the drying tower it invariably came out with resin thicker on the edges regardless of how the apparatus or drying tower parameters were adjusted. When dioxane was added to the lacquer (which normally contains only dimethylformamide), the drying proceeded somewhat better, although completely uniform thickness still was not achieved.

These tapes were B-staged at 260°C (500°F) in argon and then two ply laminates were prepared. The laminates were cured at 344°C (650°F) for 1, 5, 10, and 15 minutes. They contained many voids but the adhesion was good. Other tapes, B-staged in argon at 290°C (554°F) for 5 minutes, also were made into two ply laminates and cured at 650°F (344°C) for 1, 5, and 10 minutes. These all failed to bond, showing that the resin was too far advanced. Other conditions were subsequently investigated and were as shown in Table XIV.

Since dioxane helped to provide more uniform coatings, a sample of P13N lacquer (150 ml) was concentrated by removing a portion of the dimethylformamide (50 ml) on a rotary evaporator immersed in a 60°C water bath. To the concentrate which contained 65 g solids was added dioxane (235 ml) and fresh dimethylformamide (15 ml). The diluted lacquer (374 g) contained 18 percent resin solids by weight.

Table XIV. Subsequent B-staging Conditions for P13N-Kapton Tapes

Sample	Drying Oven			B-stage Oven			Observations on 2-ply Laminates
	Temperature		Time, sec	Temperature		Time, min	
	°C	°F		°C	°F		
1	100	212	90	260	500	5	Many voids
2	135	275	90	270	518	5	Few voids, good flow
3	170	338	90	270	518	5	Void-free, good flow
4	190	374	90	270	518	5	Poor flow, void-free

Two hundred and six feet of Kapton tape (1 mil) was coated with this modified P13N lacquer after first passing the tape through a dioxane wash. The coated tape was dried in air at 200°C (392°F), then B-staged in a 260°C (500°F) oven while drawing the tape through at a rate of 1 foot per minute. The resultant coating thickness varied from 0.4 to 1.5 mils. It was more uniform then prior samples; nevertheless, it was far from being an ideal tape for producing wrapped insulation. The total drying time was 48 seconds and the B-stage period was 2 minutes. Subsequently the coated tape was wrapped onto multifilament silver-plated wire (20 ga., 7/28) using a 50 percent overwrap. Heat was applied at the wrapping point. The indicated wrap temperature was between 370-427°C (700-800°F); however the actual tape temperature could not be established. One hundred thirty four feet of insulated wire was obtained. It was cured in part by passing it through a 10-foot oven at a rate of 10 ft/min. at 316°C (600°F), but most of it was passed through at double this rate. Postcure was carried out at 316°C (600°F) in nitrogen for 1 hour. Total insulation thickness was 3 mils. The wire, designated IV, looked excellent.

A temperature profile of this wire was made by measuring its insulation resistance at various temperatures, and the data is presented graphically in Figure 19. Subsequently it was overwrapped with TFE Teflon to provide two new types of wires.

Both utilized a 2 mil x 1/2 inch wide TFE tape for the overwrap; however, in one case two layers of directionally reversed TFE were applied (wire IVB) and in one case one layer of TFE tape was applied (wire IVA). In both cases the overlap was approximately 50 percent. Wires were sintered at 1000°F (538°C) for 1 minute while passing them through a 15-foot air oven. Wire having the single layer of TFE had a 6-1/2 mil insulation wall thickness, whereas the doubly overwrapped wire had an insulation wall thickness of 11 mils.

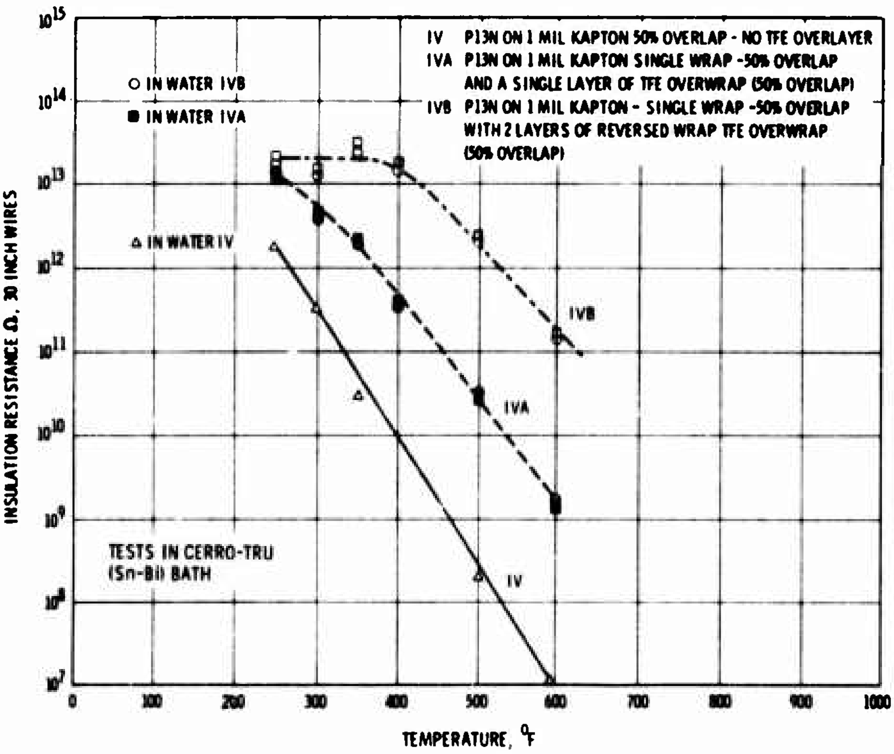


Figure 19. Insulation resistance as a function of temperature of P13N bonded Kapton insulated wires

Insulation resistance measurements, as a function of temperature, on both types of wires, are tabulated in Table XV and are also presented graphically in Figure 19.

Insulation resistance measurements were all made in duplicate. In all ambient temperature tests the wires were soaked in anionic wetted water for a minimum of 1 hour before testing. In the elevated

Table XV. Insulation Resistance as a Function of Temperature of P13N – Kapton Insulated Wires

Temperature °C	Temperature °F		Wire IVA, Ohms 30 Inch Lengths	Wire IVB, Ohms 30 Inch Lengths
23	75	a*	8×10^{13}	1×10^{14}
		b*	8×10^{13}	7×10^{13}
121	250	a	2×10^{13}	3×10^{13}
		b	1×10^{13}	3×10^{13}
149	300	a	6×10^{12}	1×10^{13}
		b	7×10^{12}	2×10^{13}
177	350	a	3×10^{12}	4×10^{13}
		b	3×10^{12}	6×10^{13}
204	400	a	5×10^{11}	2×10^{13}
		b	6×10^{11}	3×10^{13}
260	500	a	5×10^{10}	4×10^{12}
		b	5×10^{10}	4×10^{12}
316	600	a	2×10^{9}	2×10^{11}
		b	2×10^{9}	2×10^{11}

*Under water

temperature tests the 1 hour soaking periods were also used, but only after the wires had been allowed to recool to ambient temperature. Tests were conducted by applying a 500 Vdc potential between the wire conductor and a conductor immersed in the wetted water. Leakage was measured with a Hewlett Packard Model 425A DC microvolt-amp meter.

Another type of P13N-Kapton wire was also prepared. Similar conditions were used except that a double wrap of the tape was used. None of this wire, designated type V was overwrapped with Teflon. The tape used in this wire was from a different batch than that used previously. When preparing this new batch efforts were made to use a P13N lacquer which had been made up 2 months earlier. This effort was initially unsuccessful because the polymer had advanced on aging. Evidently the polymer has limited shelf life. A new batch of polymer lacquer (84 g) was subsequently obtained, and after removal of part of its solvent, dioxane was added as in earlier work.

Kapton tape (1 mil) was coated with this new lacquer to provide 400 feet of coated tape, which was double wrapped onto 20 ga. (19/32) silver-plated copper wire. Wraps were in the reverse direction with 50 percent overlap in each layer. The wrapping temperature was 427 ± 28°C (800 ± 50°F) and the resultant insulation wall thickness was 9 mils. Cure was achieved by passing the tape through a 10 foot curing oven in air at 316°C (600°F) at a rate of 20 ft/min. Total exposure time was 30 seconds. Postcure was conducted at 288°C (550°F) in nitrogen for 3 hours. Results of tests on temperature versus insulation resistance of the Type V wire are shown in Table XVI. A graphical plot of this data is shown in Figure 20.

Various evaluations were performed on the Type IV, IV A, IV B, and V wires. As a result of these studies it became quite evident that the double wrapped wire V was significantly superior to wire IV, not only in its insulation resistance versus temperature profile, but especially in its long term stability in air at 316°C (600°F). The wires also performed well in high temperature humidity tests and cold bend tests. These results are discussed in detail in the wire evaluation portion of this report.

Table XVI. Insulation Resistance as a Function of Temperature of Double Wrapped P13N-Kapton Insulated Wires

Temperature		Insulation Resistance, Ohms 30 Inch Lengths
°C	°F	
24	75	6×10^{13}
121	250	7×10^{12}
149	300	1×10^{12}
177	350	3×10^{11}
204	400	5×10^{10}
260	500	1×10^{10}
316	600	2×10^{8}

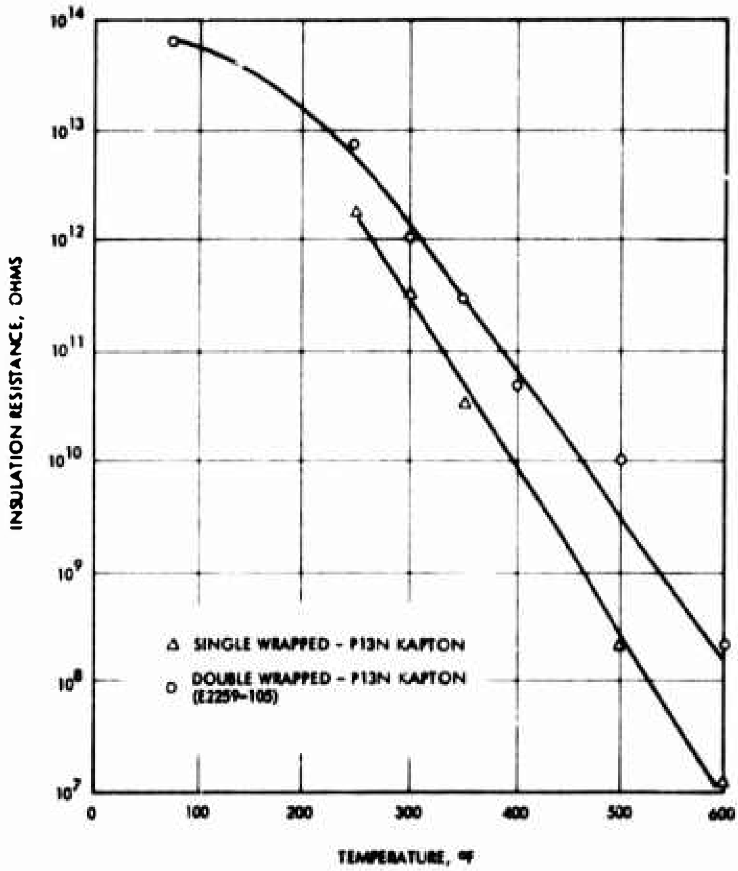
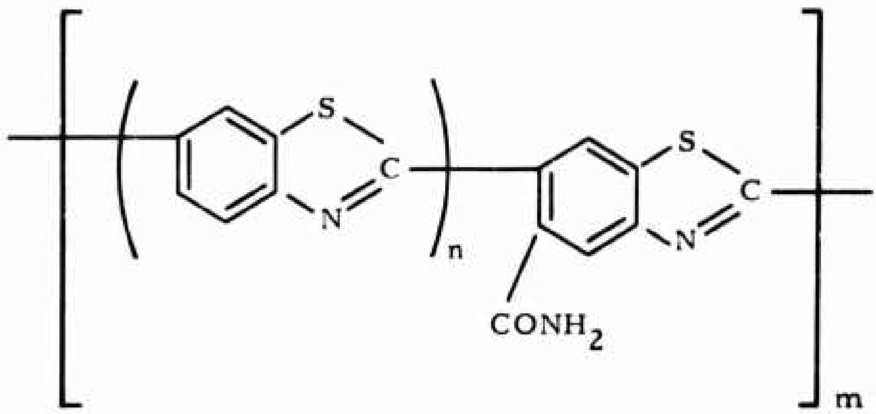


Figure 20. Insulation resistance of P13N-Kapton insulated wire as a function of temperature

5. POLYBENZOTHIAZOLE

Polybenzothiazole was also screened as a potential insulation. The polybenzothiazole AF-R-2506, described by Aponyi and Mecum*, contains approximately 25 percent by weight of 4-aminophthalimide. This reacts with the polybenzothiazole prepolymer to produce a resin with pendant carbamide groups, which serve as latent crosslinking sites. The cured polymer reportedly has the structure shown below.



This polybenzothiazole was selected as a potential high temperature insulation material because structural composites containing the resin have been shown to exhibit good strength retention (up to 75 percent) when heated in air for 200 hours at 316°C (600°F). Polymer lacquers can be prepared using dimethylformamide as a solvent and consequently the polymer can be used in impregnations, as well as in coatings and varnishes. A major problem encountered in processing AF-R-2506 is the large amount of volatiles evolved during cure. For example, as much as 50 percent of the aminophthalimide will sublime during cure and the evolved gases lead to the formation of porous structures. Long cure schedules are also required. Aponyi and Mecum, for example, describe the fabrication of an AF-R-2506 laminate

*T. J. Aponyi and W. D. Mecum, J. Composite Materials, 2, No. 2, p. 186, April 1968.

in which pressure release was required 20 seconds out of each minute the laminate remained in the press, and the total cure cycle was 35-60 minutes at 329 ± 12°C (625 ± 25°F). Furthermore, the laminates required a temperature-programmed postcure of 52 hours, during which time the temperature was raised from 177°C (350°F) to 454°C (850°F).

In the investigation conducted under this program, a suspension of AF-R-2506 (10 grams) in dimethylacetamide (28 grams) was used to coat sheets of sodium-etched Kapton polyimide film. The sheets were air dried for 30 minutes and then oven dried for 1 hour at 125°C (257°F). A two ply laminate then was prepared and cured at 750 psi and 260°C (500°F) for 30 minutes. However, the polybenzothiazole interlayer was full of voids. This indicated that B-staging would be required to allow for the escape of gases before the Kapton sheets could be bonded together.

The AF-R-2506-dimethylacetamide dispersion was also used to coat a sample of bare copper wire and the coating was dried and cured in argon for 24 hours at 260°C (500°F). The cured insulation was too brittle to permit electrical insulation measurements to be made.

In attempting to use the polybenzothiazole as a sealant for electrical wire insulation it was considered unwise to add zinc oxide to the resin because of the deleterious effect this would have on the electrical properties of the insulation. Sealant studies conducted with the polybenzothiazole (PBT) AF-R-2506 thus utilized polymer which contained the 4-aminophthalimide but not zinc oxide. The zinc oxide is, however, a recommended additive and in studies by Aponyi and Mecum it was found to improve the flexural strength retention of thermally aged laminates which incorporated it, as shown below. However, the zinc oxide does

Resin	Filler	Flex Strength of A1100E Glass Laminate	Flex Strength After 192 Hours at 600°F
PBT plus 26 percent 4-aminophthalimide	4 percent ZnO	58,700 psi	43,100 psi
	none	71,200 psi	19,400 psi

not appear to be essential in promoting cure, as indicated by the high flex strength which Aponyi and Mecum observed in the laminate without zinc oxide.

Eight 3 by 6 inch sheets of sodium etched 5 mil Kapton film were spray-coated with a solution of polybenzothiazole AF-R-2506 in dimethylacetamide containing 26 percent solids. The sheets were air-dried for 30 minutes, then B-staged in argon at 157°C (315°F) for 10, 20, 30, and 60 minutes. Pairs of sheets were then pressed together and cured at 70 psi for 60 minutes at 260°C (500°F). Laminates were cooled under pressure but were found to be full of voids, although the one having the longest B-stage period had the least voids. Qualitatively the bond strength appeared good, although the samples were too full of voids to justify quantitative testing.

Eight more sheets were similarly prepared. Laminates prepared therefrom appeared somewhat better but also contained many voids. These laminates, when postcured at 315°C (600°F) for 8 hours under argon, had increased void content and qualitatively poor adhesive bond strength.

A third set of laminates next was prepared. The sheets were B-staged at 150°C (302°F) for (a) 10 minutes, (b) 20 minutes, (c) 30 minutes, (d) 60 minutes, and (e) 120 minutes. Two ply laminates then were prepared and press-cured at 70 psi and 260°C (500°F) for 60 minutes. After press cooling, all specimens were found to have less voids than those previously prepared; sample "e" was completely free of voids. These samples were postcured for 8 hours at 315°C (600°F).

A fourth set of laminates was prepared using B-staging temperatures of 225°C (437°F), 250°C (482°F), 275°C (527°F), and 300°C (572°F) for a 2 hour period. Two ply laminates then were molded at 70 psi and 500°F for 1 hour. These samples were completely free of voids, but they had poor adhesion. They were postcured at 600°F for 8 hours in argon.

In summary it appears that when the polybenzothiazole is advanced far enough in the B-stage to give void-free adhesive interlayers, it is too far advanced to give good adhesion.

Since little success was obtained with the zinc oxide free polymer, further experimentation was subsequently carried out with zinc oxide filled resin and is described below.

Eight sheets of etched Kapton film (3 inches x 6 inches x 3 mils) were spray-coated with a lacquer of polybenzothiazole AF-R-2506 in dimethylacetamide containing 4 percent zinc oxide by weight. After a 30 minute air dry the samples were B-staged in nitrogen at 315°F (157°C) for 10, 20, 30, and 60 minutes. Two ply laminates were prepared and cured for 60 minutes at 600°F (315°C) and 150 psi and subsequently postcured for 8 hours at the same temperature. All samples contained voids, although they had good adhesion. In T-peel tests at 600°F failure occurred in the Kapton substrate rather than in the adhesive. The samples contained considerably less voids than those prepared previously without the zinc oxide.

A polybenzothiazole AF-R-2506 pellet containing 4 percent zinc oxide by weight also was molded at 600°F. Dielectric properties of this pellet were measured at ambient temperature and 500 Vdc and are shown below:

Dielectric constant	3.4
Dissipation factor	3.9 percent
Volume resistivity	7.7×10^{13} ohm-cm

The dissipation factor is obviously high and would be expected to be significantly greater at higher temperatures.

The high dissipation factor was however not unexpected because of the presence of the zinc oxide. It thus appears that although zinc oxide provides better high temperature oxidative resistance for the resin, it simultaneously deteriorates the electrical properties.

6. SILICONE XR-4-3083

Dow Corning XR-4-3083 was also screened as a potential high temperature sealant and insulation. The molecular structure of this silicone is unknown to us; thus it cannot be illustrated here.

Following the procedures recommended for cure, the XR-4-3083 silicone was sprayed onto sheets of Kapton polyimide film without thinning and was B-staged under nitrogen at 175°C for (a) 3 minutes, (b) 5 minutes, (c) 10 minutes, and (d) 15 minutes. Three minutes was the recommended period. After B-staging, the pairs of samples were pressed together by hand and the air voids worked out of the laminates until they were void-free. Sample "a" was postcured in argon for 2 hours at 260°C (500°F), whereas samples "b", "c", and "d" received a 4 hour cure at this temperature. The adhesive bonds were qualitatively poor with cohesive failure in all cases.

A second set of specimens then was prepared. Twelve sheets of Kapton film (3 inches x 6 inches x 5 mils) were spray-coated with the silicone and, after air drying for 30 minutes, were B-staged in nitrogen as shown in Table XVII. These B-staging periods were more

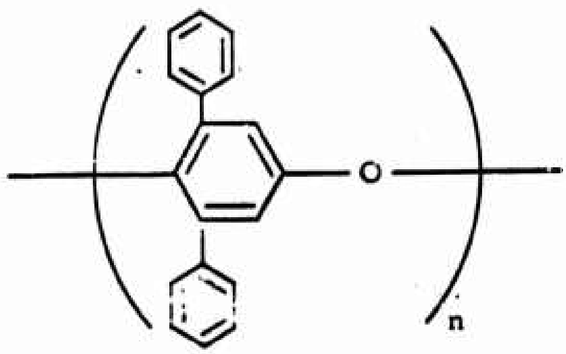
Table XVII. Conditions for B-Staging
Silicone XR-4-3083

Sample E2259-81	B-Stage Conditions		
	Time, minutes	Temperature	
		°C	°F
-1	5	200	392
-2	10	200	392
-3	20	200	392
-4	5	250	482
-5	10	250	482
-6	20	250	482

extreme than those used previously. Pairs of samples were bonded together and cured at 150 psig and 316°C (600°F) for 1 hour. These specimens were free of voids after cure. However, when they were subsequently heated in argon for 4 hours at 260°C (500°F), the adhesive bonds failed in many places, giving a highly voided adhesive interlayer. This silicone thus showed little promise as a high temperature adhesive for Kapton film, and consequently work with it was terminated.

7. POLYTERPHENYLENEOXIDE

Polyterphenyleneoxide is a new experimental resin from the General Electric Corporation. Its structure is believed to be that shown below

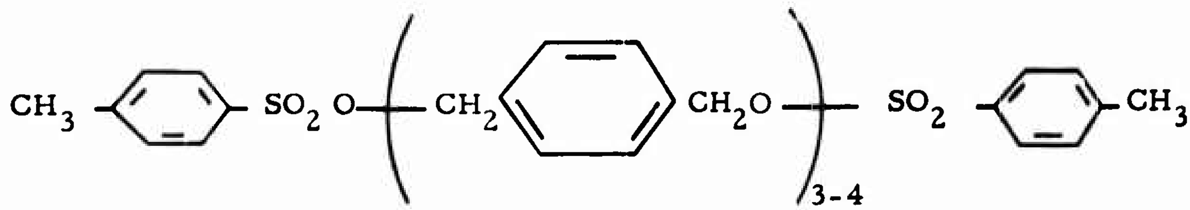


It is homologous to the poly(2, 6-dimethylphenylene oxide) which has been available for several years.

The polymer has a glass transition temperature which is reported to be near 280°C (536°F). It does, however, show some signs of softening at about 225°C (436°F). After passing through this transition temperature it resolidifies and fails to remelt at 400°C or below.

Polyterphenyleneoxide (20 grams) was dissolved in chloroform (30 ml) to provide a 7 percent by weight solids content lacquer. This lacquer was used to coat sheets of sodium etched Kapton film. After drying for 1 hour at 125°C (257°F) the sheets were bonded together in a press at 750 psi and 250°C (482°F) for 30 minutes. After cooling, they separated easily, indicating that the polyterphenyleneoxide was a poor adhesive for Kapton.

Portions of the polyterphenyleneoxide solution then were mixed with solutions of telomeric xylylene glycol capped with p-toluenesulfonic acid to provide lacquers containing 5 percent and 10 percent by



weight (based on solids) of the xylylene glycol telomer. Each of these mixed lacquers was used to bond together sheets of Kapton film and the laminates were cured for 1-1/2 hours at 750 psi and 250°C (482°F). Both of the laminates qualitatively exhibited peel strengths greater than that obtained from polyterphenyleneoxide alone. However, neither was sufficiently good to warrant quantitative tests.

Each of the three polymer lacquers was also used to coat 20 gage copper wires. The coated wires were air dried between successive applications to provide one wire with a 5-6 mil insulation and one with a 7-8 mil insulation. The former was derived from the lacquer which had only 5 percent xylylene glycol. Wires were then dried for 2 hours at 125°C (257°F) and heated for 2 hours at 250°C (482 F).

Insulation on each of the wires was too brittle to resist cracking when bent. Insulation resistance measurements were nevertheless obtained on the polyterphenyleneoxide which contained 5 percent by weight xylylene glycol. Results of these measurements are shown in Table XVIII and graphically plotted in Figure 21. Because of its very brittle character no further work was carried out.

