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DEVELOPMENT OF HIGH TEMPERATURE (1250 F) WIRE AND CONNECTORS.(U)
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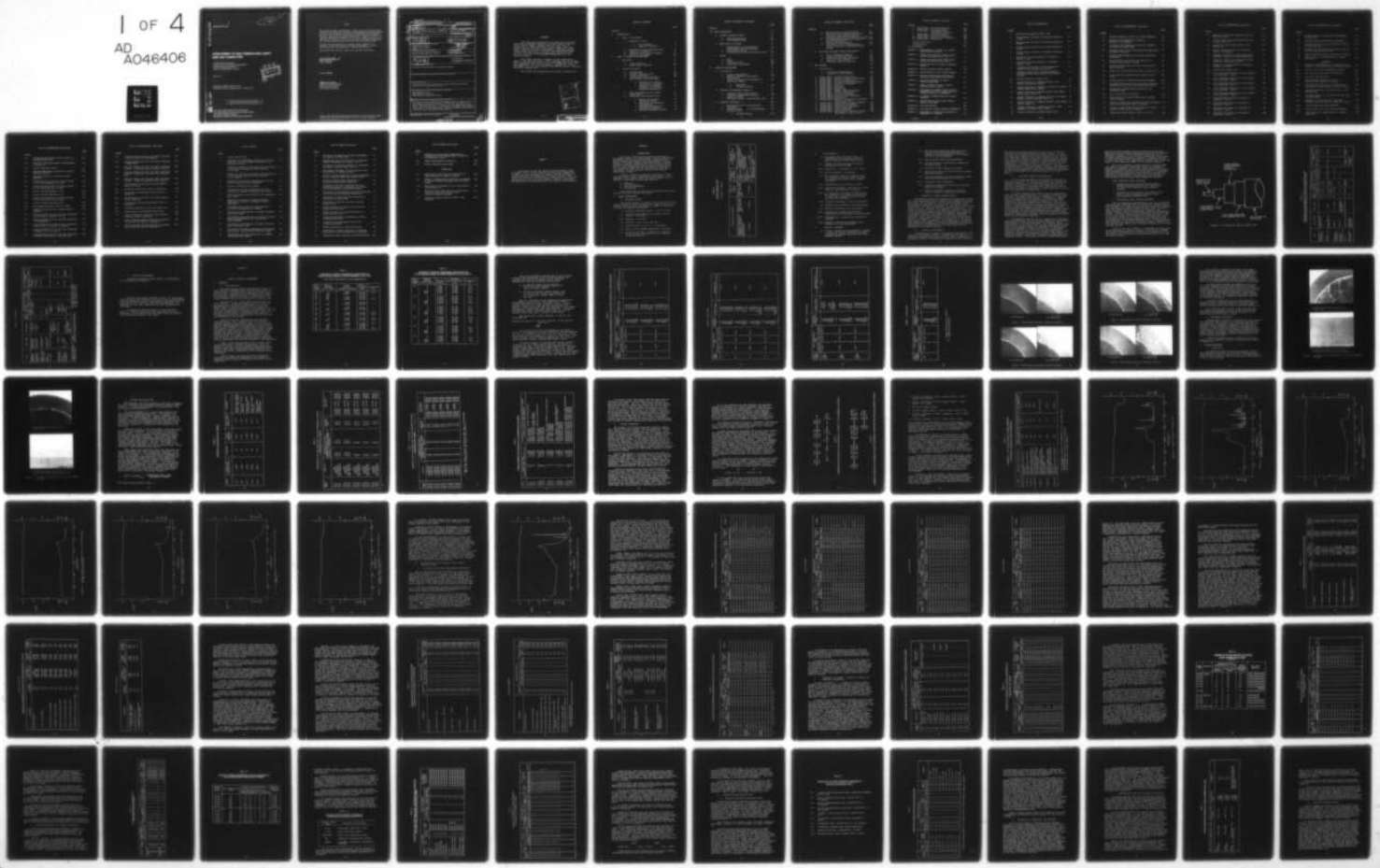

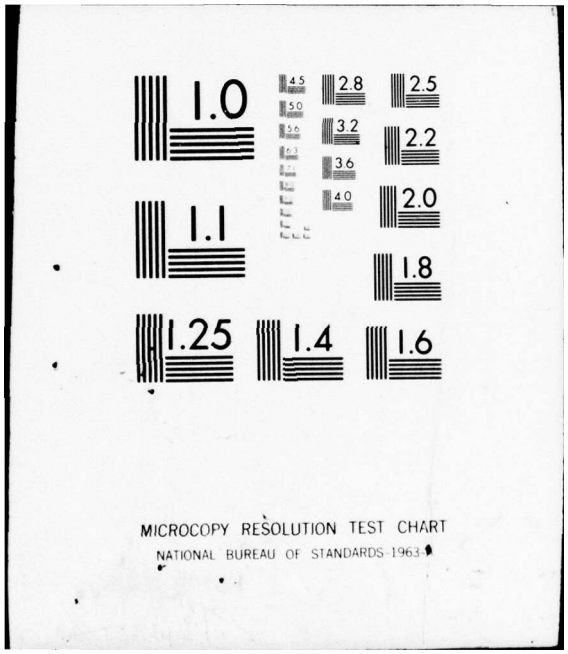
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DEVELOPMENT OF HIGH TEMPERATURE (1250°F) WIRE AND CONNECTORS