8. POLYIMIDAZOQUINAZOLINE

Polyimidazoquinazoline (PIQ) resin was also screened. Its solubility in various solvents such as dimethylformamide, dioxane, chloroform and N-methylpyrrolidone was checked. The first and last

Table XVIII. Insulation Resistance of Polyterphenyleneoxide Insulated Wire

Temperature,		Insulation Resistance, Ohms 30 Inch Lengths
°F	°C	
75	24	5×10^{11}
200	93	1×10^{11}
300	149	2×10^{12}
400	204	2×10^{11}
500	260	3×10^{10}
600	316	2×10^{9}
700	371	3×10^{8}

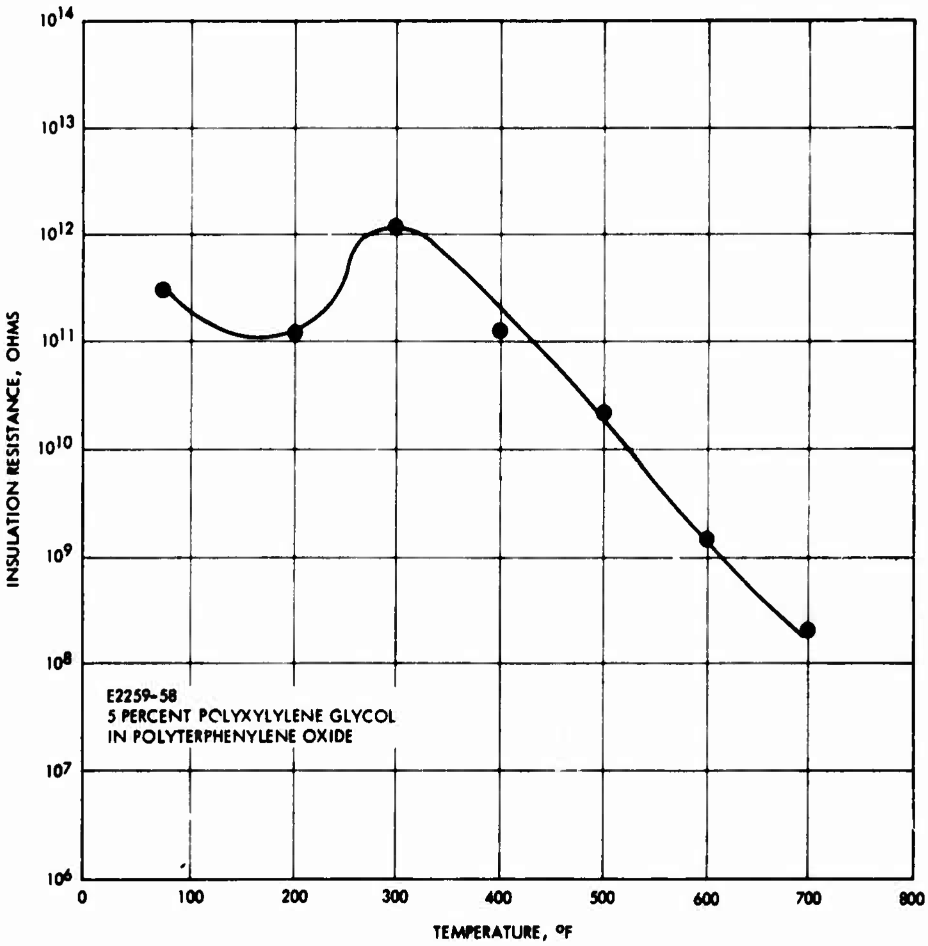


Figure 21. Insulation resistance of polyterphenyleneoxide coated wire as a function of temperature

of these yielded suspensions, but not true solutions. The other two solvents were not even satisfactory dispersion media. N-Methylpyrrolidone appeared to be the best tested. A suspension of 2.3 g of PIQ in 40 ml of solvent was prepared and used to spray-coat 3 by 6 inch sheets of etched 5 mil thick Kapton film. After several coats were applied and individually vacuum dried at 125°C (257°F), the PIQ coating was 1 mil thick. Two sheets then were bonded together under pressure (100 psi) at 427°C (800°F), but the adhesion was poor. The very high cure temperature was used because it appeared to be necessary to achieve flow.

Molded samples of PIQ were tested electrically with the following results:

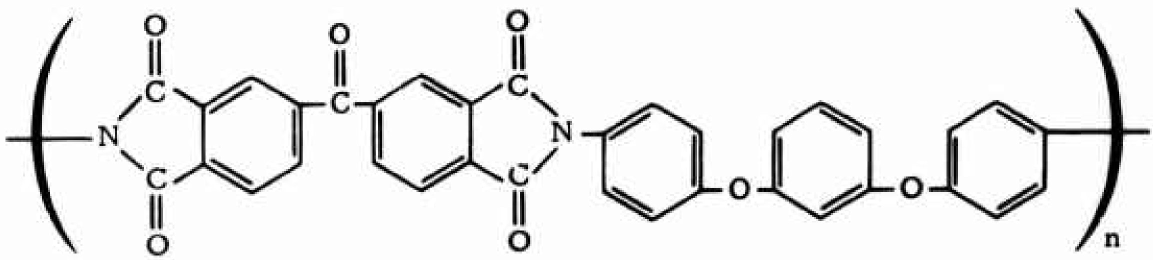
Dielectric constant	2.85 (at 1 kHz, 23°C)
Dissipation factor	Zero
Volume resistivity	1×10^{12} ohm-cm

The dielectric properties looked very good, although a dissipation factor of zero is unreasonably low. This is probably due to the relatively poor quality of the dielectric specimens. High quality molded samples were not obtained since only 8 grams of material could be allocated to the task of learning to mold this resin.

Since the curing temperatures required to process PIQ are in excess of those at which Kapton polyimide has good strength, efforts were made to use the PIQ resin alone. Thus, several attempts were made to coat a wire with PIQ "lacquer" (suspension) and subsequently to cure it on the wire. In all cases the coating was excessively brittle and cracked extensively.

9. 1,3-BIS(p-AMINOPHENOXY)BENZENE BASED POLYIMIDE

A polyimide was prepared from 1,3-bis(p-aminophenoxy)-benzene and benzophenonetetracarboxylic dianhydride and was screened as a potential wire insulation. The structure of this polymer is shown below.



The synthesis of its polyamic acid precursor or prepolymer is described below.

a. <u>Experimental F1812-11</u>

Benzophenonetetracarboxylic acid dianhydride (8.24 grams, 0.02555 mole) dissolved in 38 ml of N-methyl-2-pyrrolidinone was placed in a three necked 300 ml flask fitted with a stirrer, a condenser protected from the atmosphere with a Drierite-filled tube, thermometer and heating mantle. To the flask was added a solution of 1,3-di(4-aminophenoxy)-benzene (10.0 grams, 0.03425 mole) in 38 ml of N,N-dimethylacetamide. The solution was kept at 55°C for 16 hours. The total weight of the lacquer was 90 grams, which was equivalent to a 20 percent solids content.

Pieces of Kapton film (1/2 x 1 inch) were coated with this prepolymer and were air dried for 2 hours, then cured in argon for 6 hours at 288°C (550°F). The samples, along with two controls, were vacuum dried for 16 hours at 65°C, then cooled in vacuum and weighed. Next they were immersed in water overnight, wiped dry and reweighed. They were then redried in vacuum at 65°C. Samples coated with the triphenylene oxide based polyimide were found to have picked up

2.6 percent water, which was lost in total when redried. In contrast, the uncoated Kapton picked up 4.3 percent water, which was lost again upon redrying. The coated Kapton thus showed a significantly lower affinity for water. The difference would, of course, be even greater if a film of pure triphenyleneoxide based polyimide were used.

Kapton tapes (1/2 x 12 x 0.003 inches) next were brush-coated on one side with the prepolymer lacquer, and two ply laminates were prepared and press-cured under the following conditions:

343°C (650°F), 150 psi — 15 minutes
343°C (650°F), 150 psi — 1 hour

These samples showed marginal adhesion. The remaining samples were dried under more stringent conditions, namely, at 149°C (300°F) for 30 minutes, 149°C for 45 minutes, 149°C for 60 minutes. Samples subsequently were cured at 16 psi at 200°C (392°F), 300°C (572°F) and 400°C (752°F). Although no adhesion was observed on samples cured at 200°C, adhesion was observed on those cured at 300°C to 400°C.

Quantitative bond strength measurements thus were warranted. Consequently, sheets of sodium etched Kapton (3 x 6 inches) were brush-coated with the prepolymer lacquer. The samples were dried at 65°C (140°F), first in air, then overnight in vacuum. The samples were cut into 1 x 3 inch strips. One pair was bonded together at 232°C (450°F) and 100 psi for 2 hours. It had many small voids. The remaining samples were dried overnight in vacuum at 100°C (212°F). These were also bonded together at 232°C (450°F) and 100 psi for 2 hours yielding relatively void-free specimens with fair adhesion.

New 3 x 6 inch sheets of coated Kapton were prepared and molded (without cutting) as before; however, this time the samples were postcured overnight at 260°C (500°F). A 1 x 6 inch strip was cut from the larger laminate and the remaining 2 x 6 inch portion was postcured again, this time for 16 hours at 288°C (550°F). The 2 x 6 inch strip was cut in half and one portion was postcured a third time at 316°C (600°F) for 16 hours. Peel tests subsequently were made at 288°C (550°F) and in all cases the substrate failed rather than the adhesive.

Three "pyrrone"-Kapton insulated wires were overcoated with the prepolymer and insulation resistance measurements were made at temperatures to 316°C (600°F). The overcoats were applied by repeated brushings, air drying 4 hours between coats, then oven drying for 16 hours at 60°C (149°F), followed by 4 hours at 200°C (392°F) and 4 hours at 290°C (554°F). Insulation wall thicknesses were (A) 3 mils, (B) 2.5 mils, and (C) 2 mils. Results of these tests are shown in Table XIX. Ambient temperature tests were conducted in anionic wetted water and elevated tests in a molten Cerro-Tru bath. Graphs of these data are shown in Figures 22, 23, and 24. Also shown on each graph is a plot of data previously obtained on the insulated substrate wires prior to being overcoated.

Table XIX. Insulation Resistance versus Temperature of Insulated Wire Overcoated with Bis (p-aminophenoxy)-benzene Based Polyimide

Temperature		Insulation Resistance - 30 Inch Wires, Ohms		
°F	°C	Wire E2259-106A 3 Mil Overcoat On Type II Wire** (E2259-92)	Wire E2259-106B 2.5 Mil Overcoat On Type I Wire* (E2259-91)	Wire E2259-106C 2.0 Mil Overcoat On Type IV Wire *** (E2259-97)
75	24	7×10^{13}	6×10^{13}	1×10^{14}
250	121	3×10^{12}	5×10^{12}	4×10^{12}
300	149	4×10^{11}	6×10^{11}	7×10^{11}
350	177	1×10^{11}	1×10^{11}	2×10^{11}
400	204	2×10^{10}	3×10^{10}	6×10^{10}
500	260	1×10^{10}	1×10^{10}	3×10^{10}
600	316	2×10^{8}	2×10^{8}	4×10^{8}

*Type I wire - single wrapped, Hughes HR-100 on 2 mil Kapton
**Type II wire - double wrapped, Hughes HR-100 on 1 mil Kapton
***Type IV wire - single wrapped, P13N on 1 mil Kapton

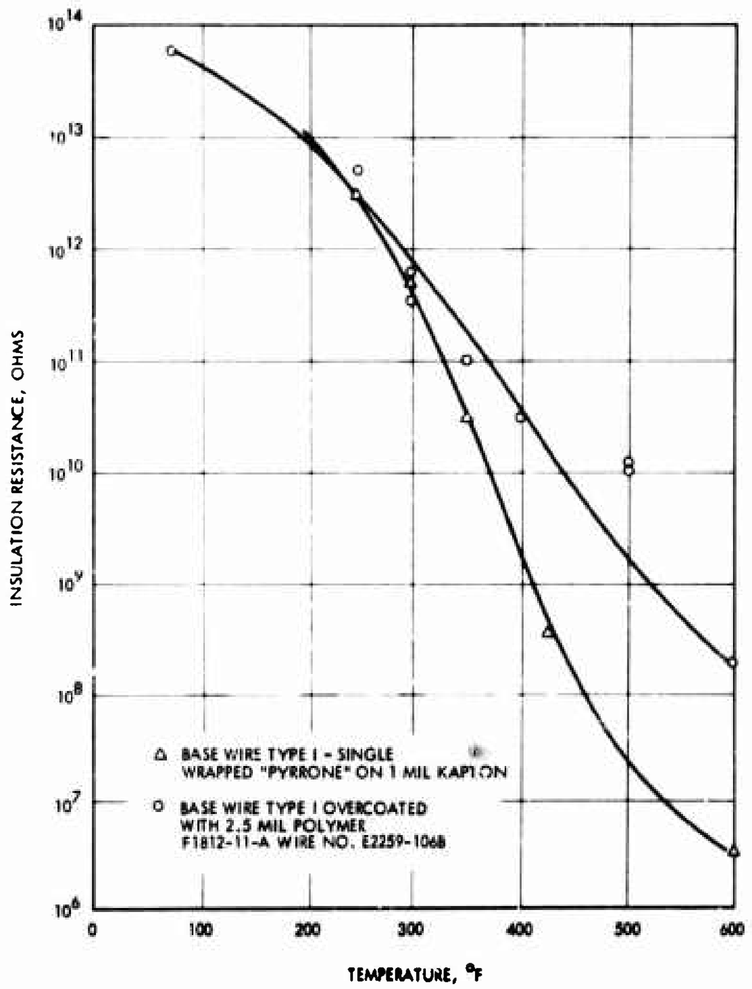


Figure 22. Insulation resistance versus temperature, Type I wire with 1,3-bis-(p-aminophenoxy) benzene based polyimide overcoat

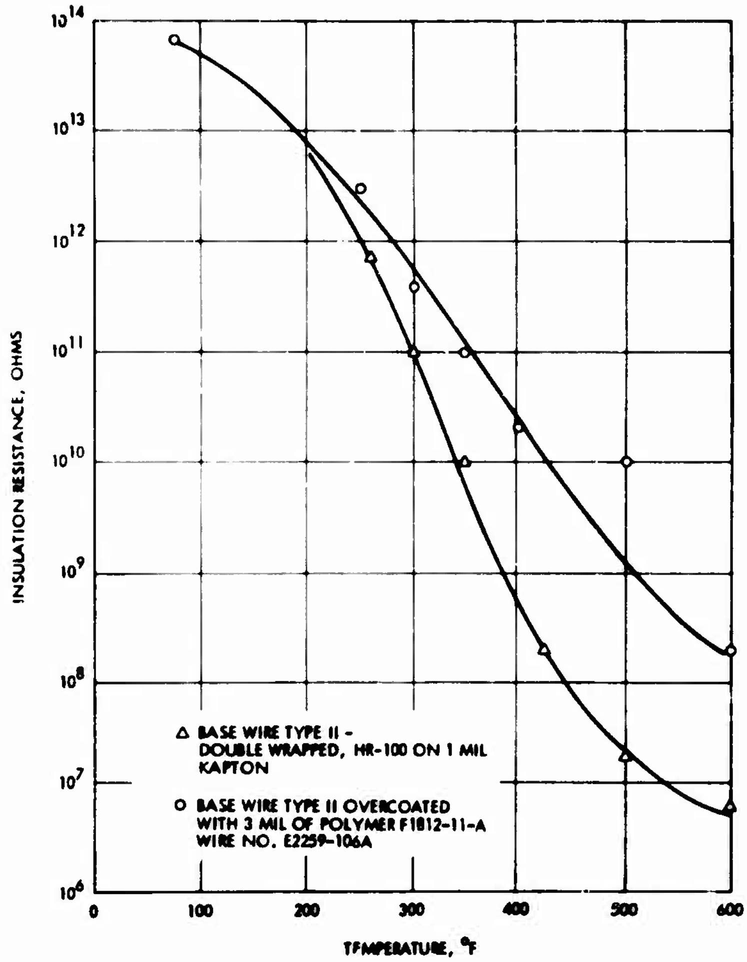


Figure 23. Insulation resistance versus temperature, Type II wire with 1,3-bis-(p-aminophenoxy) benzene based polyimide overcoat

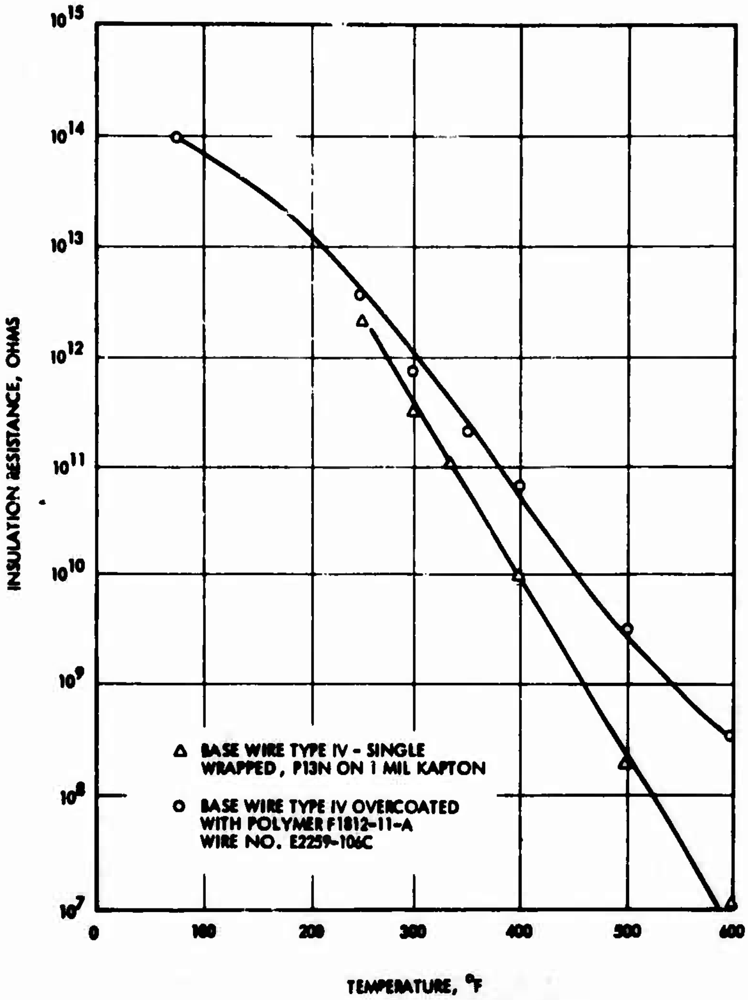


Figure 24. Insulation resistance versus temperature, Type IV wire overcoated with 1,3-bis(p-aminophenoxy) benzene based polyimide

It is evident from these studies that a 1,3-bis(p-aminophenoxy)-benzene based polyimide has several very promising characteristics. First, it has a much lower moisture pickup than conventional polyimides. Second, it has shown good adhesion for Kapton polyimide. Third, it improved the insulation resistance-temperature profile of all three types of wire on which it was coated. Fourth, it can be expected to have excellent dielectric properties, high thermal stability, and less tendency toward brittleness than conventional polyimide.

A comprehensive evaluation of this polymer thus appears to be warranted.

SECTION III

WIRE EVALUATION

Various tests were conducted to compare the different types of wires and determine the degree to which they met Specification MIL-W-81381. Not all wires developed herein were evaluated completely, since several types were fabricated very late in the program. Those not evaluated extensively include V, VII, VIII, and IX.

These are, however, expected to have outstanding properties. Table XX lists the various wires developed on this program and describes their composition and mode of construction. Results of the various life cycle tests are discussed subsequently.

1. HYPERTHERMAL AGING STUDIES

Fourteen types of insulated wire were placed in air ovens at 260°C (500°F), 288°C (550°F), and 316°C (600°F) and their insulation resistance was measured periodically after removing them from the ovens, cooling them to ambient temperature, and testing in anionic wetted water at a potential of 500 Vdc. Analogous tests were also conducted on four wires at 371°C (700°F). Included were the various "pyrrone" HR-100-Kapton insulated wires, P13N-Kapton insulated wires, HR-300-Kapton insulated wires and commercial Teflon FEP-Kapton insulated wires. Results of these tests are presented in subsequent tables. This data has also been illustrated graphically.

These graphs of insulation resistance versus thermal aging period did not all change systematically. Several of them showed rather erratic behavior indicating periodic failures and subsequent spontaneous repair. These data are real and not merely due to errors in test methods. However, the graphical plots may not represent the best method of illustrating the data and possibly smooth curves showing widely scattered data points would be better. Those which were most erratic had a "roller coaster" appearance. In contrast, many of the "curves" had a relatively uniform rate of deterioration although the "points" varied within ± one order of magnitude (for example $10^{12\pm1}$ ohms).

Table XX. Composition of the Various "Pyrrone" -Kapton Insulated Wires

Wire Type	Primary Insulation	Secondary Insulation	Primary Insulation Wall Thickness, mils	Overwrap Thickness Wall, mils
I	"Pyrrone" (HR-100) on 2 mil Kapton, single wrap, 50% overlap. Reference # E2259-91	None	6.5	0
IA	"Pyrrone" (HR-100) on 2 mil Kapton, single wrap, 50% overlap. Reference # E2259-91	2 mil TFE, single wrap, 50% overlap. Reference # E2259-96A	6.5	2.5 ± 0.5
IB	"Pyrrone" (HR-100) on 2 mil Kapton, single wrap, 50% overlap. Reference # E2259-91	2 mil TFE, double reversed wrap, 50% overlap. Reference # E2259-96B	6.5	6.5 ± 0.5
IC	"Pyrrone" (HR-100) on 2 mil Kapton, single wrap, 50% overlap. Reference # E2259-91	1 mil TFE/FEP, double reversed wrap, 50% overlap. Reference # E2259-96C	6.5	12 ± 0.5
ID	"Pyrrone" (HR-100) on 3 mil Kapton, single wrap, 50% overlap	Perfluoroalkylenetriazine		
II	"Pyrrone" (HR-100) on 1 mil Kapton, double reversed wrap, 50% overlap. Reference # E2259-92	None	8.5	0
IIA	"Pyrrone" (HR-100) on 1 mil Kapton, double reversed wrap, 50% overlap. Reference # E2259-92	2 mil TFE, single wrap, 50% overlap. Reference # E2259-96A	8.5	2.5 ± 0.5
IIB	"Pyrrone" (HR-100) on 1 mil Kapton, double reversed wrap, 50% overlap. Reference # E2259-92	2 mil TFE, double reversed wrap, 50% overlap. Reference # E2259-96B	8.5	6.5 ± 0.5
IIC	"Pyrrone" (HR-100) on 1 mil Kapton, double reversed wrap, 50% overlap. Reference # E2259-92	1-mil TFE/FEP, double reversed wrap, 50% overlap. Reference # E2259-96C	8.5	12 ± 0.5
IV	P13N polyimide on 1 mil Kapton, single wrap, approx. 50% overlap.	None	3.0	0
IVA	Reference # E2259-97	2-mil TFE, single wrap, 50% overlap. Reference # E2259-101	3.0	≈2.5
IVB	Reference # E2259-97	2-mil TFE, double reversed wrap	3.0	≈6.5
V	P13N polyimide on 1 mil Kapton, double reversed wrap, each layer with a 50% overlap. Reference # E2259-105	None	6.0	0
VI	Milo Carolina Wire & Cable, Inc. MIL-W-81381/3-20 single wrapped, FEP Teflon-Kapton	FEP	11.5	2.5
VIA	Milo Carolina Wire & Cable Inc. MIL-W-81381/1-20, double wrapped, FEP Teflon-Kapton	FEP	6.5	1.0
VII	"Pyrrone" (HR-100) on 1 mil Kapton, triple reversed wrap	None	9.0	0
VIII	HR-300 on 1 mil Kapton, double reversed wrap, 50% overlap	None	6.0	0
IX	HR-300 on 1 mil Kapton, triple reversed wrap	None	9.0	0

Teflon overwrapped wires type IA, IB, IC, IIB, and IIC, produced from "pyrrone" HR-100 on 2 mil Kapton and "pyrrone" HR-100 on 1 mil Kapton (double wrapped) were essentially equivalent and showed little significant degradation over the 1000 hour period at either 500°F or 550°F. Only one of the TFE overcoated wires behaved erratically (IIA). This wire had a single wrap of 2 mil TFE on the double wrapped "pyrrone"-2 mil Kapton. The very erratic data may not be significant, however, since the wire appeared defective visually.

Of the wires which were not overcoated with TFE Teflon the best were II and V. The former is the double wrapped "pyrrone" HR-100-Kapton insulated wire whereas the latter is the double wrapped P13N-Kapton insulated wire. The first of these had an ambient temperature insulation resistance value of approximately $10^{12\pm1}$ ohms over a 2000-hour aging period at 288°C (550°F) and over a 1000 hour period at 316°C (600°F). In comparison Wire V also had a similar insulation resistance retention over a 1000 hour period at 288°C (550°F), which was the total test duration in this case, and over a 1000 hour period at 316°C (600°F).

Insulation resistance measurements in all of these aging studies were made at ambient temperature after removing the wires from the oven and cooling. Tests were made at 500 Vdc with the wires submerged in anionic wetted water. They were then dried and replaced in the oven. The best wires saw 19 cycles over the 1019 hour test period at 316°C (600°F) and 25 cycles over the 2456 hour test period at 288°C (550°F). A more detailed discussion of these results is presented below.

Virtually all elevated temperature insulation resistance measurements were made in a molten Cerro-Tru (Bi-Sn) alloy. The reason for selecting this bath was that it guaranteed good wire-bath contact. It was also a more severe test than the one used in our prior work, namely, one in which wires were tightly wrapped in aluminum foil and tested in an oven. The difference in the two test methods is evident from the following experiments.

The insulation resistance of two Teflon TFE overwrapped HR-100 "pyrrone"-Kapton insulated wires (Types IA and IB) was measured with the wires immersed in a molten metal bath (Cerro-Tru) at various temperatures between 140°C (284°F) and 380°C (716°F) after an initial ambient temperature test in wetted water. Results of these tests are shown in Table XXI and are illustrated in Figures 25 and 26. It will be noted in the figures that in the case of the wire having only one layer of Teflon TFE the insulation resistance at all temperatures above 149°C (300°F) differed whereas the results were essentially the same on the Teflon double wrapped wires regardless of the test method.

Table XXI. Insulation Resistance of "Pyrrone"-Kapton Insulated Wires Measured in Molten Metal Bath

Temperature		Insulation Resistance,* Ohms	
°F	°C	Type IA	Type IB
73 (in H_2O)	23 (in H_2O)	5×10^{7}	5×10^{13}
284	140	1×10^{12}	3×10^{12}
338	170	2×10^{11}	2×10^{13}
392	200	1×10^{10}	5×10^{12}
446	230	2×10^{9}	1×10^{12}
500	260	8×10^{8}	1×10^{11}
554	290	7×10^{7}	4×10^{10}
608	320	3×10^{7}	2×10^{10}
662	350	4×10^{7}	2×10^{10}
716	380	1×10^{7}	2×10^{10}

*30 inch lengths.

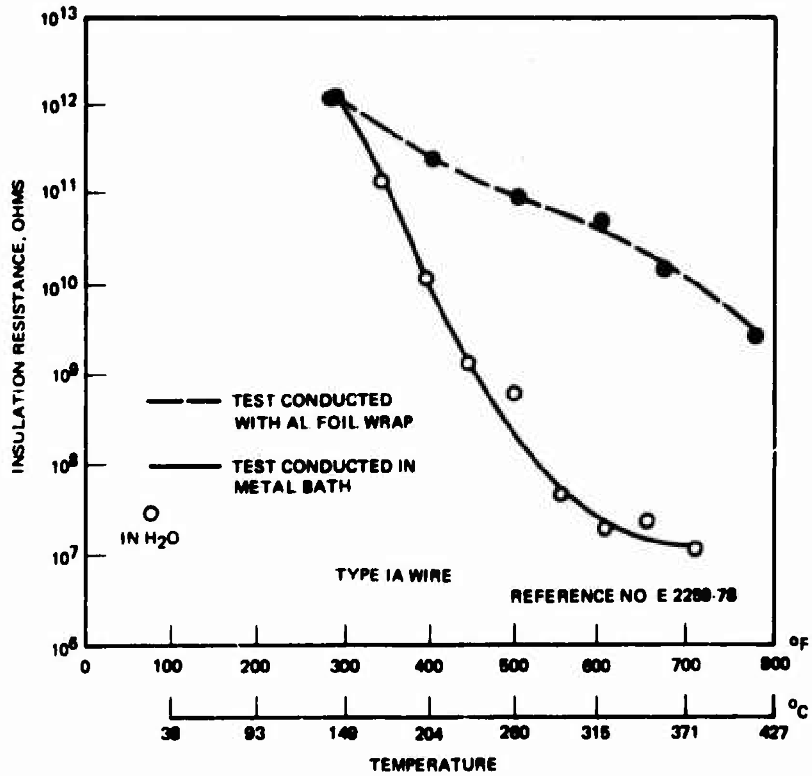


Figure 25. Insulation resistance of "pyrrone" - Kapton insulated wire with single Teflon overcoat (Wire Type IA)

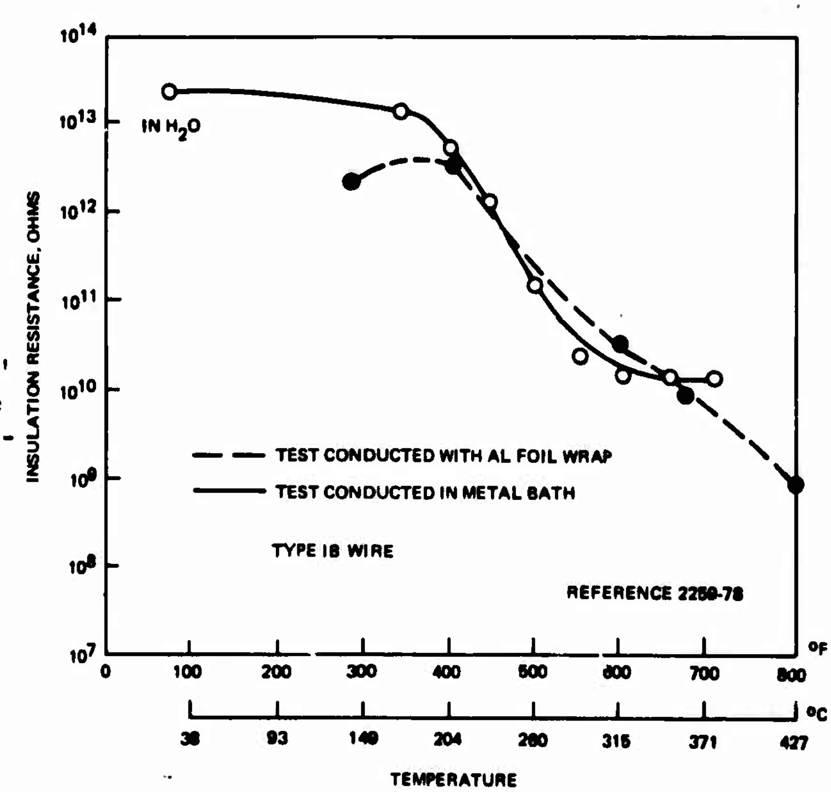


Figure 26. Insulation resistance of "pyrrone" - Kapton insulated wire with double Teflon overcoat (Wire Type IB)

a. Air Aging at 260°C (500°F)

In aging tests conducted at 260°C (500°F) (Table XXII), the single-wrapped P13N-Kapton wire (IV) was found to be somewhat erratic, although its insulation resistance remained between 10^{11} and 10^{14} ohms for about 1400 hours. By the time the test reached 1700 hours this value had fallen to near 10^5 ohms. Its double wrapped counterpart (V) was much more stable and had an insulation resistance of 10^{14} ohms after 1056 hours, at which time the test was terminated (Figure 27). Based on the results of the higher temperature test described subsequently, however, wire V would be expected to have a virtually indefinite life expectancy at this temperature.

Wire I, the single wrapped"pyrrone" (HR-100)-Kapton, had a rather uniform but slight deterioration between 200 and 2200 hours. During this time its insulation resistance dropped from 3×10^8 ohms to 10^7 ohms and at 2240 hours it was found to be shorted. When wire I was overwrapped with a single layer of Teflon TFE to yield wire IA, a significant improvement was noted in that IA had an insulation resistance of 10^{14} ohms between 400 and 1000 hours (Figure 28). It appeared to undergo a serious degradation after 200 hours but this repaired itself and the wire stabilized until 1000 hours at which point irreversible rapid deterioration occurred.

A double layer TFE overwrapped analogue of this wire (IB) and a Teflon FEP/TFE overwrapped version (IC) were very stable and showed no significant changes through the 2200 hour test period (Figure 29).

Double wrapped "pyrrone" (HR-100)-Kapton insulated wire (II) also showed no significant change over the 2200 hour test period (Figure 30). Furthermore, the TFE double overwrapped HR-100-Kapton wire (IIB) and the FEP/TFE overwrapped analogue (IIC) also showed no significant change over the 2200 hour test period (Figure 31). In contrast to these is the wire IIA, which is a single layer TFE overwrapped version of II. This wire, as shown in Figure 32, was very erratic and underwent an irreversible breakdown after 1000 hours.

Table XXII. Insulation Resistance as a Function of Thermal Aging in Air at 500°F*

Time Hours	I Ohms	IA Ohms	IB Ohms	IC Ohms	II Ohms	IIA Ohms	IIB Ohms	IIC Ohms	IV Ohms	Time Hours	V Ohms
0 (a)	8×10^{12}	3×10^{13}	6×10^{13}	6×10^{13}	5×10^{13}	4×10^{13}	6×10^{13}	7×10^{13}	5×10^{13}	0 (a)	8×10^{13}
(b)	3×10^{13}	5×10^{13}	6×10^{13}	6×10^{13}	4×10^{13}	3×10^{13}	8×10^{13}	7×10^{13}	5×10^{13}	(b)	7×10^{13}
116 (a)	3×10^{13}	6×10^{13}	3×10^{14}	2×10^{14}	1×10^{14}	5×10^{13}	2×10^{14}	3×10^{14}	8×10^{13}	48 (a)	5×10^{13}
(b)	3×10^{13}	7×10^{13}	2×10^{14}	2×10^{14}	8×10^{13}	3×10^{13}	2×10^{14}	3×10^{14}	7×10^{13}	(b)	5×10^{13}
214 (a)	4×10^{8}	1×10^{13}	3×10^{14}	2×10^{14}	2×10^{13}	4×10^{13}	2×10^{14}	2×10^{14}	3×10^{13}	216 (a)	1×10^{11}
(b)	4×10^{8}	8×10^{13}	2×10^{14}	2×10^{14}	1×10^{12}	8×10^{13}	2×10^{14}	2×10^{14}	8×10^{13}	(b)	5×10^{12}
281 (a)	4×10^{8}	8×10^{7}	9×10^{13}	3×10^{14}	1×10^{11}	3×10^{7}	5×10^{14}	5×10^{14}	4×10^{10}	384 (a)	1×10^{11}
(b)	8×10^{8}	8×10^{9}	5×10^{14}	5×10^{14}	6×10^{10}	7×10^{7}	5×10^{14}	5×10^{14}	6×10^{10}	(b)	1×10^{12}
392 (a)	3×10^{8}	5×10^{10}	3×10^{14}	2×10^{14}	2×10^{11}	2×10^{12}	3×10^{14}	3×10^{14}	3×10^{11}	552 (a)	3×10^{11}
(b)	1×10^{11}	4×10^{8}	2×10^{14}	3×10^{14}	3×10^{10}	1×10^{12}	3×10^{14}	2×10^{14}	4×10^{11}	(b)	1×10^{10}
464 (a)	6×10^{8}	1×10^{14}	5×10^{14}	3×10^{14}	7×10^{11}	3×10^{13}	5×10^{14}	3×10^{14}	8×10^{11}	720 (a)	2×10^{12}
(b)	5×10^{10}	5×10^{8}	3×10^{14}	3×10^{14}	2×10^{11}	1×10^{12}	3×10^{14}	3×10^{14}	1×10^{12}	(b)	3×10^{13}
560 (a)	3×10^{8}	1×10^{11}	3×10^{14}	3×10^{14}	4×10^{11}	4×10^{7}	3×10^{14}	5×10^{14}	1×10^{9}	1056 (a)	1×10^{14}
(b)	3×10^{9}	1×10^{8}	5×10^{14}	3×10^{14}	1×10^{12}	7×10^{8}	5×10^{14}	3×10^{14}	7×10^{9}	(b)	2×10^{8}
632 (a)	2×10^{8}	1×10^{14}	3×10^{14}	3×10^{14}	3×10^{11}	2×10^{12}	5×10^{14}	5×10^{14}	8×10^{11}		
(b)	6×10^{8}	8×10^{7}	5×10^{14}	5×10^{14}	3×10^{11}	2×10^{13}	5×10^{14}	5×10^{14}	6×10^{11}		
728 (a)	2×10^{8}	1×10^{14}	3×10^{14}	5×10^{14}	5×10^{12}	6×10^{7}	5×10^{14}	5×10^{14}	2×10^{14}		
(b)	1×10^{8}	8×10^{7}	3×10^{14}	5×10^{14}	1×10^{13}	1×10^{8}	5×10^{14}	5×10^{14}	6×10^{12}		
800 (a)	7×10^{7}	2×10^{14}	5×10^{14}	5×10^{14}	2×10^{14}	8×10^{12}	5×10^{14}	5×10^{14}	2×10^{13}		
(b)	2×10^{8}	1×10^{12}	5×10^{14}	5×10^{14}	8×10^{13}	4×10^{8}	5×10^{14}	3×10^{14}	4×10^{13}		
896 (a)	6×10^{7}	7×10^{13}	3×10^{14}	2×10^{14}	3×10^{11}	2×10^{12}	2×10^{14}	2×10^{14}	5×10^{11}		
(b)	5×10^{7}	9×10^{9}	3×10^{14}	3×10^{14}	1×10^{12}	6×10^{7}	3×10^{14}	1×10^{14}	2×10^{11}		
968 (a)	4×10^{7}	6×10^{13}	5×10^{14}	3×10^{14}	1×10^{12}	5×10^{13}	5×10^{14}	3×10^{14}	2×10^{12}		
(b)	1×10^{8}	3×10^{8}	2×10^{14}	5×10^{14}	3×10^{13}	8×10^{11}	5×10^{14}	5×10^{14}	3×10^{12}		
1064 (a)	2×10^{7}	3×10^{12}	5×10^{14}	5×10^{14}	2×10^{12}	9×10^{5}	3×10^{14}	5×10^{14}	6×10^{12}		
(b)	3×10^{7}	7×10^{9}	5×10^{14}	5×10^{14}	5×10^{12}	4×10^{8}	5×10^{14}	5×10^{14}	3×10^{12}		
1136 (a)	2×10^{7}	1×10^{11}	3×10^{14}	5×10^{14}	3×10^{11}	7×10^{5}	5×10^{14}	5×10^{14}	3×10^{11}		
(b)	2×10^{7}	6×10^{7}	5×10^{14}	5×10^{14}	3×10^{11}	4×10^{7}	3×10^{14}	5×10^{14}	1×10^{14}		
1232 (a)	2×10^{7}	3×10^{9}	3×10^{14}	3×10^{14}	1×10^{14}	3×10^{4}	3×10^{14}	3×10^{14}	8×10^{13}		
(b)	2×10^{7}	1×10^{8}	5×10^{14}	5×10^{14}	2×10^{14}	2×10^{6}	3×10^{14}	3×10^{14}	1×10^{14}		
1400 (a)	1×10^{7}	2×10^{6}	2×10^{14}	2×10^{14}	2×10^{12}	2×10^{4}	2×10^{14}	2×10^{14}	2×10^{9}		
(b)	3×10^{7}	8×10^{5}	2×10^{14}	2×10^{14}	7×10^{10}	1×10^{6}	3×10^{14}	2×10^{14}	1×10^{13}		
1568 (a)	7×10^{6}	3×10^{4}	2×10^{14}	3×10^{14}	1×10^{13}	1×10^{4}	3×10^{14}	3×10^{14}	2×10^{12}		
(b)	6×10^{6}	2×10^{4}	3×10^{14}	3×10^{14}	7×10^{11}	4×10^{4}	3×10^{14}	3×10^{14}	2×10^{8}		
1736 (a)	8×10^{6}	Shorted	3×10^{14}	2×10^{14}	1×10^{12}	Shorted	5×10^{14}	3×10^{14}	3×10^{5}		
(b)	2×10^{4}	Shorted	2×10^{14}	3×10^{14}	3×10^{11}	Shorted	3×10^{14}	3×10^{14}	2×10^{5}		
1904 (a)	3×10^{10}	...	2×10^{13}	1×10^{13}	2×10^{12}	...	3×10^{13}	3×10^{13}	Shorted		
(b)	Shorted	...	4×10^{12}	3×10^{13}	7×10^{12}	...	5×10^{13}	6×10^{13}	Shorted		
2240 (a)	Shorted	...	1×10^{7}	6×10^{13}	2×10^{11}	...	1×10^{14}	1×10^{14}	...		
(b)	...	...	5×10^{13}	1×10^{13}	3×10^{6}		1×10^{14}	8×10^{13}	...		

*Insulation resistance measurements were made at ambient temperature.

PRECEDING PAGE BLANK-NOT FILMED

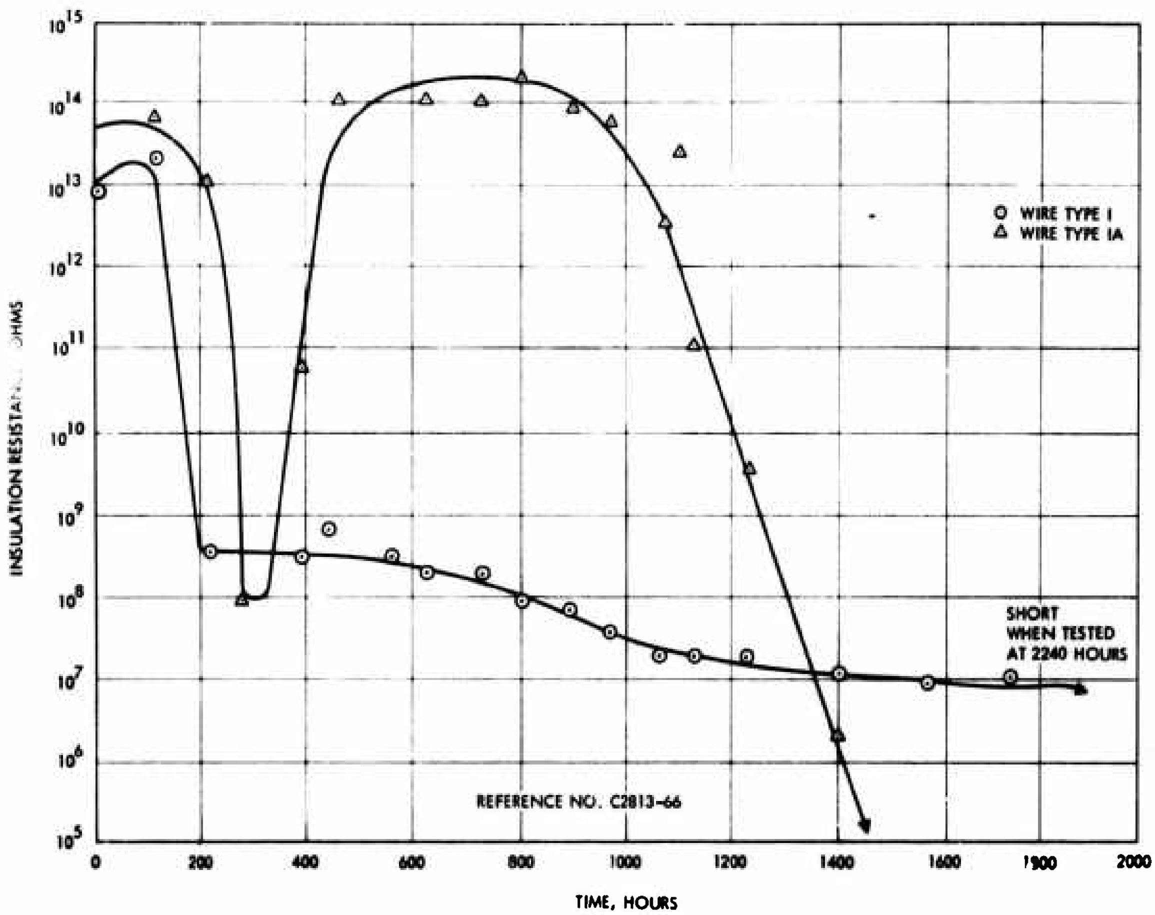


Figure 28. Insulation resistance as a function of thermal aging in air at 500°F – Wires type I and IA

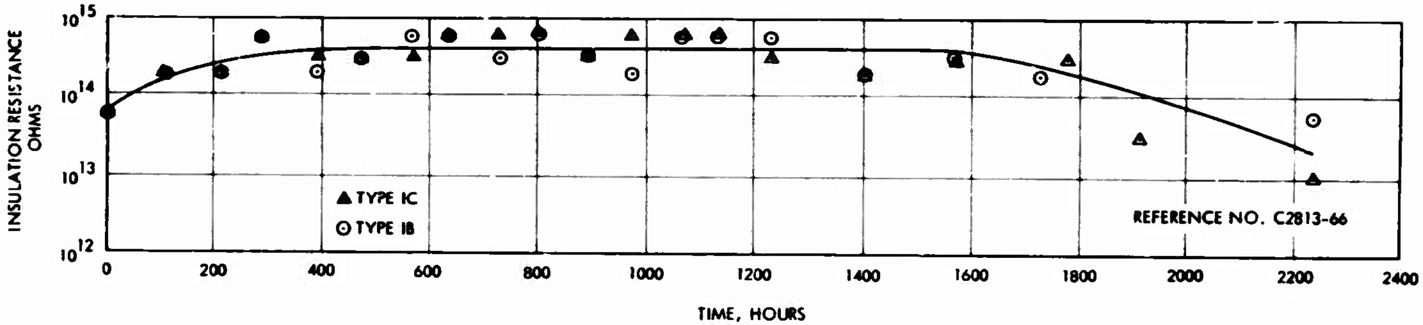


Figure 29. Insulation resistance as a function of thermal aging in air at 500°F – Wires type IB and IC

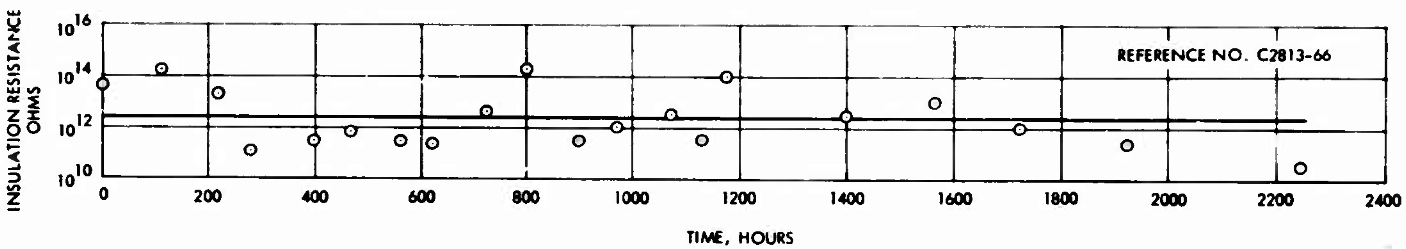


Figure 30. Insulation resistance as a function of thermal aging in air at 500°F – Wire type II

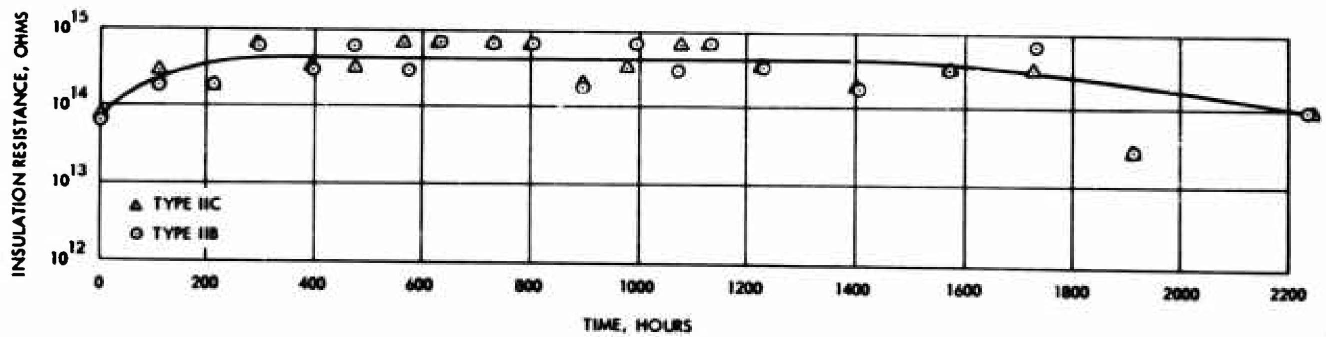


Figure 31. Insulation resistance as a function of thermal aging in air at 500°F – Wires type IIB and IIC

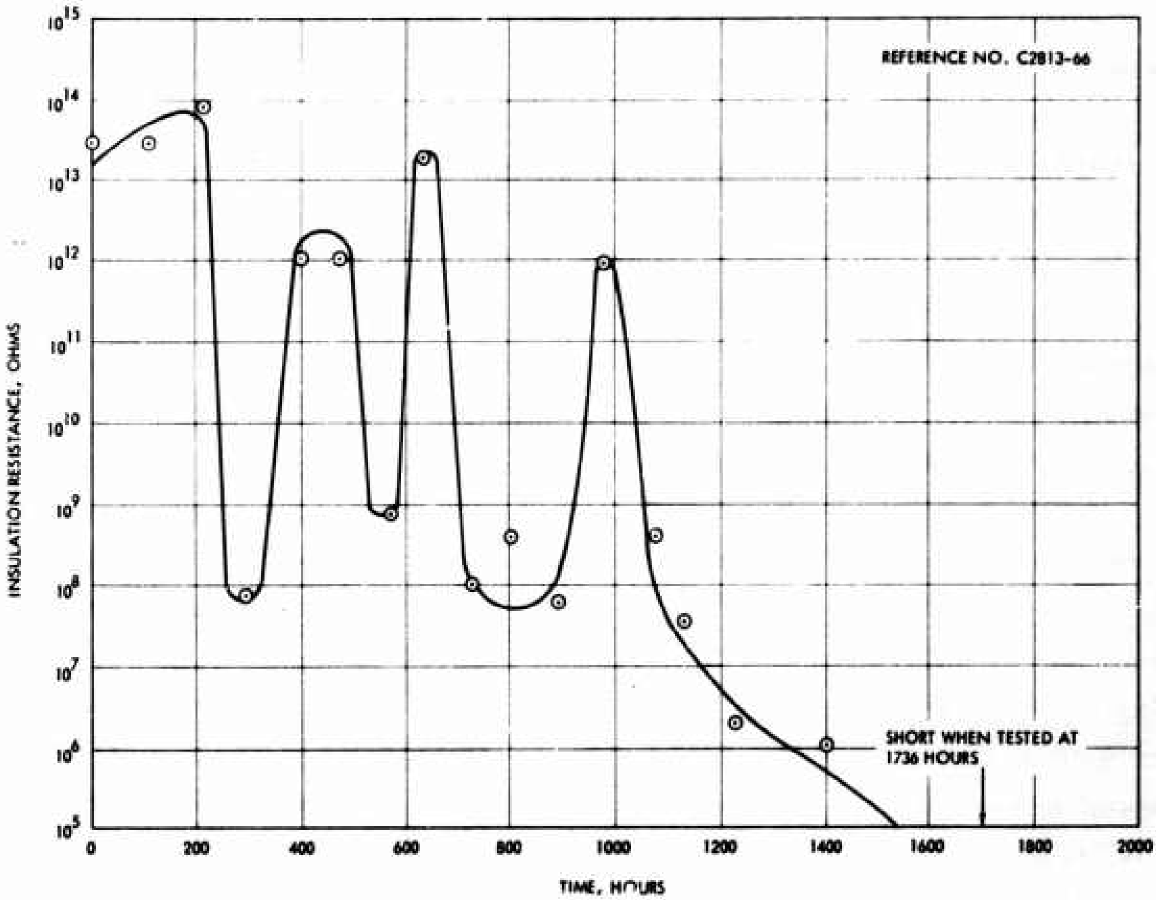


Figure 32. Insulation resistance as a function of thermal aging in air at 500°F — Wire type IIA

However, this performance is probably not indicative of the potential of this type of wire but can probably be attributed to an excessively long TFE sintering period which was carried out at 1000°F.

b. Air Aging at 288°C (550°F)

Wires were also aged in air at 288°C (550°F) (Table XXIII). At this temperature the single wrapped "pyrrone"-Kapton type wire (I) showed gross degradation after 600 hours, whereas its TFE overwrapped counterpart (IA) showed irreversible gross degradation after 800 hours with very erratic results much before this time (Figures 33 and 34). A double overwrap of TFE on the Type I wire provided a significantly better wire (IB) which performed well for 1100 hours but showed gross degradation at 1200 hours. In contrast, when wire I was overwrapped with FEP/TFE Teflon, it yielded wire IC which showed no gross degradation for 2400 hours (Figure 35).