CANADA WIRE AND CABLE
TECHNOLOGY DEVELOPMENT DEPARTMENT
NORANDA RESEARCH CENTRE
POINTE CLAIRE, QUEBEC, CANADA

JULY 1977

TECHNICAL REPORT AFAPL-TR-77-25
Final Report for Period 1 February 1975 – 31 January 1977

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This technical report has been reviewed and is approved for publication.

Paul R. Bertheaud
Project Engineer

FOR THE COMMANDER

Stephen P. Condon, Major USAF
Chief, Vehicle Power Branch
Aerospace Power Division
Aero Propulsion Laboratory

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This report covers the research and development efforts in Phases I and II of the program to develop high temperature (1250°F) wire and connectors for general purpose aerospace use. Included are sections summarizing the Phase I effort, fabrication and testing of the previously established prime conductor, optimization of the insulation package, connector development and fabrication, and the drafting of the requisite test methods.

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FOREWORD

This report was prepared by the Canada Wire and Cable Technology Development Department, Noranda Research Centre, Pointe Claire, Quebec, Canada, under the joint U.S.A.F. Contract No. AF 33615-75-C-2040, project number 3145, task number 31452935 and Canadian Department of National Defence D REO Project DIR XE-175. This work was administered under the Air Force Aero Propulsion Laboratory, Aerospace Power Division, with Mr. P. R. Bertheaud (AFAPL/POP-2) as the Project Engineer.

This report contains a summary of work conducted in Phase I, (AF 33615-71-C-1768), from 1 February 1972 to 30 April 1974, previously published in July 1974 as AFAPL-TR-74-57, NTIS AD74-786890/4GA, and the final report, (sequence No. A-007), for Phase II of the titled program covering the period 1 February 1975 to 31 January 1977.

This report was submitted by the authors in March 1977.

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SUMMARY

This report covers the research and development effort in Phases I and II of the program to develop high temperature (1250°F) wire and connectors for general purpose aerospace use. Included are sections summarizing the Phase I effort, fabrication and testing of the previously established prime conductor, optimization of the insulation package, connector development and fabrication, and the drafting of the requisite test methods.

SECTION I

INTRODUCTION

The overall program was designed to be executed in two phases, namely: (1) a design study, including experimental determination of appropriate electrical, mechanical and material properties, leading to the establishment of design criteria for general purpose electrical wire and connectors capable of continuous operation in a 1250°F ambient environment to various performance specifications as established in a Statement of Work, and (2) the fabrication and test of experimental wire and connectors based on the design(s) established in Phase I.

A report covering the successful completion of Phase I has been issued([1]) and is summarized in this section. The remainder of this report covers the Phase II effort, and, for clarity is subdivided into four main sections:

1. Materials
2. Wire Fabrication
3. Connector Development
4. Test Methods

Within these main sections are various subsections which discuss individual component development.

A Phase I Development

1. Statement of Work

The primary objective of Phase I was to develop the technology required to permit the design of a family of high temperature insulated wires and connectors capable of meeting the following performance requirements:

(i) Operating temperature range - -65°F to 1250°F.

(ii) Pressures corresponding to altitudes from sea level to 110,000 feet.

(iii) Humidity - 0 to 100%.

(iv) Vibration - 20 g, 10 to 2000 cps.

(v) Physical shock - 50 g, 11 ± 1 millisecond.

(vi) Design life at maximum temperature, 1000 hours.

(vii) Flight profile test - connector/wire assembly, 200 cycles, as detailed in Table 1 (to be performed in Phase II).

TABLE 1

FLIGHT PROFILE TEST

1 cycle = 5 hours

Waiting for Mission		Start Up on Ground	Extremes of Flight Conditions	Operating Conditions upon Landing
Temperature/humidity/ pressure, standard conditions of MIL-STD-202	Moisture soak 100% humidity 25°C	Moisture soak 100% humidity 25°C	-65°F* Rated power 1 hour	Temperature/humidity/ pressure, standard conditions of MIL-STD-202 Rated power 1 hour
			1250°F* Rated power 1 hour	
2 hours	1/2 hour	1/2 hour	* Samples are divided for two simultaneous tests	

a. Wire assembly

(i) Candidate electrical conductors to be determined; for evaluation purposes, sizes 12 AWG and 22 AWG shall be used.