The double wrapped "pyrrone" (HR-100)-Kapton (Type II) also performed extremely well at this temperature and showed no gross degradation for over 2200 hours (Figure 35) even without a Teflon overcoat. Both the TFE and the FEP/TFE overcoated double wrapped "pyrrone"-Kapton wires IIB and IIC also showed no significant change over the 2400 hour period. Consequently, no evident advantages accrued as a result of the overcoating, other than increasing the mean insulation resistance value by about one order of magnitude (from 10^{13} to 5×10^{14} ohms) (Figure 36). This relatively insignificant increase was achieved, however, at the expense of much added weight.

Single wrapped P13N-Kapton wire (Type IV) showed gross degradation after 400 hours (Figure 37); however, its double wrapped counterpart (Type V) (Figure 38) showed a change of only three orders of magnitude after 1000 hours ($5 \times 10^{13} \rightarrow 10^{10}$ ohms). When the single wrapped P13N-Kapton wire (IV) was overcoated with one or two layers of TFE Teflon to provide wires IVA and IVB, respectively, the resultant wires showed no significant degradation after 1700 hours, which was the duration of the test (Figure 39).

Table XXIII. Insulation Resistance as a Function of Thermal Aging in Air at 550°F*

Time Hours	I Ohms	IA Ohms	IB Ohms	IC Ohms	II Ohms	IIA Ohms	IIB Ohms	IIC Ohms	Time Hours	IV Ohms	Time Hours	IVA Ohms	IVB Ohms	Time Hours	V Ohms	Time Hours	VI Ohms
0 (a)	7×10^{13}	7×10^{11}	5×10^{13}	2×10^{14}	3×10^{13}	1×10^{13}	2×10^{14}	2×10^{14}	0	5×10^{13}	0 (a)	1×10^{14}	1×10^{14}	0 (a)	5×10^{13}	0 (a)	6×10^{10}
(b)	1×10^{13}	3×10^{12}	1×10^{14}	1×10^{14}	5×10^{13}	2×10^{13}	2×10^{14}	2×10^{14}	40	5×10^{13}	(b)	6×10^{13}	1×10^{14}	(b)	6×10^{13}	(b)	7×10^{10}
40 (a)	4×10^{13}	4×10^{8}	2×10^{14}	2×10^{14}	6×10^{13}	6×10^{13}	2×10^{14}	3×10^{14}	112	1×10^{12}	41 (a)	6×10^{13}	2×10^{14}	48 (a)	4×10^{13}	48 (a)	8×10^{13}
(b)	7×10^{13}	1×10^{14}	1×10^{14}	3×10^{14}	6×10^{13}	6×10^{13}	3×10^{14}	3×10^{14}	156	5×10^{13}	(b)	6×10^{13}	5×10^{14}	(b)	5×10^{13}	(b)	4×10^{12}
112 (a)	3×10^{12}	8×10^{6}	2×10^{14}	1×10^{14}	1×10^{14}	1×10^{7}	2×10^{12}	2×10^{12}	216	1×10^{13}	113 (a)	2×10^{13}	5×10^{14}	216 (a)	7×10^{12}	96 (a)	8×10^{13}
(b)	1×10^{12}	1×10^{7}	2×10^{12}	2×10^{12}	1×10^{12}	5×10^{6}	1×10^{12}	2×10^{12}	334	4×10^{13}	(b)	3×10^{13}	5×10^{14}	(b)	2×10^{12}	(b)	3×10^{14}
156 (a)	8×10^{11}	7×10^{7}	1×10^{14}	8×10^{13}	2×10^{12}	3×10^{8}	5×10^{13}	8×10^{12}	440	2×10^{13}	209 (a)	3×10^{13}	5×10^{14}	384 (a)	1×10^{12}	288 (a)	3×10^{13}
(b)	5×10^{14}	6×10^{7}	8×10^{13}	7×10^{13}	5×10^{11}	1×10^{8}	8×10^{13}	8×10^{13}	512	4×10^{8}	(b)	4×10^{13}	5×10^{14}	(b)	7×10^{11}	(b)	Shorted
216 (a)	7×10^{12}	1×10^{13}	2×10^{13}	2×10^{13}	1×10^{13}	4×10^{8}	1×10^{13}	2×10^{13}	560	2×10^{8}	281 (a)	1×10^{14}	5×10^{14}	552 (a)	3×10^{12}	652 (a)	1×10^{14}
(b)	6×10^{12}	2×10^{8}	2×10^{13}	2×10^{13}	2×10^{13}	6×10^{7}	2×10^{13}	2×10^{13}	608	3×10^{8}	(b)	7×10^{13}	5×10^{14}	(b)	8×10^{13}	(b)	...
334 (a)	3×10^{13}	3×10^{10}	8×10^{13}	7×10^{13}	5×10^{13}	1×10^{8}	1×10^{14}	8×10^{13}	680	1×10^{9}	377 (a)	8×10^{13}	1×10^{14}	720 (a)	4×10^{10}		
(b)	6×10^{12}	2×10^{12}	8×10^{13}	8×10^{13}	3×10^{13}	3×10^{13}	8×10^{13}	8×10^{13}	776	3×10^{8}	(b)	7×10^{13}	1×10^{14}	(b)	6×10^{12}		
440 (a)	2×10^{13}	2×10^{7}	2×10^{14}	3×10^{14}	2×10^{13}	6×10^{7}	6×10^{13}	2×10^{14}	848	9×10^{8}	449 (a)	3×10^{13}	3×10^{14}	1056 (a)	1×10^{10}		
(b)	7×10^{11}	3×10^{13}	3×10^{14}	3×10^{14}	2×10^{13}	2×10^{13}	3×10^{14}	1×10^{14}	944	2×10^{5}	(b)	6×10^{13}	5×10^{14}	(b)	1×10^{10}		
512 (a)	6 10^{10}	2×10^{7}	5×10^{14}	5×10^{14}	2×10^{11}	8×10^{7}	2×10^{14}	3×10^{14}	1016	4×10^{4}	545 (a)	1×10^{14}	3×10^{14}				
(b)	2×10^{11}	5×10^{6}	5×10^{14}	5×10^{14}	4×10^{8}	5×10^{12}	5×10^{14}	5×10^{14}	1112	2×10^{4}	(b)	4×10^{13}	5×10^{13}				
560 (a)	2×10^{14}	2×10^{8}	5×10^{14}	5×10^{14}	7×10^{11}	4×10^{7}	5×10^{14}	5×10^{14}	1184	3×10^{3}	617 (a)	5×10^{14}	5×10^{13}				
(b)	3×10^{11}	2×10^{7}	5×10^{14}	5×10^{14}	1×10^{12}	4×10^{7}	5×10^{14}	5×10^{14}	1280	4×10^{3}	(b)	8×10^{13}	3×10^{8}				
608 (a)	6×10^{11}	3×10^{8}	3×10^{14}	3×10^{14}	1×10^{12}	9×10^{7}	3×10^{14}	3×10^{14}	1352	2×10^{3}	713 (a)	3×10^{14}	5×10^{14}				
(b)	8×10^{11}	3×10^{12}	1×10^{14}	3×10^{14}	4×10^{12}	4×10^{12}	3×10^{14}	3×10^{14}	1440	2×10^{3}	(b)	1×10^{13}	2×10^{7}				
680 (a)	7×10^{12}	2×10^{12}	3×10^{14}	3×10^{14}	1×10^{12}	6×10^{7}	3×10^{14}	5×10^{14}	1616	6×10^{2}	881 (a)	3×10^{14}	5×10^{14}				
(b)	7×10^{12}	9×10^{12}	2×10^{14}	5×10^{14}	1×10^{13}	2×10^{13}	3×10^{14}	3×10^{14}	1784	Shorted	(b)	4×10^{7}	1×10^{7}				
776 (a)	1×10^{11}	9×10^{6}	3×10^{14}	5×10^{14}	2×10^{12}	3×10^{13}	2×10^{14}	1×10^{9}			1049 (a)	5×10^{14}	2×10^{14}				
(b)	8×10^{8}	1×10^{7}	5×10^{8}	5×10^{14}	5×10^{10}	7×10^{7}	5×10^{14}	3×10^{13}			(b)	2×10^{5}	2×10^{4}				
848 (a)	3×10^{12}	1×10^{11}	3×10^{14}	5×10^{14}	2×10^{12}	3×10^{7}	1×10^{14}	5×10^{14}			1217 (a)	5×10^{14}	5×10^{14}				
(b)	3×10^{8}	6×10^{13}	5×10^{14}	5×10^{14}	4×10^{13}	8×10^{7}	5×10^{14}	3×10^{14}			(b)	9×10^{5}	3×10^{5}				
944 (a)	5×10^{8}	4×10^{7}	2×10^{14}	[illegible] $\times10^{14}$	4×10^{13}	2×10^{7}	1×10^{14}	3×10^{14}			1385 (a)	5×10^{13}	2×10^{13}				
(b)	1×10^{7}	7×10^{7}	1×10^{14}	3×10^{14}	8×10^{13}	3×10^{7}	3×10^{14}	3×10^{14}			(b)	3×10^{10}	Shorted				
1016 (a)	2×10^{8}	4×10^{13}	5×10^{14}	5×10^{14}	6×10^{13}	8×10^{6}	6×10^{8}	5×10^{14}			1721 (a)	3×10^{14}	1×10^{14}				
(b)	2×10^{6}	2×10^{8}	3×10^{14}	5×10^{14}	7×10^{13}	2×10^{7}	5×10^{14}	5×10^{14}			(b)	Shorted	...				
1112 (a)	7×10^{6}	2×10^{9}	1×10^{14}	1×10^{14}	4×10^{13}	1×10^{6}	7×10^{8}	1×10^{14}									
(b)	7×10^{6}	5×10^{6}	1×10^{14}	1×10^{14}	2×10^{13}	5×10^{6}	1×10^{14}	3×10^{14}									
1184 (a)	3×10^{6}	4×10^{8}	2×10^{8}	3×10^{14}	1×10^{12}	3×10^{6}	1×10^{14}	3×10^{14}									
(b)	8×10^{6}	6×10^{8}	1×10^{8}	3×10^{14}	3×10^{12}	3×10^{6}	3×10^{14}	3×10^{14}									
1280 (a)	2×10^{6}	7×10^{6}	3×10^{7}	5×10^{14}	3×10^{12}	1×10^{6}	6×10^{7}	1×10^{14}									
(b)	6×10^{6}	9×10^{7}	2×10^{7}	2×10^{9}	2×10^{12}	2×10^{6}	5×10^{14}	5×10^{14}									
1352 (a)	2×10^{5}	3×10^{5}	4×10^{7}	5×10^{14}	3×10^{13}	2×10^{4}	3×10^{14}	3×10^{14}									
(b)	2×10^{6}	9×10^{6}	1×10^{7}	3×10^{14}	1×10^{13}	6×10^{5}	3×10^{14}	2×10^{14}									
1440 (a)	2×10^{4}	2×10^{4}	2×10^{7}	3×10^{14}	1×10^{7}	2×10^{4}	4×10^{8}	5×10^{14}									
(b)	2×10^{6}	4×10^{5}	7×10^{6}	3×10^{14}	5×10^{13}	4×10^{5}	3×10^{14}	5×10^{14}									
1616 (a)	1×10^{4}	7×10^{3}	4×10^{6}	3×10^{14}	5×10^{7}	2×10^{4}	3×10^{14}	5×10^{14}									
(b)	9×10^{3}	1×10^{4}	1×10^{6}	3×10^{14}	3×10^{12}	3×10^{3}	5×10^{14}	5×10^{14}									
1784 (a)	Shorted	Shorted	4×10^{6}	5×10^{14}	1×10^{8}	Shorted	2×10^{14}	5×10^{14}									
(b)	Shorted	Shorted	4×10^{4}	5×10^{14}	5×10^{12}	Shorted	5×10^{14}	3×10^{14}									
1952 (a)	...	...	7×10^{5}	3×10^{14}	1×10^{8}	...	3×10^{7}	5×10^{14}									
(b)	...	...	4×10^{5}	5×10^{14}	3×10^{13}	...	2×10^{14}	3×10^{14}									
2120 (a)	...	...	Shorted	3×10^{10}	3×10^{10}	...	7×10^{12}	3×10^{13}									
(b)	...	...	Shorted	1×10^{13}	3×10^{10}	...	1×10^{13}	3×10^{13}									
2456 (a)	...	...	...	Shorted	Shorted	...	Shorted	8×10^{13}									
(b)	...	...	...	1×10^{14}	Shorted	...	8×10^{13}	3×10^{13}									

*Insulation resistance measurements were made at ambient temperature.

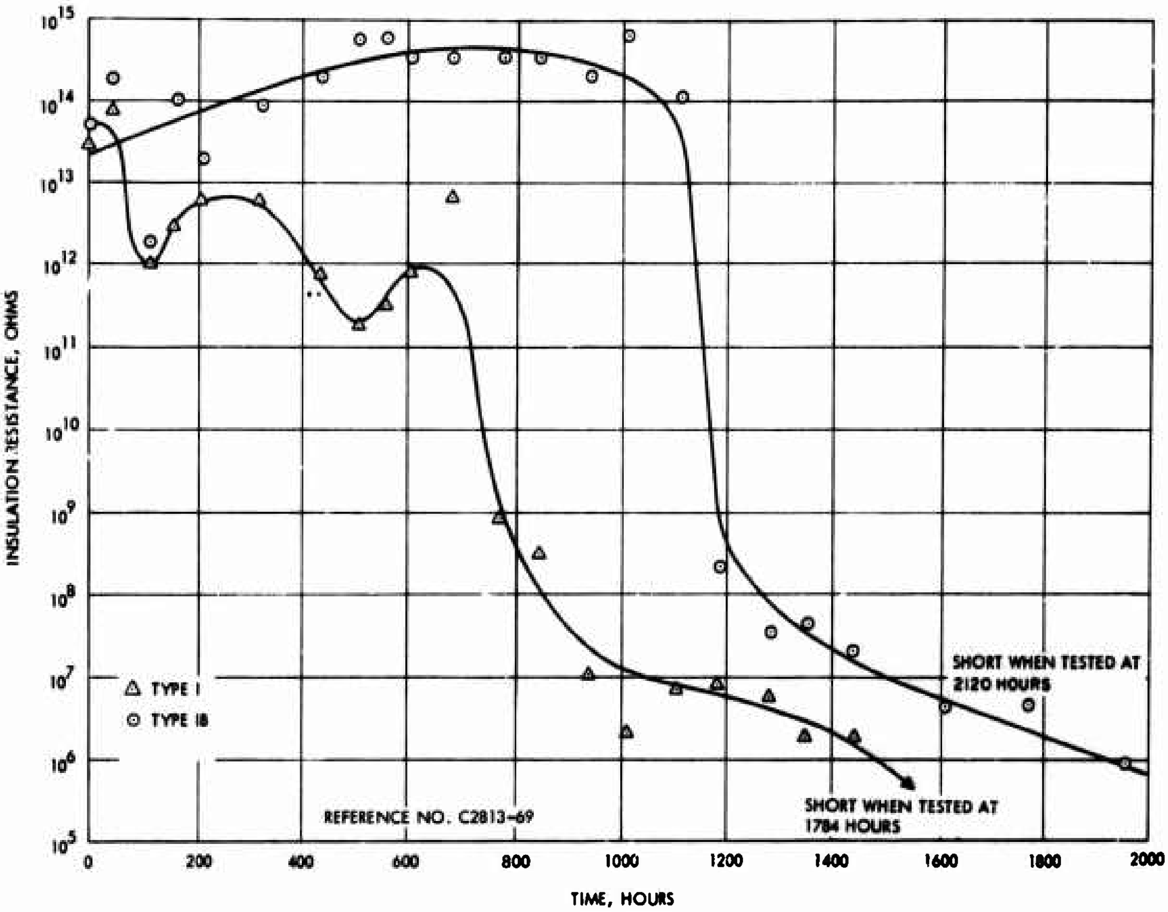


Figure 33. Insulation resistance as a function of thermal aging in air at 550°F — Wires type I and IB

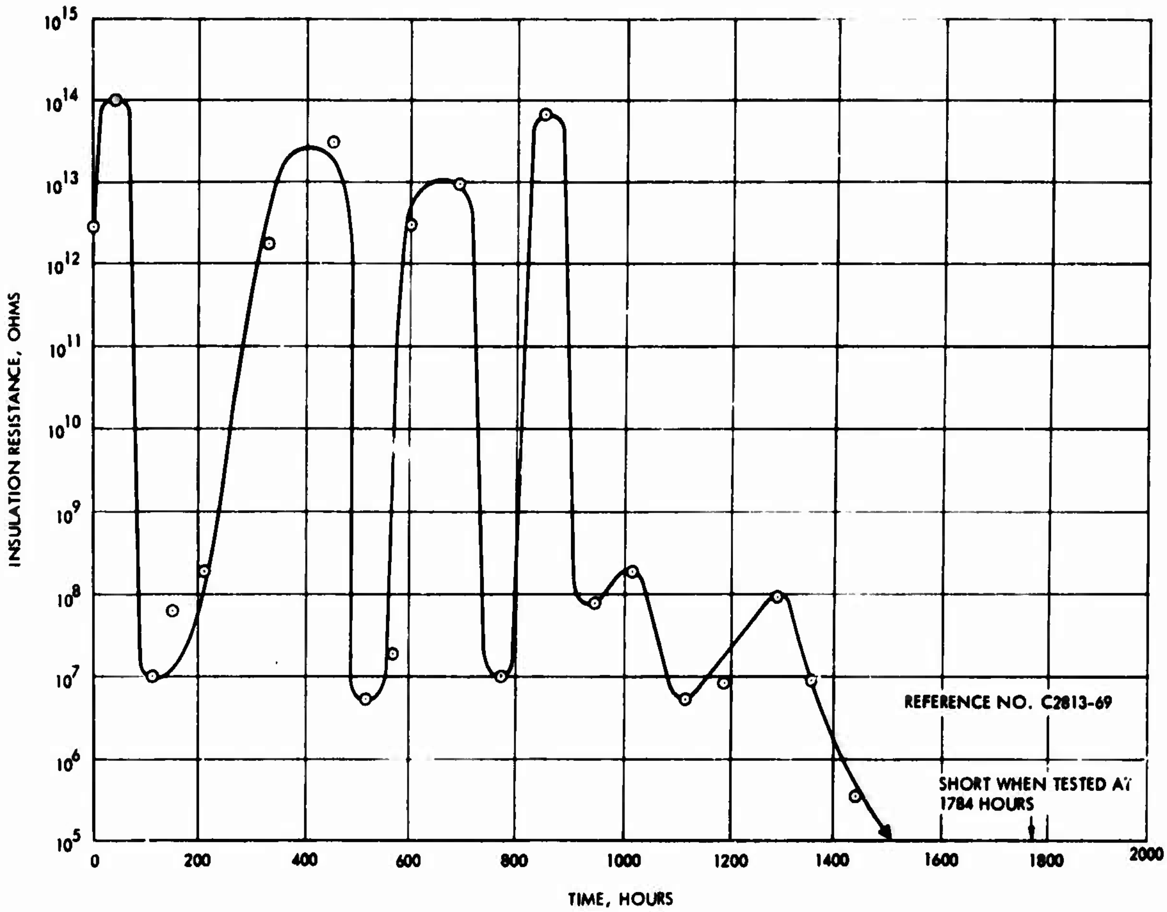


Figure 34. Insulation resistance as a function of thermal aging in air at 550°F — Wire type IA

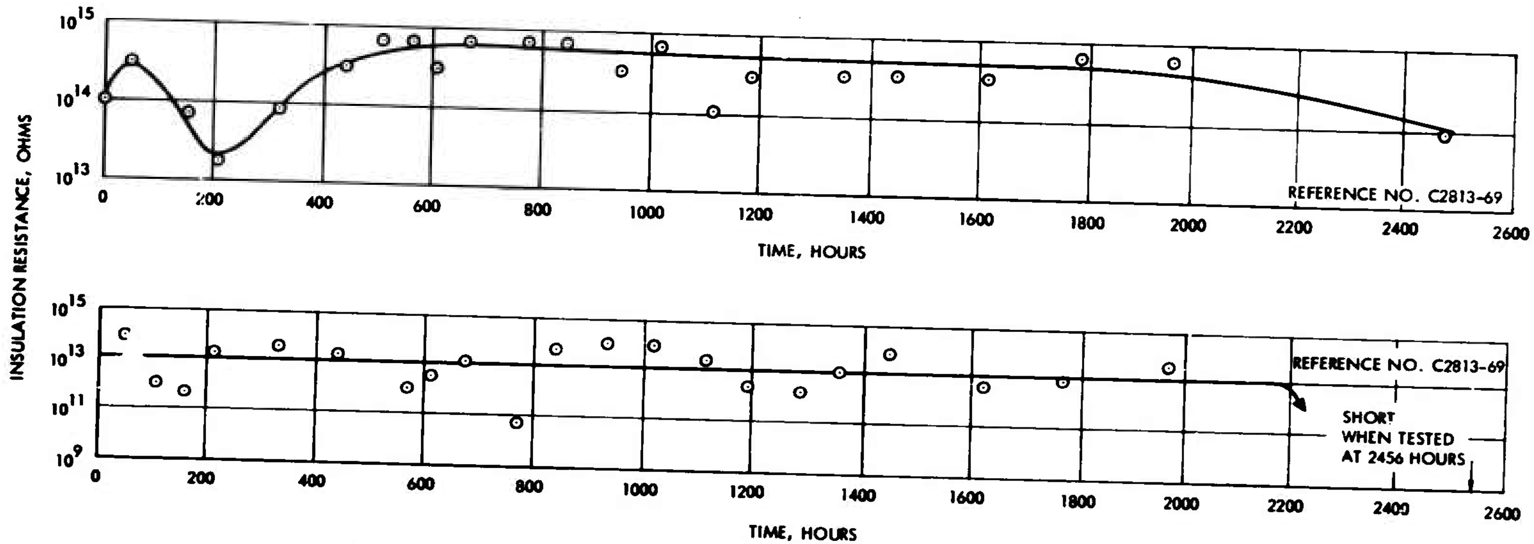


Figure 35. Insulation resistance as a function of thermal aging in air at 550°F – Wires type IC (top) and II (bottom)

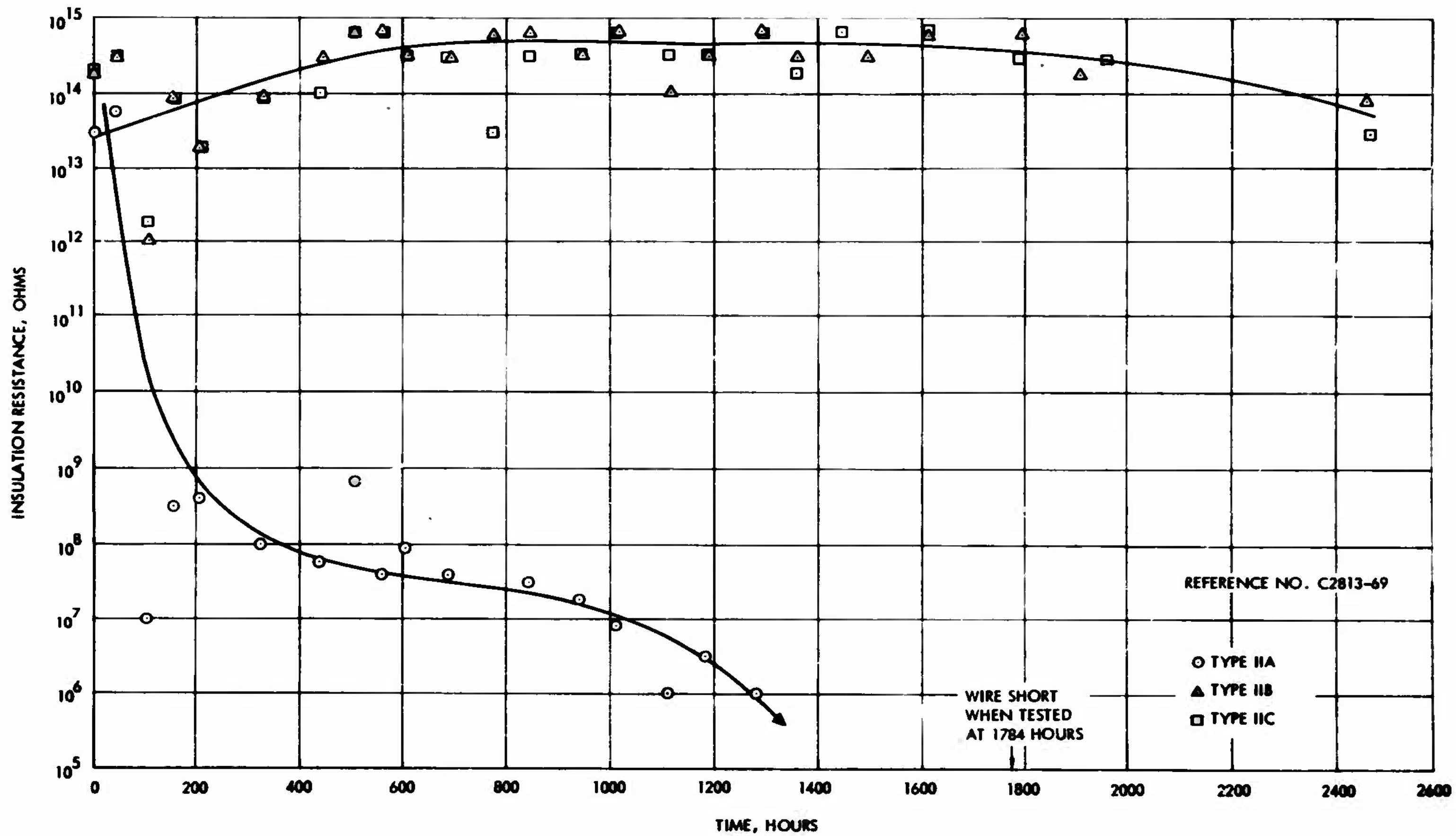


Figure 36. Insulation resistance as a function of thermal aging in air at 550°F – Wires type IIA, IIB and IIC

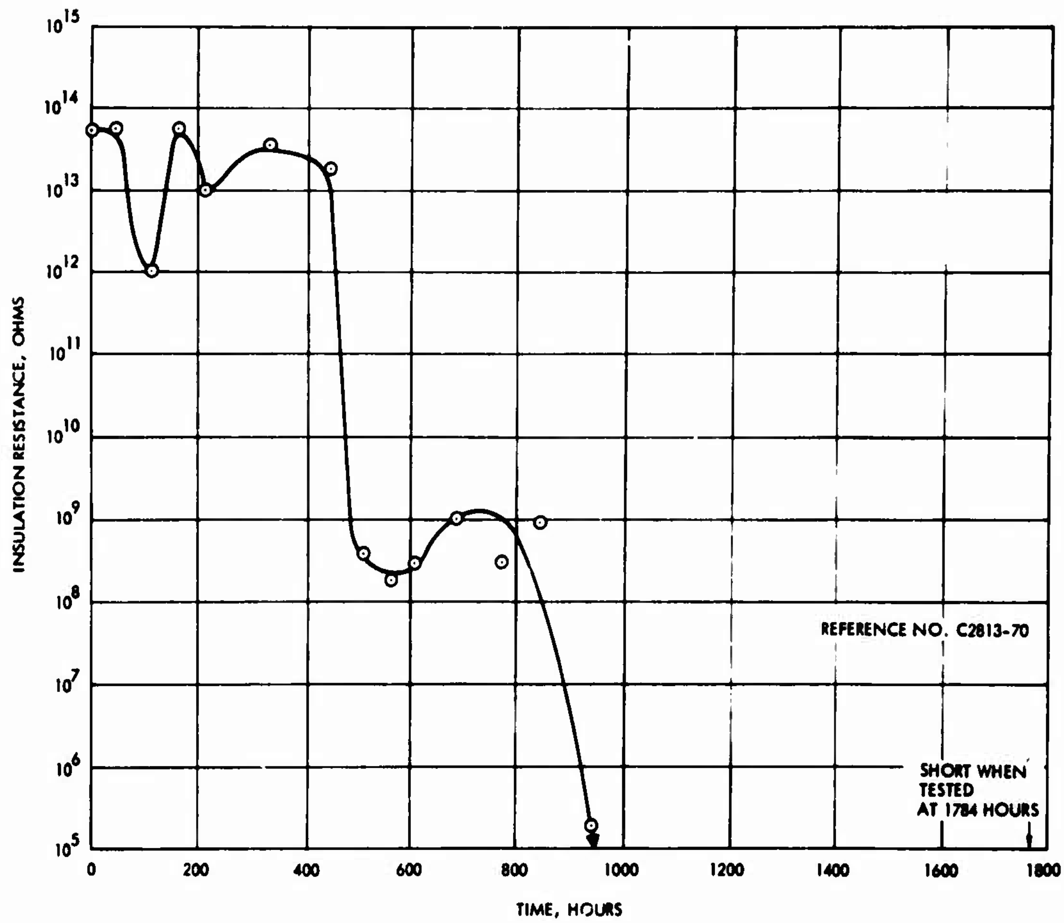


Figure 37. Insulation resistance as a function of thermal aging in air at 550°F – Wire type IV

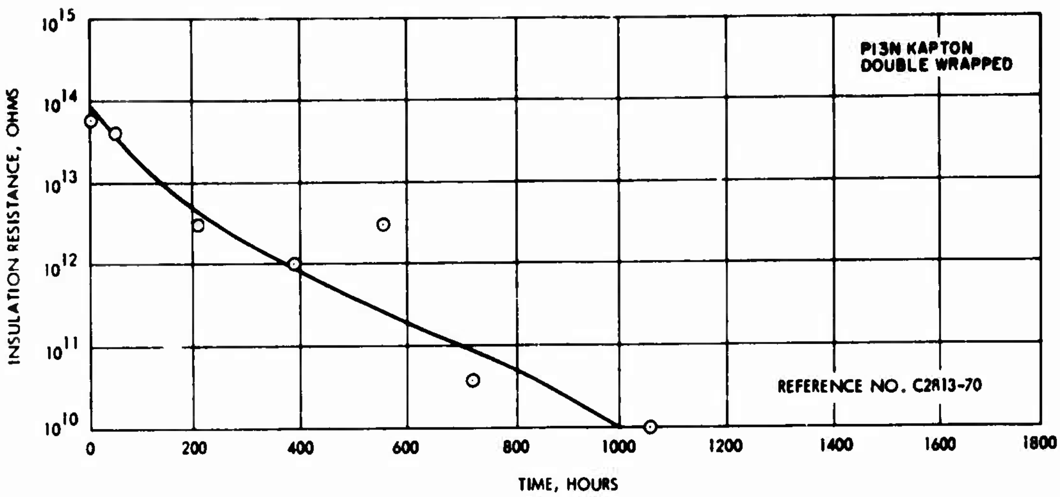


Figure 38. Insulation resistance as a function of thermal aging in air at 550°F – Wire type V

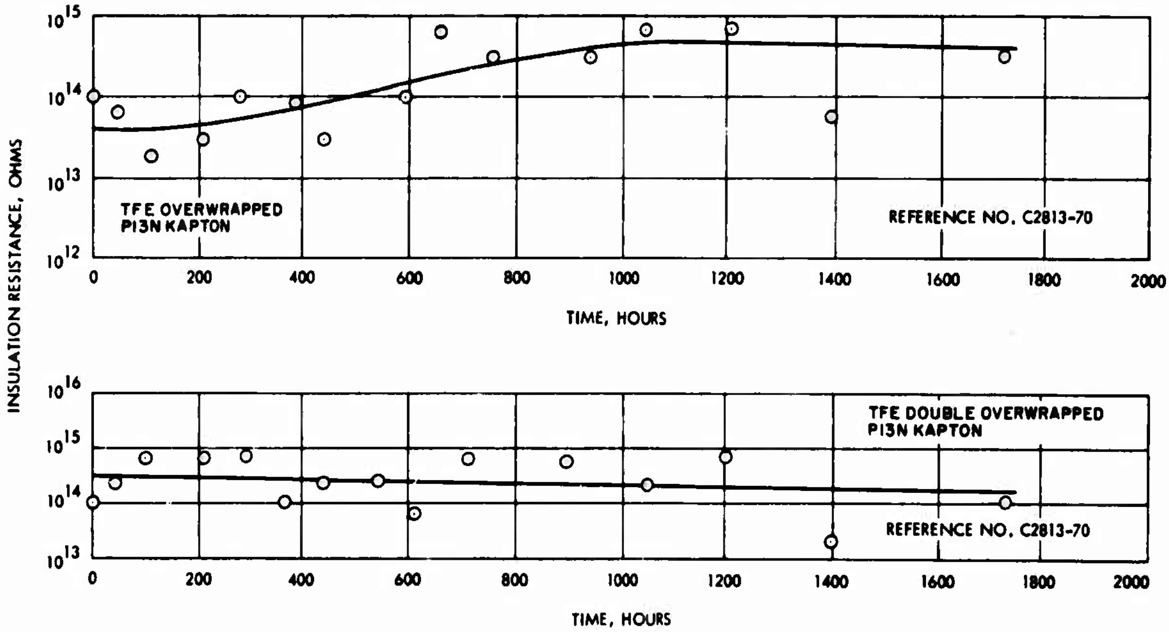


Figure 39. Insulation resistance as a function of thermal aging in air at 550°F - Wire type IVA (top) and IVB (bottom)

It is also to be noted that the double wrapped "pyrrone" (HR-100)-Kapton wire (II) was superior to the double wrapped P13N-Kapton wire V at this temperature.

Also of importance are the results of tests on a MIL-W-81381 wire from Milo Carolina Corp. This wire has a Teflon FEP-Kapton wrapped insulation and is available in both a double and single wrap. A graph of data on the single wrapped wire is shown in Figure 40. Not evidenced in this graph, however, is the observation that the wire was peeling at both ends for a distance of 1 to 2 inches and that the insulation resistance measurements were obtained by keeping the unraveled ends out of the water. Furthermore, at the aging temperature the exposed FEP fuses together and wires left in contact cannot separate easily.

Data used in plotting the graphs described above are tabulated in Table XXIII. It is to be noted, however, that although duplicate wires were run, the graphs were plotted from the best of the two because this provided a better indication of the wire potential, especially considering the fact that these wires are all experimental and optimum production parameters had not been established. It should be noted, however, that for wires I, IB, IC, IIC, IV, V and VI there was no significant difference between the two wires of the pairs.

c. Air Aging at 316°C (600°F)

Tests conducted at 316°C (600°F) were terminated after 1019 hours. Even after this elapsed time several wires had not failed although their insulation resistance had dropped. Test results are tabulated in Table XXIV and graphs of the data are illustrated in Figures 41 through 45.

Figures are drawn using data on the best of the duplicate wires for the same reasons mentioned above. Furthermore, for the "pyrrone" type polymers HR-100 and HR-300, scale-up synthesis parameters have

Table XXIV. Insulation Resistance as a Function of Thermal Aging in Air at 600°F*

Time Hours	I Ohms	IB Ohms	IC Ohms	II Ohms	IIA Ohms	IIB Ohms	IIC Ohms	IV Ohms	IVA Ohms	IVB Ohms	V Ohms	VI Ohms	VIA Ohms
0 (a)	3×10^{13}	5×10^{14}	3×10^{14}	2×10^{13}	(c) 2×10^{13}	3×10^{14}	5×10^{14}	2×10^{14}	7×10^{13}	1×10^{13}	6×10^{13}	7×10^{10}	6×10^{13}
(b)	4×10^{13}	1×10^{14}	5×10^{14}	4×10^{13}	(d) 1×10^{13}	5×10^{14}	5×10^{14}	8×10^{13}	6×10^{13}	3×10^{13}	6×10^{13}	2×10^{10}	7×10^{13}
48 (a)	8×10^{13}	2×10^{14}	3×10^{14}	3×10^{13}	(c) 8×10^{13}	3×10^{14}	2×10^{14}	1×10^{14}	5×10^{13}	6×10^{13}	5×10^{13}	1×10^{14}	1×10^{14}
(b)	5×10^{13}	2×10^{14}	3×10^{14}	5×10^{13}	(d) 7×10^{13}	8×10^{14}	6×10^{13}	1×10^{14}	5×10^{13}	6×10^{13}	4×10^{13}	6×10^{13}	2×10^{14}
14[illegible] (a)	9×10^{11}	7×10^{8}	3×10^{14}	1×10^{13}	(c) 1×10^{14}	1×10^{14}	1×10^{14}	6×10^{13}	6×10^{13}	7×10^{13}	1×10^{14}	Delamination 4-6"	Delamination 1-2"
(b)	1×10^{13}	1×10^{14}	2×10^{14}	8×10^{12}	(d) 4×10^{13}	3×10^{14}	1×10^{14}	8×10^{13}	5×10^{13}	3×10^{13}	8×10^{13}		
216 (a)	1×10^{9}	7×10^{8}	3×10^{14}	1×10^{12}	(c) 5×10^{13}	3×10^{14}	2×10^{14}	1×10^{12}	4×10^{13}	6×10^{13}	1×10^{13}		
(b)	1×10^{11}	1×10^{14}	3×10^{14}	5×10^{13}	(d) 3×10^{12}	3×10^{14}	8×10^{13}	1×10^{13}	1×10^{14}	1×10^{14}	3×10^{12}		
288 (a)	4×10^{8}	5×10^{8}	5×10^{14}	3×10^{11}	(c) 5×10^{8}	3×10^{14}	3×10^{12}	5×10^{12}	4×10^{13}	5×10^{12}	3×10^{12}		
(b)	3×10^{8}	1×10^{9}	3×10^{14}	3×10^{12}	(d) 3×10^{11}	3×10^{14}	8×10^{11}	5×10^{12}	3×10^{13}	3×10^{13}	1×10^{12}		
336 (a)	5×10^{8}	8×10^{13}	1×10^{14}	5×10^{11}	(c) 1×10^{9}	5×10^{14}	2×10^{14}	1×10^{10}	3×10^{12}	3×10^{13}	5×10^{12}		
(b)	3×10^{8}	4×10^{8}	1×10^{14}	3×10^{12}	(d) 7×10^{12}	5×10^{14}	3×10^{10}	5×10^{11}	3×10^{13}	3×10^{13}	5×10^{12}		
384 (a)	1×10^{9}	8×10^{13}	2×10^{9}	3×10^{12}	(c) 6×10^{8}	1×10^{14}	3×10^{12}	5×10^{12}	1×10^{8}	5×10^{10}	5×10^{12}		
(b)	1×10^{8}	6×10^{13}	3×10^{14}	3×10^{12}	(d) 6×10^{10}	2×10^{14}	2×10^{12}	5×10^{12}	5×10^{7}	6×10^{13}	2×10^{12}		
4[illegible]6 (a)	1×10^{8}	3×10^{13}	5×10^{8}	1×10^{12}	(c) 5×10^{9}	7×10^{8}	1×10^{13}	2×10^{12}	5×10^{7}	5×10^{7}	3×10^{13}		
(b)	4×10^{7}	2×10^{12}	3×10^{9}	5×10^{12}	(d) 3×10^{12}	3×10^{12}	2×10^{12}	1×10^{11}	4×10^{7}	3×10^{5}	1×10^{13}		
504 (a)	5×10^{7}	4×10^{8}	3×10^{14}	1×10^{12}	(c) 8×10^{7}	3×10^{14}	2×10^{13}	4×10^{8}	1×10^{7}	2×10^{6}	3×10^{11}		
(b)	3×10^{7}	2×10^{8}	3×10^{14}	3×10^{12}	(d) 3×10^{8}	1×10^{14}	3×10^{10}	3×10^{8}	4×10^{7}	9×10^{5}	5×10^{11}		
552 (a)	3×10^{7}	1×10^{8}	5×10^{8}	5×10^{10}	(c) Shorted	5×10^{12}	5×10^{11}	5×10^{10}	Shorted	Shorted	3×10^{12}		
(b)	1×10^{7}	5×10^{6}	5×10^{12}	5×10^{11}	(d) Shorted	1×10^{13}	5×10^{11}	5×10^{10}	Shorted	Shorted	2×10^{11}		
624 (a)	1×10^{10}	9×10^{9}	8×10^{9}	9×10^{9}		2×10^{13}	5×10^{12}	8×10^{9}			5×10^{10}		
(b)	1×10^{10}	1×10^{10}	5×10^{12}	7×10^{11}		1×10^{11}	2×10^{12}	5×10^{9}			1×10^{10}		
672 (a)	1×10^{10}	3×10^{6}	6×10^{12}	5×10^{11}		3×10^{13}	4×10^{12}	2×10^{10}			3×10^{5}		
(b)	1×10^{10}	2×10^{6}	3×10^{12}	2×10^{11}		5×10^{12}	1×10^{12}	1×10^{10}			1×10^{11}		
720 (a)	2×10^{7}	3×10^{6}	4×10^{10}	5×10^{11}		3×10^{13}	5×10^{12}	Shorted			5×10^{6}		
(b)	3×10^{7}	2×10^{6}	3×10^{7}	4×10^{11}		5×10^{12}	5×10^{11}	Shorted			1×10^{12}		
789 (a)	Shorted	Shorted	1×10^{9}	3×10^{11}		3×10^{13}	3×10^{12}				3×10^{6}		
(b)	Shorted	Shorted	2×10^{7}	3×10^{11}		3×10^{7}	7×10^{6}				2×10^{11}		
809 (a)			2×10^{9}	6×10^{11}		3×10^{13}	5×10^{12}				4×10^{6}		
(b)			7×10^{7}	5×10^{11}		3×10^{7}	2×10^{7}				5×10^{10}		
854 (a)			5×10^{9}	1×10^{11}		3×10^{13}	1×10^{12}				3×10^{6}		
(b)			2×10^{8}	3×10^{11}		3×10^{7}	2×10^{6}				5×10^{10}		
923 (a)			4×10^{6}	8×10^{6}		1×10^{13}	2×10^{11}				Shorted		
(b)			3×10^{6}	8×10^{10}		2×10^{6}	9×10^{6}				3×10^{10}		
971 (a)			5×10^{6}	9×10^{6}		3×10^{13}	3×10^{11}						
(b)			3×10^{6}	Shorted		1×10^{6}	9×10^{6}				1×10^{12}		
1019 (a)			Shorted	1×10^{7}		1×10^{14}	7×10^{6}						
(b)			3×10^{6}	---		Shorted	2×10^{6}				3×10^{11}		

*Insulation resistance measurements were made at ambient temperature.

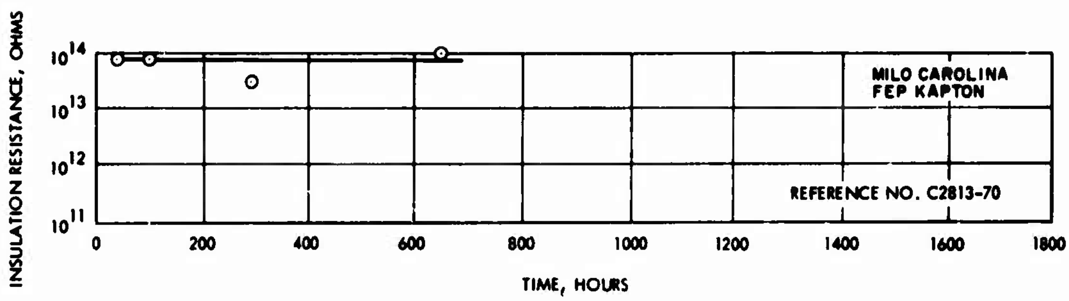


Figure 40. Insulation resistance as a function of thermal aging in air at 550°F — Wire type VI

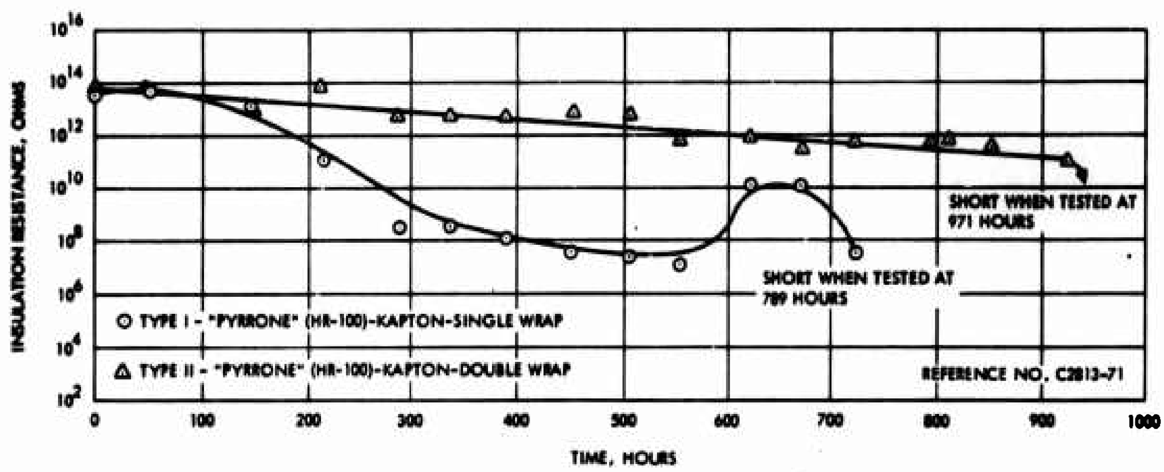


Figure 41. Insulation resistance as a function of thermal aging in air at 600°F — Wires type I and II

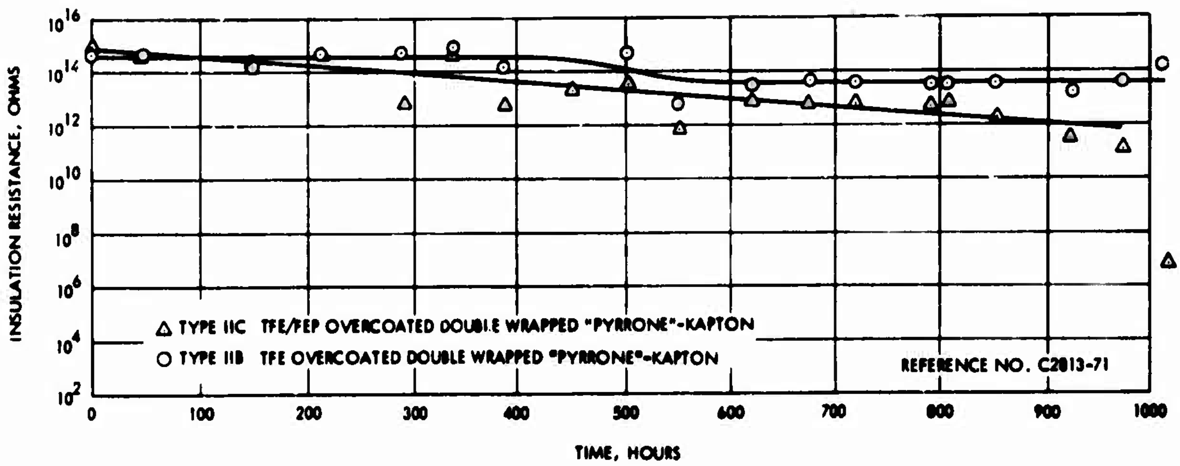


Figure 42. Insulation resistance as a function of thermal aging in air at 600°F — Wires type IIB and IIC

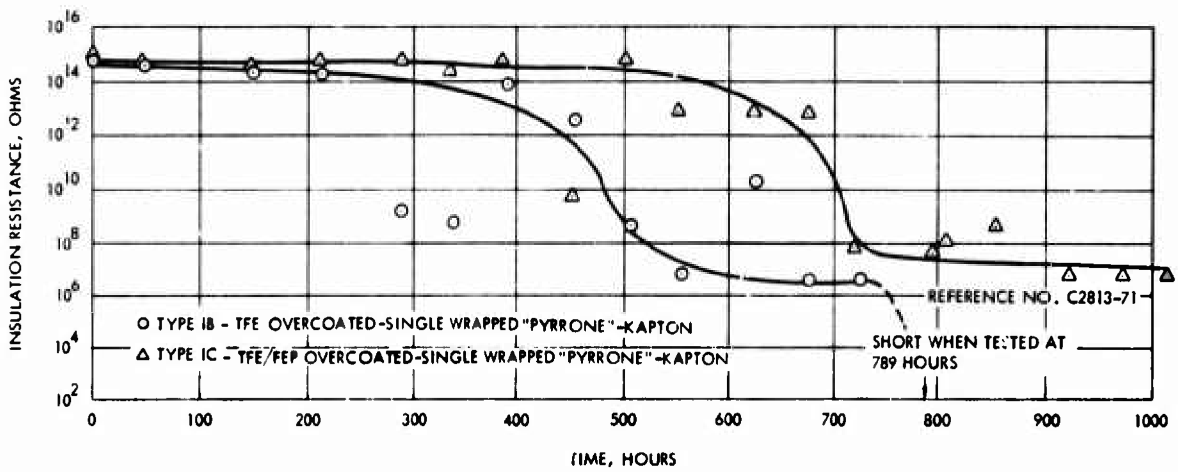


Figure 43. Insulation resistance as a function of thermal aging in air at 600°F — Wires type IB and IC

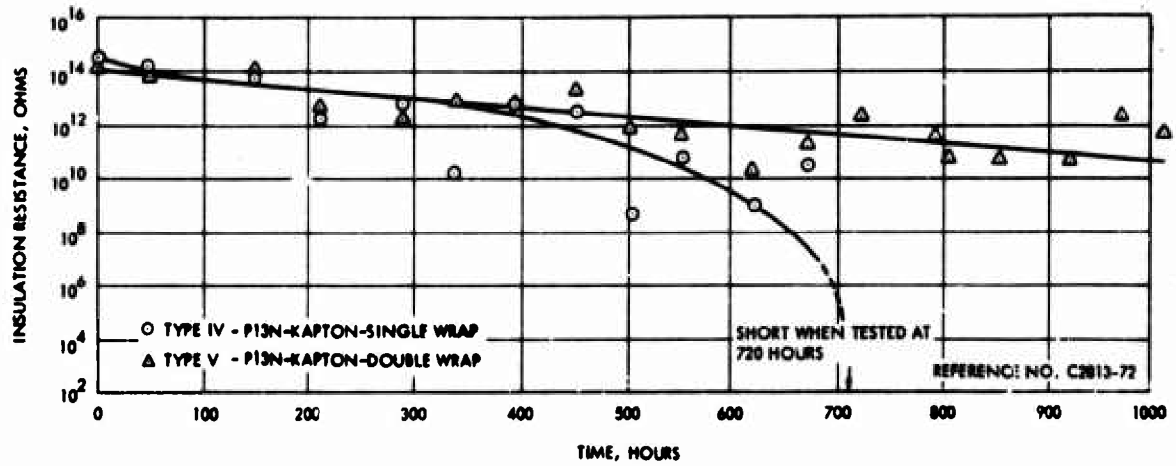


Figure 44. Insulation resistance as a function of thermal aging in air at 600°F – Wire types IV and V

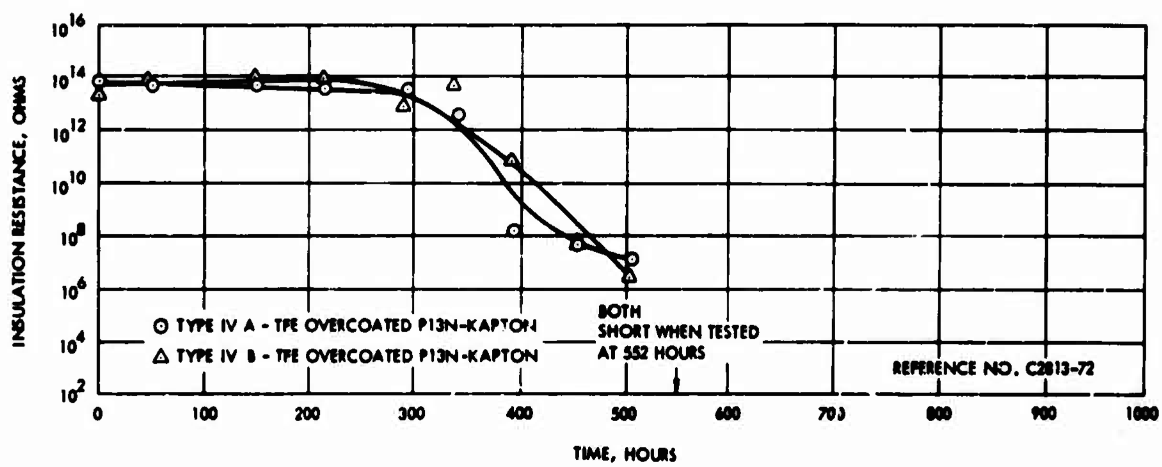


Figure 45. Insulation resistance as a function of thermal aging in air at 600°F – Wires type IVA and IVB

also not been established and resins synthesized on the bench scale tend to vary somewhat from batch to batch. Nevertheless, the graphs as plotted demonstrate the potential of the various wires.

There is little doubt that the double wrapped HR-100-Kapton wire (II) was superior to its single wrapped counterpart (I) (Figure 41). Not only did it have a higher insulation resistance throughout the test, but also it shorted much later, 971 hours versus 789 hours. Furthermore, even after 854 hours the insulation resistance of the double wrapped wire (II) had fallen only 2 orders of magnitude ($10^{13} \rightarrow 10^{11}$ ohms) whereas the single wrapped wire (I) lasted only 216 hours before a comparable drop was observed.

Teflon type overcoats, on either single wrapped or double wrapped wires, improved performance presumably because the overcoats retarded oxidation. However, the overcoats themselves failed due to shrinkage prior to the failure of the substrates. Overcoated wires IIB and IIC (Figure 42) as would be expected, outperformed the overcoated wires IB and IC (Figure 43). Both wires IIB and IIC passed 1000 hours without failure and without gross degradation, whereas IB shorted near 789 hours and exhibited significant degradation near 300 hours. In contrast, wire IC showed signs of gross degradation near 700 hours.

With the P13N-Kapton type wires the double wrap again proved to be best (Figure 44). In this case the double wrap survived 1000 hours without gross degradation, whereas the single wrap was shorted when tested at 720 hours. Overwrapping the single wrapped P13N-Kapton wire (IV) with Teflon TFE yielded the wires IVA and IVB but in this case the overwrap not only failed to improve the wires, but also appeared to make them fail sooner, both at 552 hours (Figure 45).

Wires VI and VIA are the commercial FEP-Kapton insulated wires from Milo Carolina Corp. The former is a single wrapped wire and the latter is double wrapped. Tests with these were terminated after only 48 hours because the wire ends had delaminated, the former about 4 to 6 inches and the latter about 1 to 2 inches. The wires also had become fused together.

d. Air Aging at 371°C (700°F)

Hyperthermal aging studies in air at 371°C (700°F) were also conducted on the type II, VII, VIII, and IX wires. More specifically these are the HR-100-Kapton double wrap, HR-100-Kapton triple wrap, HR-300-Kapton double wrap, and the HR-300-Kapton triple wrap, respectively. Results of these tests are shown in Table XXV.

Graphs of these results are shown in Figure 46. It is evident from these data that the double wrapped wire II survived 80 hours whereas VIII survived for about 90 to 9 hours. In contrast the triple wrapped wires VII and IX were both still good after 150 hours. Tests were terminated at this time.

e. Non-Cyclic Thermal Soaks

Another type of thermal soak test was conducted on "pyrrone"-Kapton, (Type I) and on Teflon TFE overcoated "pyrrone"-Kapton (Type IA) insulated wires

In these tests the wires were wrapped in aluminum foil, then placed in an oven, and insulation resistance measurements were made periodically at the soak temperature. Soak temperatures were 600°F for 120 hours and 700°F for 48 hours. Results of these tests are shown in Table XXVI and were obtained at the soak temperatures.

At 316°C (600°F) and 371°C (700°F) the "pyrrone" -Kapton and both Teflon overcoated "pyrrone" -Kapton insulated wires held up remarkably well. The Teflon overcoated wires performed better than the "pyrrone" (HR 100)-Kapton alone. Test results generally showed a ±1 order of magnitude variation if the single low value is discounted; thus, over the 120-hour period at 316°C (600°F) the insulation resistance of the "pyrrone" -Kapton insulated wire (I) remained in the 10^8 to 10^9 ohm range throughout the test, the single wrapped Teflon on "pyrrone" - Kapton (IA) remained in the 10^{11} ohm region, and the double wrapped Teflon on "pyrrone" -Kapton (IB) remained in the 10^{12} ohm range. A comparable order of stability was observed over the 48 hour period at 371°C (700°F). Wires became blackened and embrittled as a result of the 371°C (700°F) exposures.

Table XXV. Insulation Resistance as a Function of Thermal Aging in Air at 700°F*

Time Hours		II Ohms	VII Ohms	VIII Ohms	IX Ohms
0	(a)	3×10^{12}	5×10^{12}	3×10^{12}	5×10^{12}
	(b)	3×10^{12}	5×10^{12}	3×10^{12}	5×10^{12}
16	(a)	3×10^{12}	5×10^{12}	3×10^{12}	5×10^{12}
	(b)	3×10^{12}	5×10^{12}	3×10^{12}	5×10^{12}
40	(a)	5×10^{11}	3×10^{11}	5×10^{11}	3×10^{11}
	(b)	3×10^{11}	2×10^{11}	5×10^{11}	5×10^{11}
56	(a)	2×10^{11}	3×10^{11}	1×10^{10}	1×10^{12}
	(b)	5×10^{11}	5×10^{11}	5×10^{11}	1×10^{12}
62	(a)	3×10^{11}	5×10^{10}	1×10^{11}	1×10^{10}
	(b)	1×10^{11}	3×10^{10}	2×10^{10}	2×10^{10}
70	(a)	1×10^{9}	2×10^{10}	2×10^{9}	5×10^{10}
	(b)	5×10^{9}	5×10^{10}	1×10^{9}	5×10^{10}
77	(a)	2×10^{8}	5×10^{9}	5×10^{10}	1×10^{11}
	(b)	5×10^{9}	6×10^{8}	5×10^{10}	2×10^{11}
86	(a)	2×10^{10}	5×10^{9}	1×10^{10}	8×10^{10}
	(b)	8×10^{8}	5×10^{10}	5×10^{9}	1×10^{10}
93	(a)	Shorted	6×10^{6}	3×10^{10}	1×10^{11}
	(b)	5×10^{6}	5×10^{10}	2×10^{10}	1×10^{11}
101	(a)	–	5×10^{10}	5×10^{8}	5×10^{9}
	(b)	Shorted	8×10^{6}	1×10^{10}	5×10^{10}
117	(a)	–	1×10^{10}	Shorted	5×10^{10}
	(b)	–	5×10^{10}	Shorted	3×10^{10}
125	(a)	–	5×10^{11}	–	1×10^{12}
	(b)	–	5×10^{11}	–	1×10^{11}
141	(a)	–	3×10^{11}	–	5×10^{11}
	(b)	–	3×10^{11}	–	1×10^{11}
150	(a)	–	1×10^{11}	–	3×10^{10}
	(b)	–	5×10^{9}	–	3×10^{6}

*Insulation resistance measurements were made at ambient temperature.

Table XXVI. Insulation Resistance of "Pyrrone"-Kapton Insulated Wire as a Function of Thermal Aging – Type IA and IB Wires – Non-cyclic Test

Test Conditions			Insulation Resistance, Ohms		
Temperature,*		Time, hours	Type I	Type IA	Type IB
°F	°C				
75	23	0	1×10^{12}	3×10^{11}	8×10^{12}
600	316	4	2×10^{8}	2×10^{11}	2×10^{12}
600		8	3×10^{8}	5×10^{11}	4×10^{12}
600		24	2×10^{8}	2×10^{12}	2×10^{13}
600		48	3×10^{9}	2×10^{10}	1×10^{10}
600		76	2×10^{9}	5×10^{9}	1×10^{13}
600		100	1×10^{9}	5×10^{12}	1×10^{13}
600		120	2×10^{9}	5×10^{12}	2×10^{11}
700	371	8	2×10^{7}	3×10^{10}	3×10^{11}
700		24	3×10^{7}	1×10^{10}	4×10^{10}
700		48	2×10^{7}	2×10^{9}	4×10^{11}

*Note: The 371°C (700°F) measurements were made after the 120 hour, 316°C (600°F) test was completed.

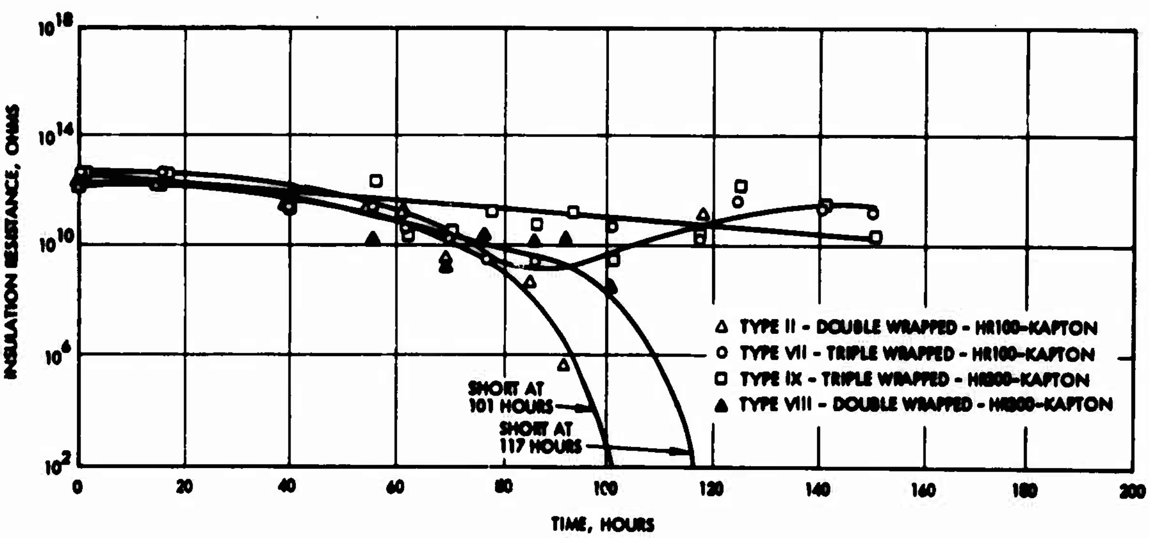


Figure 46. Insulation resistance as a function of thermal aging in air at 700°F – Wire types II, VII, VIII, and IX

2. HUMIDITY TESTS

"Pyrrone" (HR-100)-Kapton insulated wires overcoated with perfluoroalkylenetriazine and Teflon TFE were heated at 85°C (185°) for 28 days at a relative humidity of 95 percent. Insulation resistance measurements then were made with the wires submerged in water as well as out of water. For the elevated temperature tests wires were wrapped in aluminum foil, then gradually heated in a tube furnace with measurements being made after each temperature equilibrium was achieved.

Wires first were tested at ambient temperature, at 93°C (200°F), and at 149°C (300°F). They were then cooled to ambient temperature and retested over the full temperature range to 315°C (600°F). After reaching 315°C they were allowed to remain at this temperature for 1 hour. They were again cooled and retested at various temperatures between ambient and 427°C (800°F). During the first heating cycle (to 315°C, 600°F) water was evolved in progressively smaller amounts as the wires dried, but after the 1 hour heating period, water evolution was no longer evident.

Results of tests on the perfluoroalkylenetriazine overcoated wire are presented in Table XXVII (Figure 47), whereas results obtained on the Teflon TFE overcoated wire are shown in Table XXVIII.

Graphs showing the insulation resistance of the perfluoroalkylenetriazine as a function of temperature as shown in Figures 48, 49 and 50. Figure 48 represents wire in which the perfluoroalkylenetriazine was cured at 50°C (122°F) (4 hours), Figure 49 illustrates a similar wire cured at 100°C (212°F) (4 hours), and Figure 50 illustrates a similar wire cured at 150°C (302°F) (4 hours).

Results of these studies may best be compared by an examination of Figures 51 and 52. Figure 51 compares the three wires after they had been predried to 149°C (300°F). From these comparisons it appears that the longer the perfluoroalkylenetriazine overcoat is cured the better is its resistance to water in the 95 percent relative humidity test [28 days at 85°C (185°F)]. A similar

Table XXVII. Insulation Resistance of Perfluoroalkylenetriazine Overcoated "Pyrrone"-Kapton Wire as a Function of Temperature

Cycle	Temperature		Insulation Resistance,** Ohms		
	°F	°C	50°C Triazine Cure	100°C Triazine Cure	150°C Triazine Cure
I	75*	24*	6×10^6	6×10^5	1×10^8
	75	24	1×10^8	4×10^7	1×10^8
	200	93	3×10^8	1×10^8	5×10^8
	300	148	1×10^9	2×10^9	4×10^9
II	75	24	6×10^7	7×10^7	1×10^8
	200	93	1×10^8	5×10^8	1×10^9
	300	148	4×10^8	8×10^8	2×10^9
	400	204	4×10^8	3×10^9	5×10^9
	500	260	4×10^7	4×10^7	1×10^8
	600	315	6×10^6	8×10^6	1×10^7
	650	343	5×10^6	8×10^6	1×10^7
III	75	24	6×10^{12}	6×10^{12}	6×10^{12}
	200	93	3×10^{12}	3×10^{12}	4×10^{12}
	300	148	7×10^{11}	4×10^{11}	6×10^{11}
	400	204	3×10^{10}	3×10^{10}	4×10^{10}
	500	260	2×10^9	2×10^9	3×10^9
	600	315	4×10^7	6×10^7	8×10^7
	700	371	3×10^6	3×10^6	4×10^6
	800	427	3×10^5	3×10^5	4×10^5

*Readings taken in water bath. **30 inch lengths.

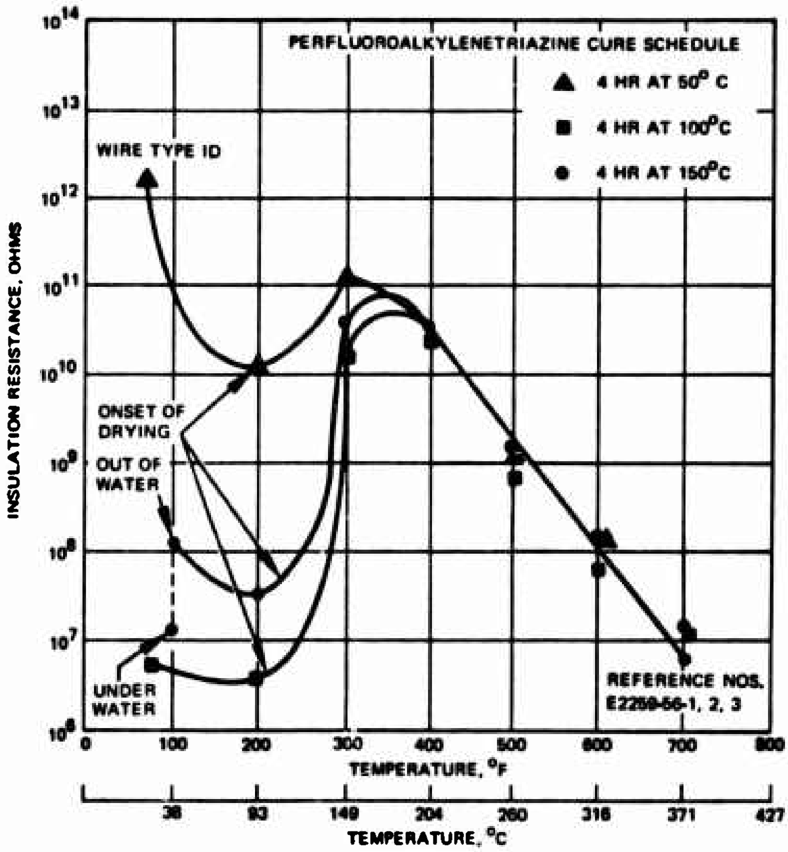


Figure 47. Insulation resistance of perfluoroalkylenetriazine overcoated "pyrrone"-Kapton wire as a function of temperature

Table XXVIII. Insulation Resistance of Teflon TFE Overcoated "Pyrrone"-Kapton Insulated Wire as a Function of Temperature

Cycle	Temperature		Insulation Resistance, Ohms		
	^{o}F	^{o}C	Wire IA (E2259-62D)	Wire IA† (E2259-62E)	Wire IB (E2259-62F)
I	75*	24	3×10^{9}	3×10^{6}	7×10^{12}
	75	24	1×10^{10}	1×10^{8}	1×10^{13}
	200	93	3×10^{8}	6×10^{7}	3×10^{12}
	300	148	2×10^{9}	3×10^{8}	3×10^{12}
II	75	24	3×10^{8}	8×10^{7}	3×10^{12}
	200	93	8×10^{8}	3×10^{8}	7×10^{12}
	300	148	3×10^{9}	3×10^{8}	6×10^{12}
	400	204	3×10^{11}	8×10^{9}	2×10^{12}
	500	260	7×10^{10}	3×10^{9}	3×10^{11}
	600	315	2×10^{11}	8×10^{9}	4×10^{11}
	650**	343	2×10^{11}	2×10^{9}	2×10^{11}
III	75	24	3×10^{13}	2×10^{13}	2×10^{13}
	200	93	2×10^{13}	2×10^{13}	2×10^{13}
	300	148	7×10^{12}	5×10^{12}	9×10^{12}
	400	204	4×10^{12}	1×10^{12}	1×10^{13}
	500	260	3×10^{12}	2×10^{11}	7×10^{12}
	600	315	2×10^{12}	2×10^{10}	2×10^{12}
	700	371	6×10^{11}	2×10^{8}	6×10^{11}
	800	427	5×10^{10}	3×10^{6}	4×10^{10}

*Readings taken with wire under water
**Readings taken before heat soak at 650°F in argon
†The TFE sintering time was slightly longer in this case.

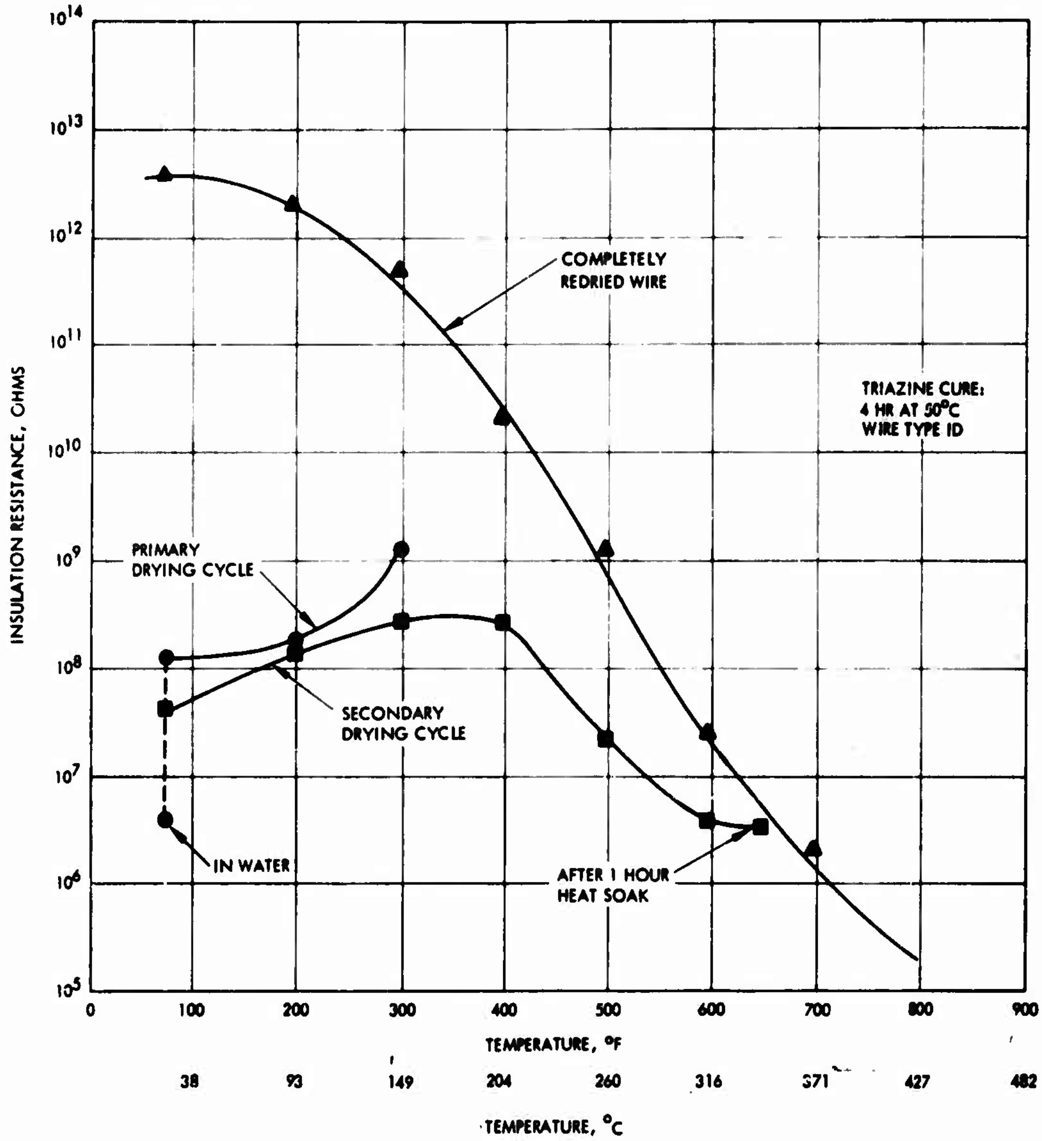


Figure 48. Insulation resistance as a function of temperature—perfluoroalkylenetriazine overcoated "pyrrone"-Kapton

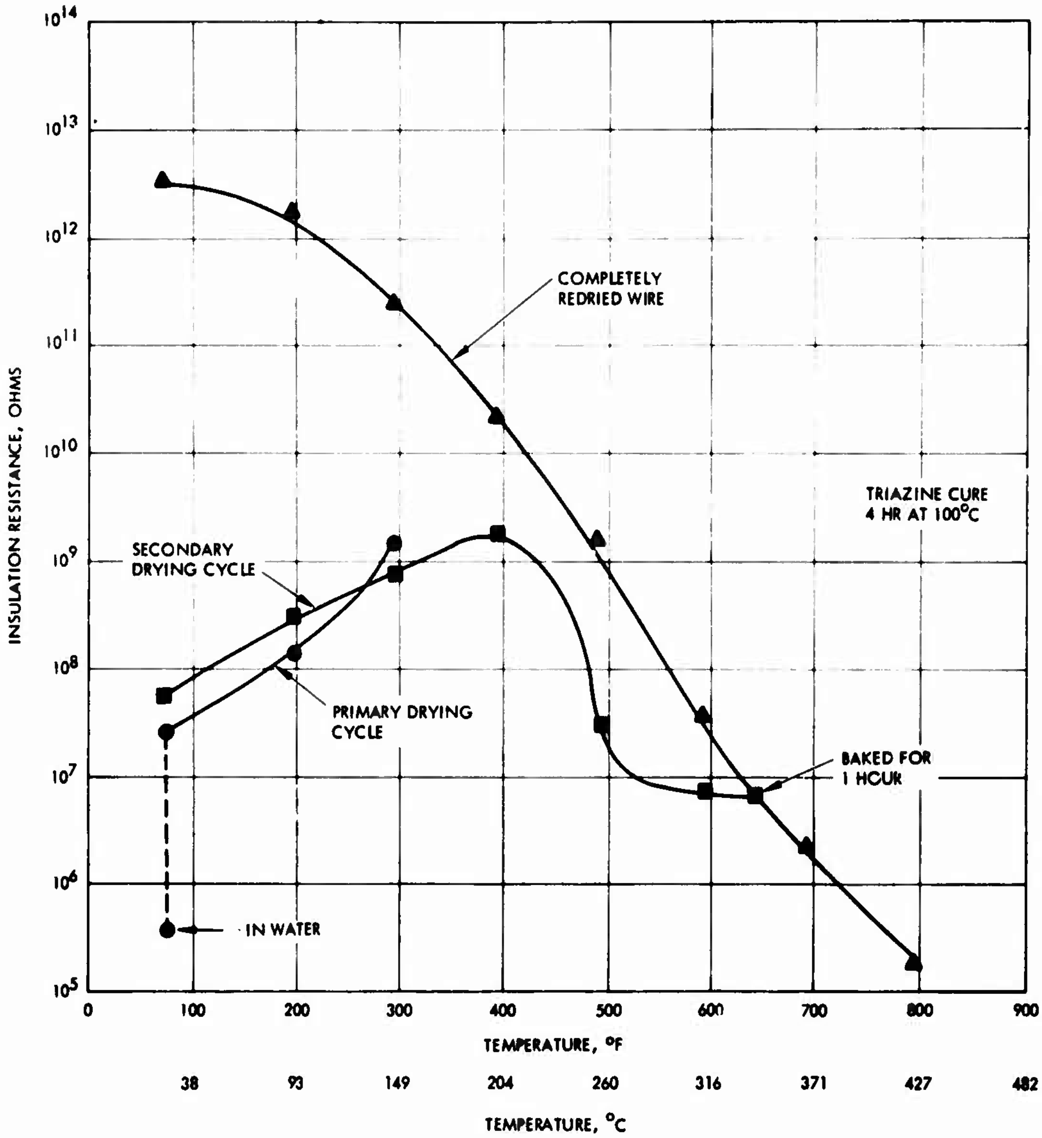


Figure 49. Insulation resistance as a function of temperature—perfluoroalkylenetriazine overcoated "pyrrone"-Kapton

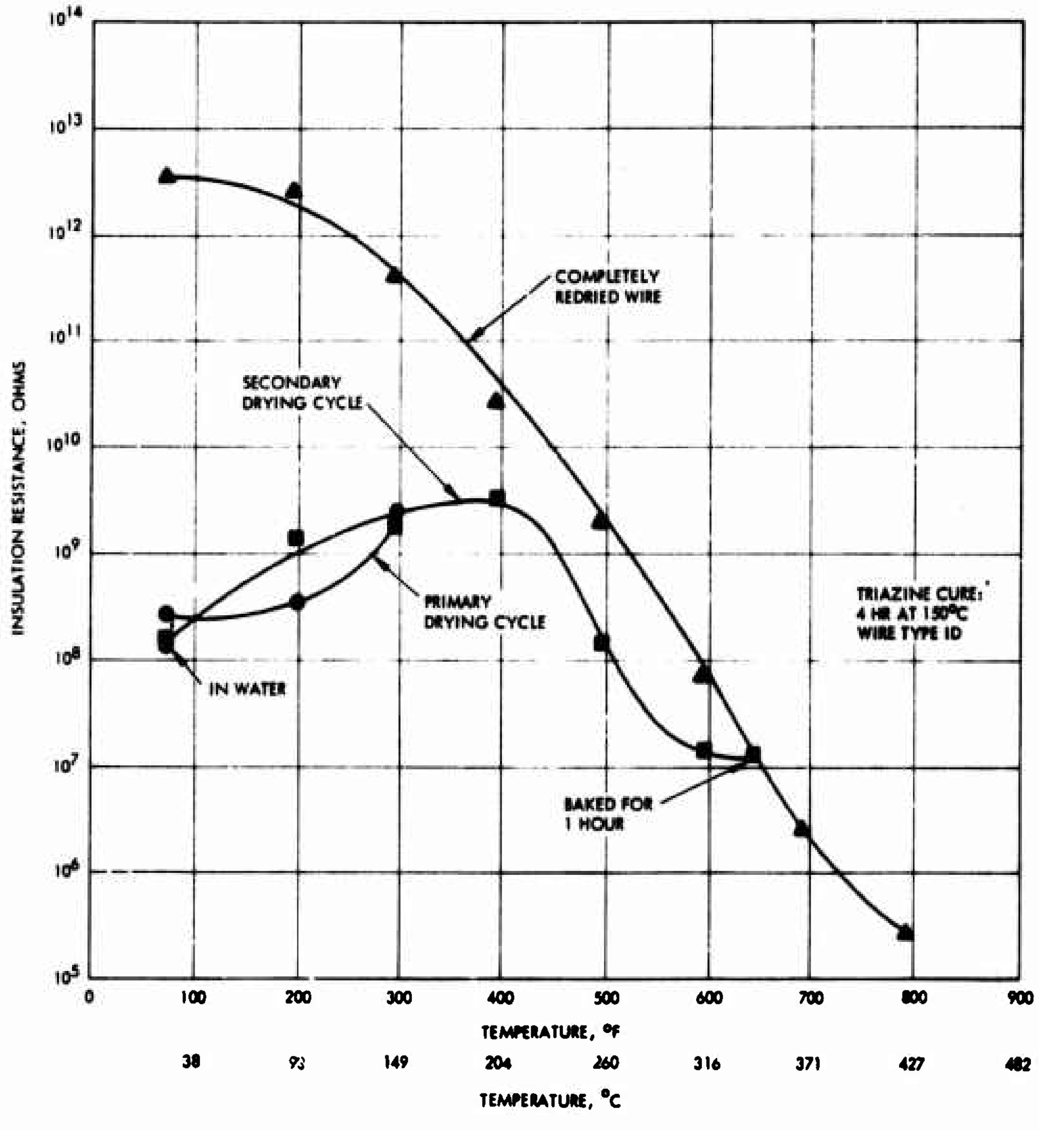


Figure 50. Insulation resistance as a function of temperature — perfluoroalkylenetriazine overcoated "pyrrone"-Kapton

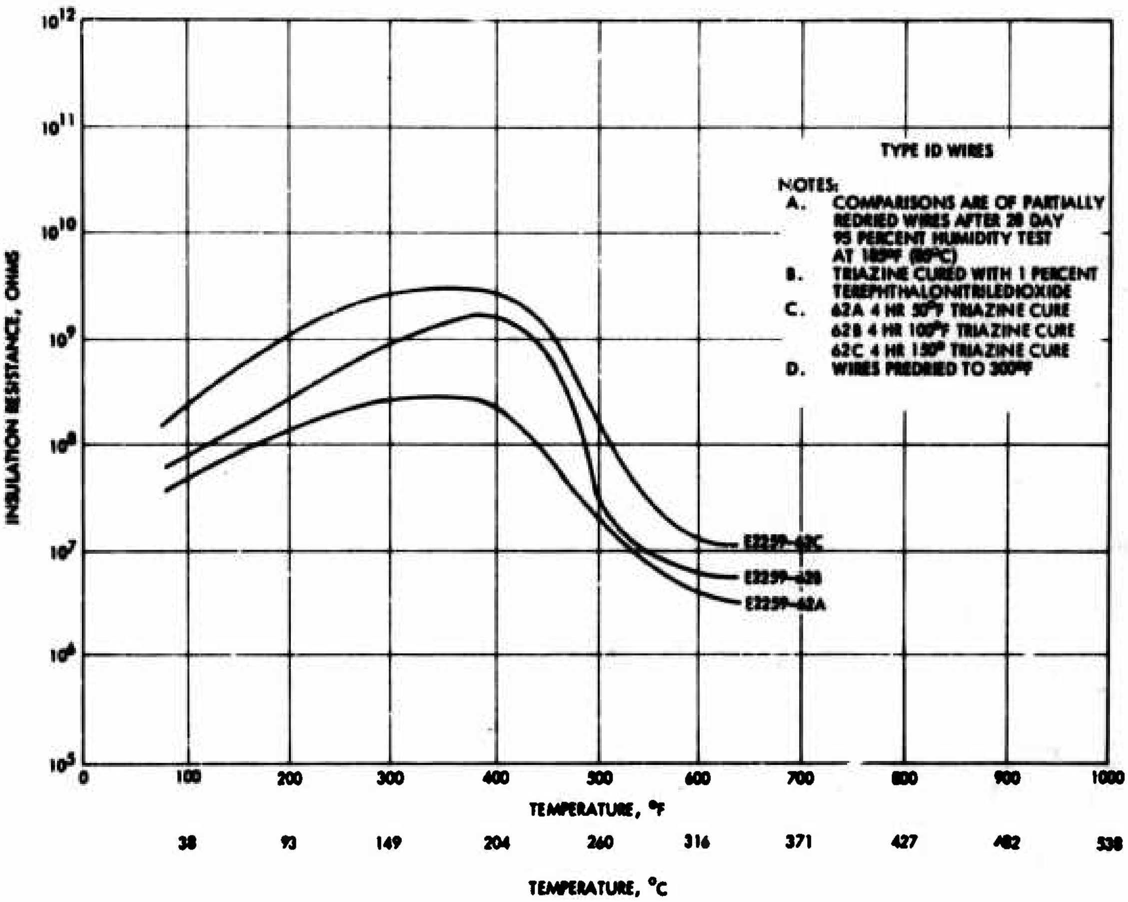


Figure 51. Insulation resistance as a function of temperature—perfluoroalkylenetriazine overcoated "pyrrone"-Kapton

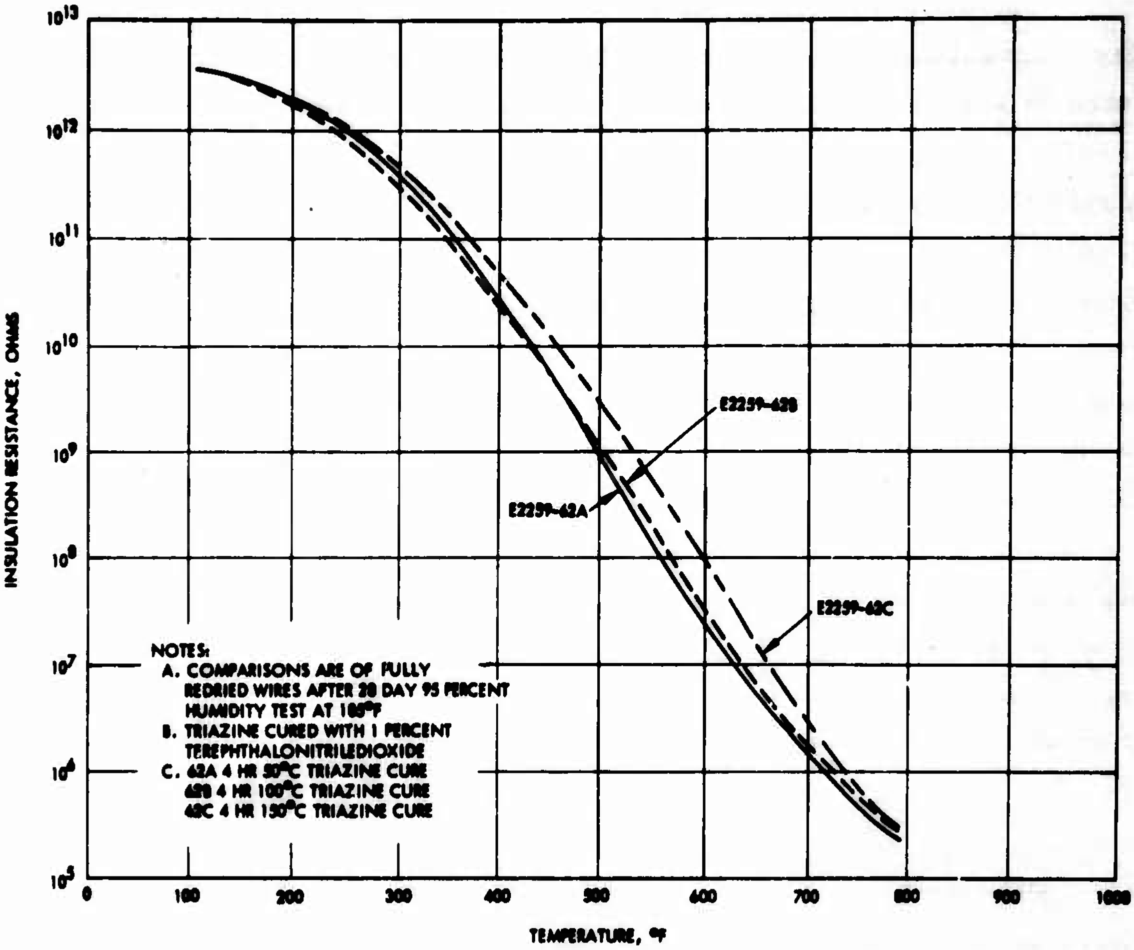


Figure 52. Insulation resistance as a function of temperature - perfluoroalkylenetriazine overcoated "pyrrone"-Kapton

conclusion is reached when comparing the wires after they have been fully dried [to 343°C (650°F)] ; however in this case the relative advantage of one over the other was relatively minor and virtually insignificant. This probably indicates that very little perfluoroalkylenetriazine remained on the wires after the 343°C (650°F) drying cycle. Decomposition of the perfluoroalkylenetriazine above 316°C (600°F) is certainly evidenced.

Insulation resistance of the Teflon TFE overwrapped "pyrrone"-Kapton insulated wire is graphically illustrated as a function of temperature in Figures 53 through 56. Figures 53, 54, and 55 illustrate the results on individual wires and show how the insulation resistance varies not only with temperature, but also with the degree of redrying. Figure 56 is a comparison of the various wires after thorough redrying. From this illustration it is readily apparent that the wire in which the Teflon TFE was sintered for 126 seconds at 538°C (1000°F) was inferior to the wire in which the sintering was completed in 66 seconds. This is very likely due to the degradation of either the Kapton or "pyrrone" undercoat at the 538°C (1000°F) sintering temperature.

It will also be noted that the double wrapped Teflon TFE overcoated wire was superior to its single wrapped counterpart up to 343°C (650°F), but that it is essentially equal to or slightly poorer than the single wrapped wire between 343°C (650°F) and 427°C (800°F). Both the double wrapped and single wrapped wires certainly recovered very well from the humidity exposure and both show excellent insulation resistance up to 427°C (800°F).

Figures 57 and 58 are comparisons between Teflon overcoated "pyrrone"-Kapton insulated wires which had not experienced high temperature humidity exposure and which had been redried after the high humidity exposure. In both cases the humidity exposed wire was better after drying than a similar wire which had not experienced the extended humidity test. This observation was also made on "pyrrone"-Kapton wire which had no Teflon overcoat. No explanation for this phenomenon is available at the present time.

Another set of humidity tests was conducted on nine wires. This series included various "pyrrone" (HR-100)-Kapton insulated wires as well as one P13N-Kapton insulated wire. Insulation resistance measurements were made before exposure and after exposure to the 95 percent relative humidity and 82°C (180°F) for 28 days (672 hours). Wires were retested, then dried at 120°C for 16 hours and retested again. Results are shown in Table XXIX.

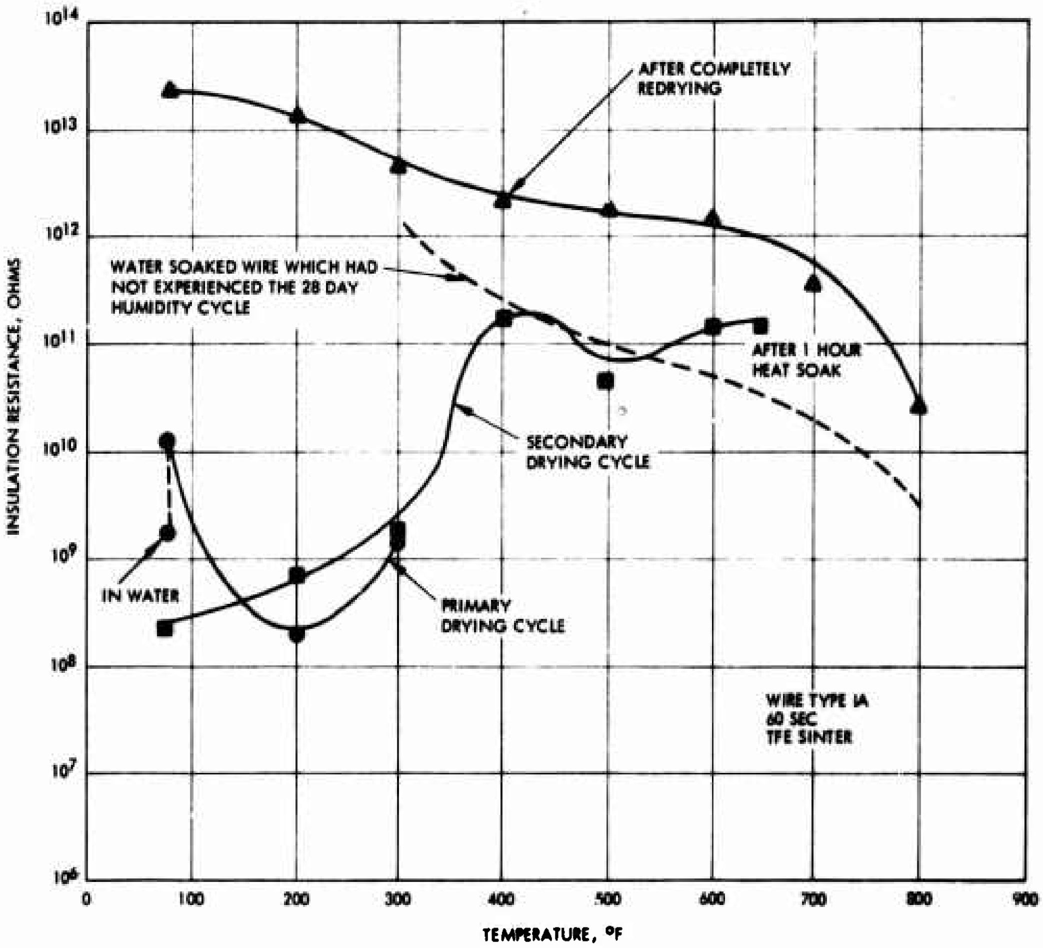


Figure 53. Insulation resistance of Teflon TFE overwrapped "pyrrone"-Kapton insulated wire after 28 days at 95 percent relative humidity (single wrap, 60 sec sinter)

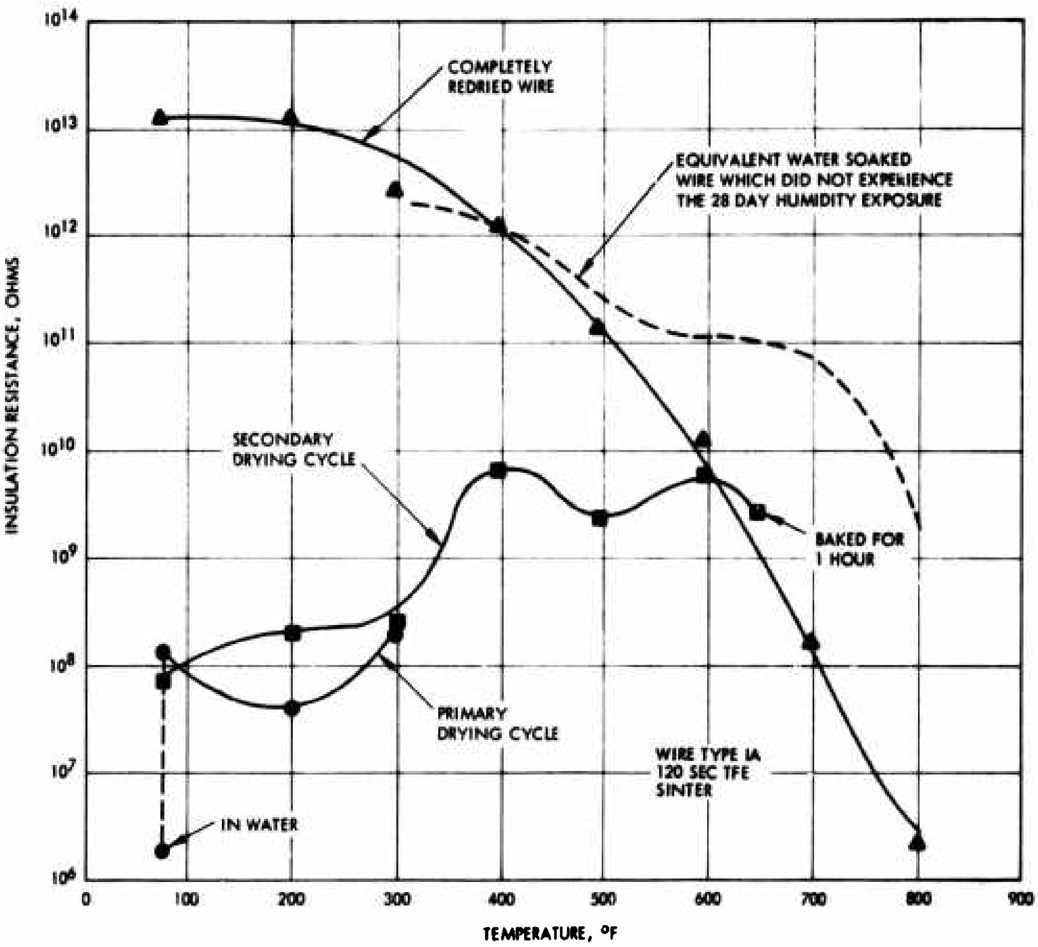


Figure 54. Insulation resistance of Teflon TFE overwrapped "pyrrone"-Kapton insulated wire after 28 days at 95 percent relative humidity (single wrap - 120 sec TFE sinter)

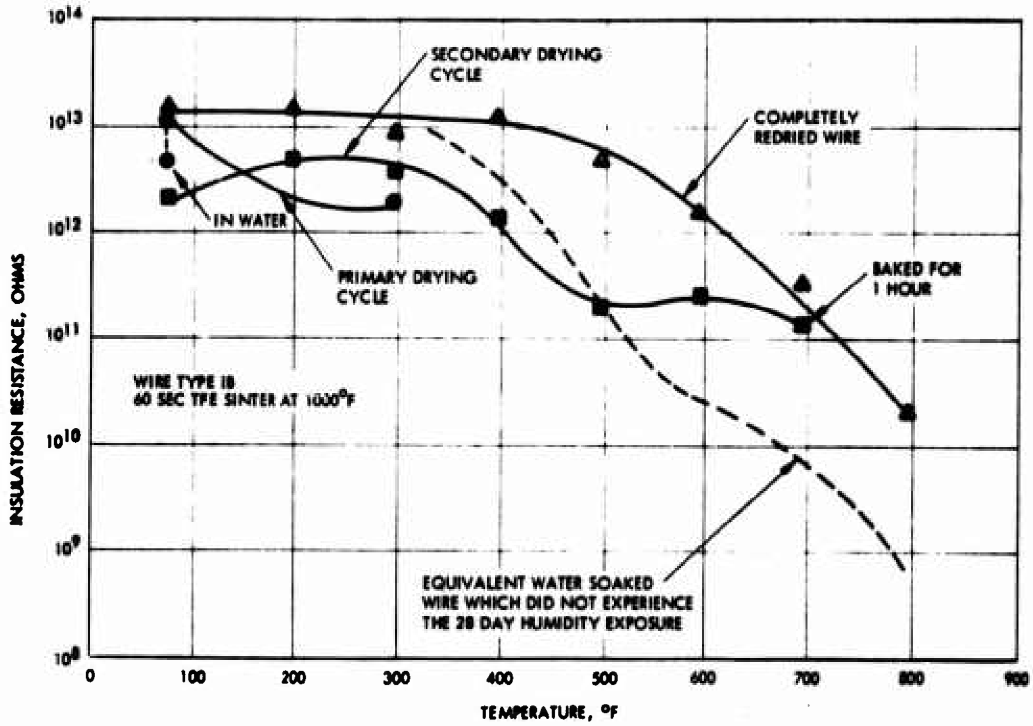


Figure 55. Insulation resistance of Teflon TFE overwrapped "pyrrone"-Kapton insulated wire after 28 days at 95 percent relative humidity (double wrap, 60 sec sinter)

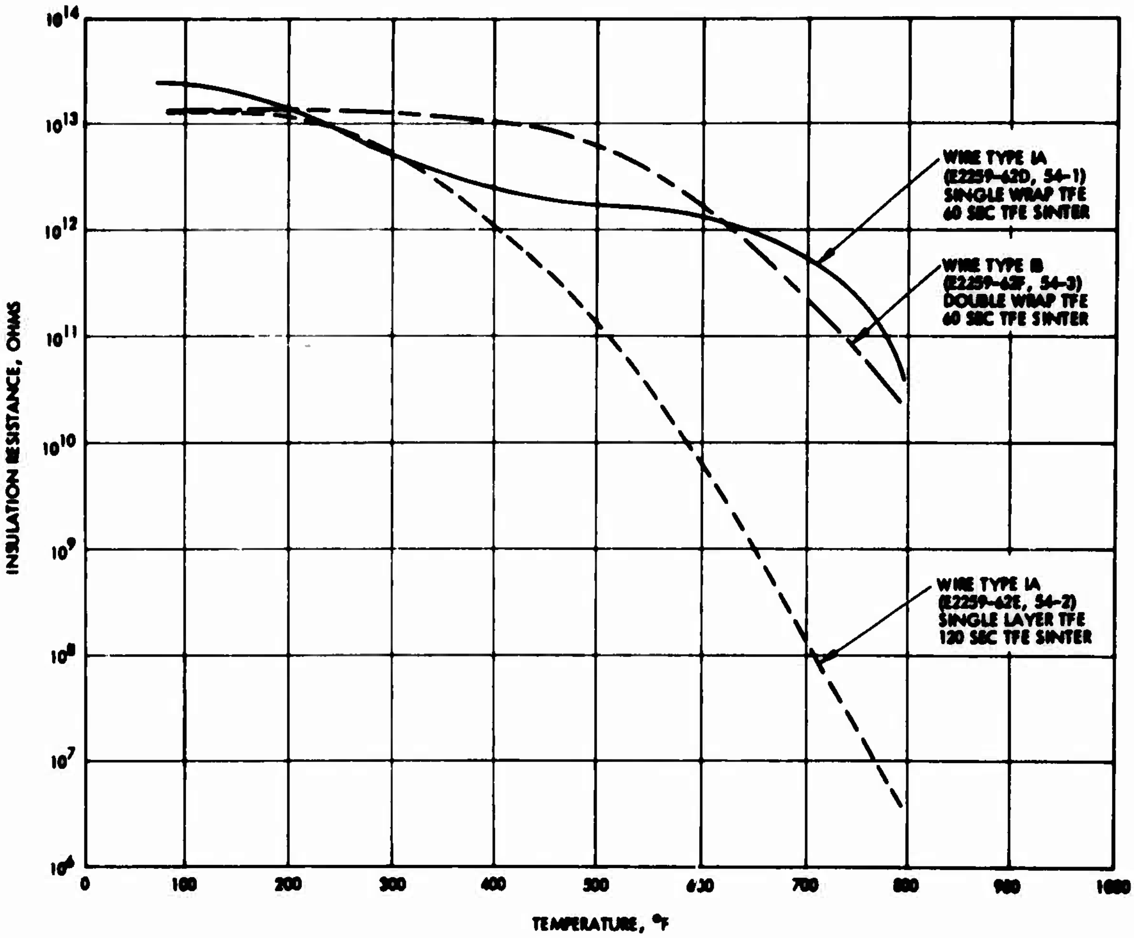


Figure 56. Insulation resistance of redried Teflon TFE overwrapped "pyrrone"-Kapton insulated wire after the 28-day exposure at 95 percent relative humidity

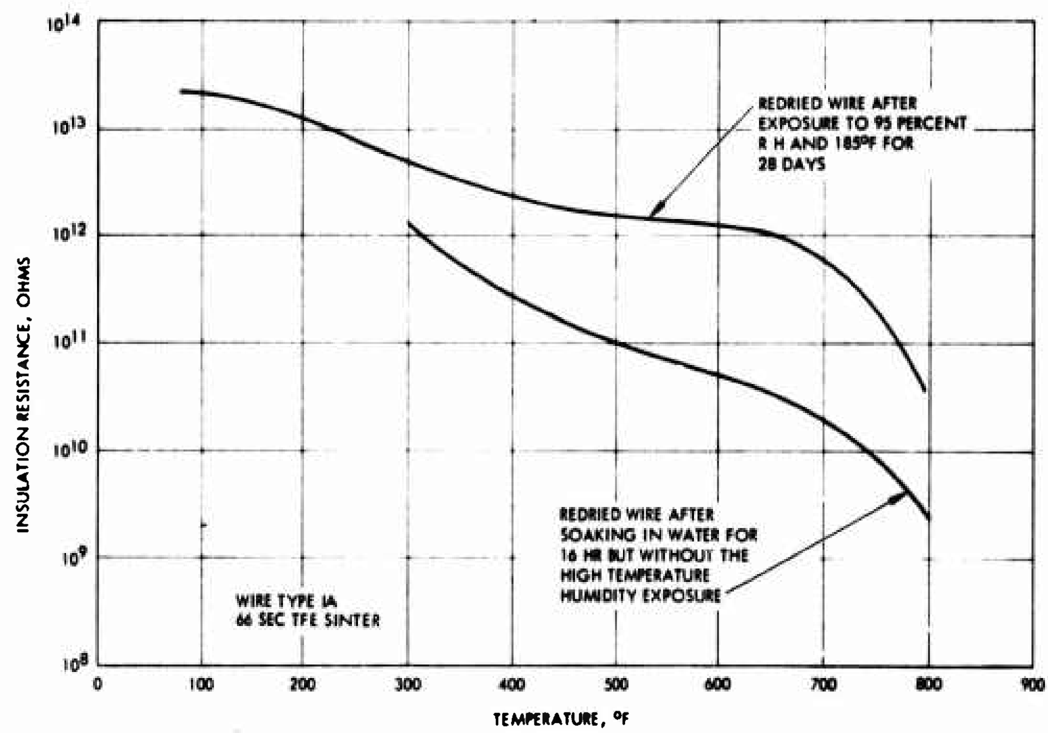


Figure 57. Insulation resistance of Teflon TFE overwrapped "pyrrone"-Kapton insulated wire before and after humidity test

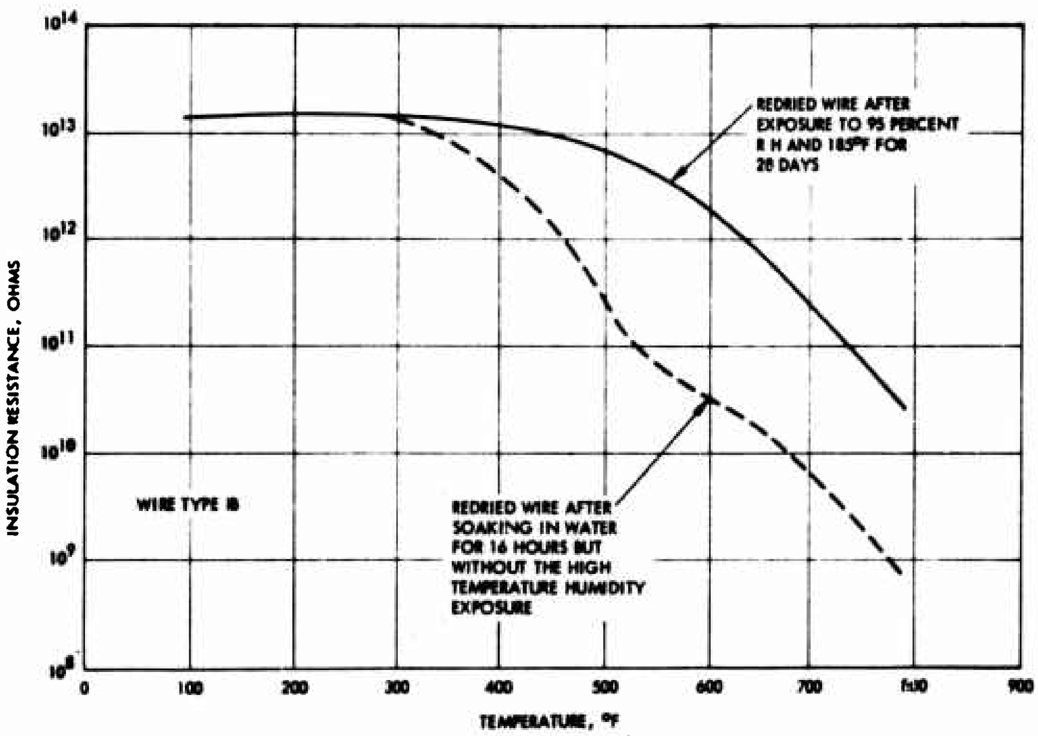


Figure 58. Insulation resistance of double overwrapped Teflon TFE on "pyrrone"-Kapton insulated wire before and after humidity test

Table XXIX. Insulation Resistance of Various Wires After 28-Day Humidity Exposure

Wire Type	Wire No. E2259-98		Insulation Resistance,* Ohms		
			Before Exposure	After Exposure	After Exposure and Redrying
I	-1	a	2×10^{13}	4×10^{8}	5×10^{10}
		b	1×10^{13}	5×10^{10}	2×10^{13}
II	-2	a	6×10^{13}	3×10^{13}	2×10^{13}
		b	7×10^{13}	2×10^{13}	4×10^{13}
IA	-3	a	6×10^{13}	6×10^{12}	6×10^{13}
		b	4×10^{13}	6×10^{12}	5×10^{13}
IB	-4	a	2×10^{14}	6×10^{13}	3×10^{14}
		b	2×10^{14}	3×10^{13}	5×10^{14}
IC	-5	a	2×10^{14}	7×10^{13}	5×10^{14}
		b	2×10^{14}	1×10^{14}	5×10^{14}
IIA	-6	a	2×10^{13}	2×10^{11}	2×10^{13}
		b	5×10^{13}	1×10^{8}	3×10^{8}
IIB	-7	a	2×10^{14}	1×10^{14}	5×10^{14}
		b	2×10^{14}	1×10^{14}	3×10^{14}
IIC	-8	a	3×10^{14}	1×10^{13}	3×10^{14}
		b	3×10^{14}	6×10^{13}	5×10^{14}
IV	-9	a	3×10^{13}	3×10^{13}	5×10^{12}
		b	4×10^{13}	3×10^{13}	1×10^{13}

*30 inch lengths.

It is evident from these tests that most of the wires held up very well. One of the non-coated single wrapped "pyrrone"-Kapton wires (I) and one of the overcoated double wrapped "pyrrone"-Kapton wires showed a significant drop in the insulation resistance but in both cases the corresponding duplicate wire was affected much less.

The effect of humidity on wires tested in this series was, in general, much less than that observed on earlier series such as the perfluoroalkylenetriazine overcoated wires (Type ID). The results shown in Table XXIX do, however, contain data on wires in a more advanced state of development.

3. DIELECTRIC BREAKDOWN

Dielectric breakdown measurements were also made on nine types of wires. Tests were performed by applying an A.C. potential between the anionic wetted water and the wire conductors in which the wires were submerged. Voltage was increased from zero, at a rate not to exceed 500 volts per second, to a potential of 2500 volts. After 1 minute at 2500 volts, the voltage was decreased to zero, at a rate of 500 volts/sec. After this test was completed, total breakdown was run. Results are shown in Table XXX.

4. INSULATION CUT-THROUGH TESTS

Cut-through tests were performed on "pyrrone" (HR-100)-Kapton insulated wire (Type I) and on Teflon TFE overwrapped "pyrrone"-Kapton insulated wire (Type IA and IB). Ambient temperature tests were conducted by applying an 8000 g load to the insulated wire. The deformation wedge had a radius of 0.015 inch. All three wires passed the 24-hour test without electrical shorting. Tests were performed at 260°C (500°F), 316°C (600°F), and 371°C (700°F), using a 3000 g load. The 0.015 inch radius deformation wedge was used in all cases. The wires also passed these tests after 24 hours at the designated temperature. Electrical shorting was not observed in any case. The test device is illustrated in Figure 59.

Table XXX. Dielectric Breakdown Measurements on Various Wires

Wire Type	Insulation Resistance,** ohms		Test to 2500 * Vac	Breakdown Voltage KV	Total Insulation Thickness, mils (Both walls)	Kilovolts per mil of wall
	Before	After				
I	4×10^{13}	1×13^{13}	Passed	7.5	6.5	2.4
II	6×10^{13}	4×10^{13}	Passed	16.5	8.5	3.8
IA	7×10^{13}	1×10^{13}	Passed	16.0	10.5	3.2
IB	1×10^{14}	6×10^{13}	Passed	15.0	14.5	2.0
IC	2×10^{14}	2×10^{14}	Passed	22.5	19.0	2.4
IIA	2×10^{13}	2×10^{13}	Passed	7.0	12.5	1.2
IIB	2×10^{14}	1×10^{14}	Passed	18.0	16.5	2.2
IIC	2×10^{14}	2×10^{13}	Passed	25.0	21.0	2.4
IV	7×10^{13}	8×10^{13}	Passed	16.0	3.0	10.6

*Requirement of MIL-W-81381 **30 inch lengths

5. LIFE CYCLE BEND TESTS

Life cycle bend tests were conducted in accordance with specification MIL-W-81381 (Paragraph 4.7.5.12-1, 2, and 3). The wire samples with 3/4 pound weights on each end were hung over a 1/2 inch diameter mandrel in an air oven at 316°C (600°F) for 120 hours. They were then cooled to ambient temperature, removed from tension, and straightened. The non-overcoated "pyrrone"-Kapton insulated wire (Type I) showed some signs of delamination and there were fine cracks along the full length of the wire. However, neither of the Teflon overwrapped wires (IA and IB) showed visible damage.

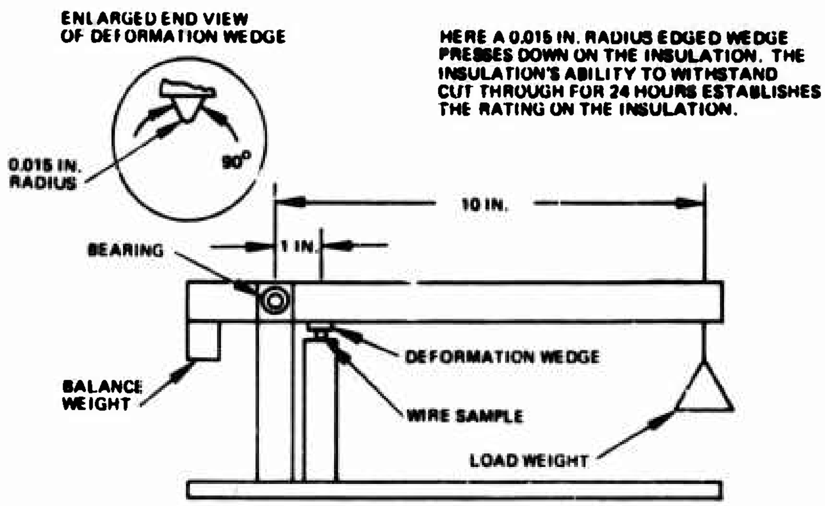


Figure 59. Insulation cut through tester

Wires were then wound with tension around the 1/2" diameter mandrel for their full length, and then unwound and rewound in the opposite direction. As a result of this wrapping and rewrapping, the Type I wire delaminated approximately every 1/2 inch. However, neither of the Teflon overwrapped wires (IA and IB) showed evidence of damage.

Subsequently, insulation resistance measurements were made at ambient temperature in a 5 percent aqueous sodium chloride solution. Both of the overwrapped "pyrrone"-Kapton insulated wires (IA and IB) held up extremely well, whereas the non-wrapped single layer "pyrrone"-Kapton insulated wire shorted. The test results are shown in Table XXXI.

Life cycle tests on the non-coated (Type I) and the Teflon TFE overcoated wires (IA and IB) were also conducted using a 1/4 inch diameter mandrel. Here also samples were exposed to air at 316°C (600°F) for 120 hours. None of the wires passed this test since they all shorted when insulation resistance measurements were made (in 5 percent NaCl solution).

Thus, although none of these three wires could pass the 1/4 inch mandrel test, the Teflon overcoated wires successfully passed the 1/2 inch diameter mandrel test.

Table XXXI. Insulation Resistance of Various Wires After 1/2 Inch Diameter Mandrel Bend Tests

Insulation	Wire No.	Test No. E2259-	Insulation Resistance, Ohms	
			Before	After
"Pyrrone"-Kapton single layer	I	71A	1×10^{7}	Shorted
"Pyrrone"-Kapton with Teflon over-wrap	IA	71B	2×10^{12}	3×10^{11}
"Pyrrone"-Kapton with Teflon over-wrap (double)	IB	71C	1×10^{12}	6×10^{11}

6. COLD BEND TESTS (MIL-W-81381-4.7.5.9)

Cold bend tests were conducted on various wires at -65° ±2°C after they had been exposed to this temperature for 4 hours. These tests were performed by wrapping the wires around a 1/4 inch diameter mandrel revolving at a rate of 2 r.p.m. Wires were removed and inspected for damage and insulation resistance measurements were taken and compared to measurements made before exposure.

Results of these tests are shown in Table XXXII. Wire I showed the greatest change, falling from 10^{13} ohms to 10^{6} and 10^{10}. It was a single wrapped "pyrrone" (HR-100) on Kapton (2 mil) insulation. In an earlier test a similar wire also showed significant change when wrapped on the 1/4 inch mandrel, but performed well when wrapped on a 1/2 inch diameter mandrel.

In contrast to wire I, wire II showed virtually no change. The latter was a double wrapped "pyrrone" (HR-100) on 1 mil Kapton. Thus, the advantages of double wrapping are quite evident. Wires IA and IIA have the same substrates as I and II respectively, but in addition they have a single layer Teflon TFE overwrap. These wires showed 2 and 3 orders of magnitude change respectively (from 10^{13} to 10^{11} and 10^{13} to 10^{10} ohms). A change of this magnitude does not

Table XXXII. Insulation Resistance as a Result of Cold Bend Tests

Wire Type	Insulation Resistance (Ohms)		Comments
	Before (average of duplicates)	After (average of duplicates)	
I	3×10^{13}	2×10^{6} and 9×10^{9}	Wrinkled but no evident cracks
II	5×10^{13}	1×10^{13}	Wrinkled but no evident cracks
IA	4×10^{13}	5×10^{11}	Wrinkled but no evident cracks
IB	8×10^{13}	4×10^{12}	Wrinkled, one crack evident in the TFE overwrap
IC	2×10^{14}	2×10^{13}	
IIA	4×10^{13}	1×10^{10}	Badly cracked
IIB	2×10^{14}	1×10^{13}	
IIC	2×10^{14}	2×10^{13}	
IV	6×10^{13}	5×10^{12}	

appear serious although it certainly is undesirable. The remaining wires, IB, IC, IIB, IIC, and IV all showed only one order of magnitude change. This is definitely insignificant and is typical of the variations normally experienced.

Cold bend tests were also performed on four other types of wires. These tests were run in accordance with MIL-W-81381, paragraph 4.7.5.9, with the exception that the weight applied to the wire was 1.5 pounds rather than 0.75 pound because the latter was insufficient to provide a tight wrap on the mandrel. Samples were tested in duplicate and results of these tests are shown in Table XXXIII.

Table XXXIII. Insulation Resistance of "Pyrrone"(HR-100)-Kapton Insulated Wires After Cold Bend Tests

Wire Type	Type of Overcoat	Insulation Resistance, Ohms		Comments
		Before	After	
I	None	3×10^6	Shorted	No cracking, but some delamination
		4×10^7	5×10^5	No cracking, but some delamination
ID	Perfluoro-alkylene-triazine	1×10^{13}	7×10^{11}	No cracking, but some delamination
		2×10^{12}	6×10^6	No cracking, but some delamination
IA	Single wrapped Teflon	8×10^{11}	1×10^{13}	No cracking, and no delamination
IB	Double wrapped Teflon	8×10^{12} 6×10^{12}	3×10^{10} 2×10^{12}	No cracking, but some delamination

Tests were conducted by winding the wires on a mandrel at -65°C (-85°F) after a 4-hour conditioning at this temperature, and insulation resistance measurements then were made at ambient temperature in a 5 percent sodium chloride solution. The Teflon TFE overcoated wires showed very minor changes, whereas one of the perfluoroalkylenetriazine overcoated wires and one of the noncoated wires showed a significant loss of properties.

PRECEDING PAGE BLANK-NOT FILMED

SECTION IV
SUMMARY AND CONCLUSIONS

Perfluoroalkylenetriazine, polybenzothiazole, polyterphenylene-oxide, silicone XR-4083, P13N polyimide, polyimidazoquinazoline and two types of "pyrrone" polymers were evaluated as potential electrical wire insulation.

All of these materials have good electrical properties at ambient temperature although zinc oxide filled polybenzothiazole has a high dissipation factor and the insulation resistance of perfluoroalkylenetriazine drops somewhat more rapidly with increasing temperature than several of the other polymers.

Polyterphenyleneoxide had exceptionally good electrical properties but was too brittle to use as a wire insulation. Polyimidazoquinazoline was also too brittle.

All of these polymers were evaluated as adhesives for Kapton polyimide in an effort to utilize them in wrapped insulation. In this respect the perfluoroalkylenetriazine, polyterphenyleneoxide and silicone XR-4083 were especially poor, particularly at temperatures over 260°C (500°F).

Materials which failed to provide adequate adhesion for Kapton polyimide were evaluated as overcoats for "pyrrone" bonded Kapton insulation. In this capacity the perfluoroalkylenetriazine was useful for short periods up to 600-700°F (316-371°C) but it could not take prolonged exposure in this temperature range.

Polybenzothiazole was a good adhesive for Kapton when filled with zinc oxide, but the filler degraded its electrical properties. Without the zinc oxide it was not as good an adhesive and its thermal stability was poorer.

A large portion of the research effort was devoted to the study of HR-100 pyrrone and P13N polymide because these materials provided excellent adhesion for Kapton as well as excellent long term thermal stability and good dielectric properties.

Both of these materials were successfully converted into wrapped insulations after they were coated onto 1 mil Kapton polyimide tape. In both cases double wrapped wires were significantly superior to single wrapped wires. Triple wraps were better yet, but by a small margin. Both types of wires appeared to have virtually indefinite life expectancy at 260°C (500°F), survived over 2200 hours at 288°C (550°F) and over 1000 hours at 316°C (600°F). "Pyrrone"-Kapton insulation also survived 100 hours at 371°C (700°F) in a double wrap, and over 150 hours in a triple wrap.

Teflon (TFE or TFE/FEP) overcoats on the P13N-Kapton or "pyrrone"-Kapton insulation improved wires which were to see temperatures of up to 288°C (550°F). However, at 316°C (600°F) such overwraps generally reduced the rate of electrical breakdown but they cracked and peeled and consequently cannot be recommended for such exposure.

Cold bend tests, life cycle bend tests, 95 percent R.H. - 85°C humidity tests, cut-through resistance tests and insulation breakdown tests were also made on the "pyrrone" (HR-100)-Kapton wrapped wires. All of these tests were successful.

In conclusion it is evident that "pyrrone"-Kapton or P13N-Kapton combinations can provide useful wrapped insulations for multifilament connector wire which is to see temperatures of up to 371°C (700°F).

Unclassified
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1 ORIGINATING ACTIVITY (Corporate author) Hughes Aircraft Company Centinela and Teale Culver City, California 90230	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3 REPORT TITLE

HIGH TEMPERATURE ELECTRICAL WIRE COATINGS

4 DESCRIPTIVE NOTES (Type of report and inclusive dates)
Technical Report - Feb 1969 - April 1970

5 AUTHOR(S) (First name, middle initial, last name)
Norman Bilow
Kenneth L. Rose

6 REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
July 1970	112	

8a. CONTRACT OR GRANT NO. FB3615-68-C-1322 b. PROJECT NO. 7340 c. Task No. 734007 d.	9a. ORIGINATOR'S REPORT NUMBER(S) AFML-TR-69-111, Part II 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10 DISTRIBUTION STATEMENT Each transmittal of this document outside of the Department of Defense must have prior approval of the Elastomers and Coatings Branch, MANE, Nonmetallic Materials Division, Air Force Materials Laboratory, Wright Patterson AFB, Ohio 45433

11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY
	Air Force Materials Laboratory Wright-Patterson Air Force Base, Ohio 45433

13 ABSTRACT

The purpose of this program was to develop high temperature resistant electrical insulation coatings for aerospace or hook-up wire for use on advanced high speed Air Force systems. Perfluoroalkylenetriazine, polybenzothiazole, polyterphenyleneoxide, silicone XR-4083, P13N polyimide, polyimidazoquinazoline and two types of "pyrrone" polymers were investigated as potential electrical wire coatings. All of these materials have good electrical properties at ambient temperature although zinc oxide filled polybenzothiazole has a high dissipation factor and the insulation resistance of perfluoroalkylenetriazine drops somewhat more rapidly with increasing temperature than several of the other polymers. Polyterphenyleneoxide had exceptionally good electrical properties but was too brittle to use as a wire insulation. Polyimidazoquinazoline was also too brittle. All of these polymers were evaluated as adhesives for Kapton polyimide tape in an effort to utilize them in wrapped insulation. In this respect the perfluoroalkylenetriazine, polyterphenyleneoxide and silicone XR-4083 were especially poor, particularly at temperatures over 260°C (500°F). Materials which failed to provide adequate adhesion for Kapton polyimide tape were evaluated as overcoats for "pyrrone" bonded Kapton insulation. A large portion of the research effort was devoted to the study of HR-100 "pyrrone" and P13N polymide because these materials provided excellent adhesion for Kapton as well as excellent long term thermal stability and good dielectric properties. It was determined that "pyrrone" (HR-100)-Kapton or P13N-Kapton combinations can provide useful wrapped insulations for multifilament connector wire which is to see temperatures of up to 371°C (700°F).

DD FORM 1 NOV 65 1473

Unclassified
Security Classification

Unclassified
Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Electrical Wire						
High Temperature Polymeric Insulation						
Wire Coatings						
Insulation						
Polyimidazopyrrolone						
Perfluoroalkylenetriazine						
Silicone Carborane						
Polyphenylsilane						
Curing Agent						
Polyimide						
Pyrrone						
Polybenzothiazole						
Polyterphenyleneoxide						
Polyimidazoquinazoline						
Polyimide P13N						

Unclassified
Security Classification