(ii) Maximum continuous potential rating at temperature 600 volts RMS.

(iii) Resistivity - less than 6×10^{-6} ohm cm.

(iv) Tensile strength - 35,000 psi.

(v) The insulation shall be compatible with the conductor chosen and have an insulation resistance greater than 1 megohm at 1250°F.

(vi) Susceptibility to blocking at 1250°F.

(vii) Dielectric strength - 1200 volts to ground under all environmental conditions.

(viii) The wire shall be capable of being wound on a mandrel of not greater than 20X the finished wire diameter without physical or electrical detriment.

(ix) Insulation shrinkage - 0.06 inch maximum.

(x) Thermal shock resistance to be determined.

(xi) Abrasion resistance to be determined.

(xii) Sublimation at normal and elevated temperatures and altitudes to be determined.

(xiii) Producibility (including long production lengths).

(xiv) Degree of flexibility including flex-fatigue life to be determined.

b. Connector assembly

(i) Contact spacing and configuration to permit a contact to contact and contact to shell steady state working voltage of 600 volts maximum, 400 Hz.

(ii) The insulation resistance between any two contacts and between the shell and any contact of mated connectors at high temperature shall be 2000 megohms.

(iii) Define contact design and arrangement.

(iv) Determine contact resistance at temperature and after aging.

(v) Determine contact retention strength.

(vi) Define shell design including coupling method and cable support.

(vii) Provide resistance to ozone, hydraulic fluids, salt spray and kerosene (JP-4 or JP-5).

(viii) Determine corona resistance.

(ix) Establish compatibility with wire developed under this program.

(x) Provide interfacial sealing of connector halves to achieve a moisture-proof seal.

2 Theoretical Design Considerations

Considerable effort was expended to consider and evaluate the various approaches taken to develop high temperature resistant wires, by an exhaustive review of the published literature. Four potential approaches to wire design and assembly were established as potential candidates, (i) sheathed, gas dielectric filled wire, (ii) sheathed, dense dielectric packed wire, (iii) coated dielectric wire and (iv) composite dielectric wire. The latter was finally chosen as the most practical way to design a wire which would meet the program performance requirements as it offered a starting point based on previously disclosed approaches, and a high potential for significant technological advances. Furthermore, a composite dielectric wire was potentially most capable of meeting a high proportion of the design requirements and allowed the potential opportunity to employ a high proportion of commercially available materials which would keep the final system within the cost range that could be considered truly general purpose.

3 Conductor Development

Considering the conductor performance requirements stipulated in the Statement of Work, it became obvious that an acceptable conductor would be required to have a combination of excellent electrical and mechanical properties, both initially

and after aging while maintaining a high level of oxidation resistance for extended periods of time at 1250°F. It was felt that this could only be achieved economically by the use of a composite conductor which would combine a highly conductive core with a protective sheath of high mechanical strength. After canvassing numerous potential suppliers and evaluating submissions, a conductor based on a core of fine silver oversheathed with a protective layer of Inconel 600 alloy was established as the preferred conductor design. The performance of test sections of 12 and 22 AWG met or exceeded the design criteria in the Statement of Work and fulfilled the requirement for the establishment of a prototype design at the end of Phase I.

4 Insulation Materials Development

A brief study was made of the potential usefulness of dielectric thin films or refractory coatings for use over the preferred conductor. It was concluded that these materials offered little advantage in that they generally had a poor dielectric strength to thickness ratio, were brittle and had poor shock and impact resistances. Further exploration in this area was considered of little value.

In order to become more familiar with the approaches taken by other research workers, an in-depth literature review was made of programs describing the development of composite based high temperature wire systems. Concurrently, commercially available wires potentially useful in the service temperature range were obtained and evaluated. None of the wires as received were found to be of any value for multiple cycling in high temperature aerospace environments as they all lost a significant proportion of mechanical and electrical integrity after only short exposures to 1250°F. However, based on performance and theoretical considerations gathered from the literature, it was concluded that a wire having a primary dielectric consisting of multiple layers of tapes and/or braids should be the preferred approach.

Consideration was then given to selection of candidate insulation materials, the search being made under the following restrictions: a) the material should be thermally stable, significantly in excess of 1250°F, b) the material should show insignificant weight change, be preferably non-absorptive and show little tendency to smoke or exudation; c) the material should be available in a form that is easily handled and relatively thin in section, d) the material should have a high dielectric strength to thickness ratio and e) the material should be stable in the cyclic environment as described in the Statement of Work. Using these criteria, reconstituted mica

paper and braided sheaths based on refractory silica were identified as the preferred candidates from among the numerous materials evaluated. As it was realized that the use of refractory braids alone would require inordinately thick layers to achieve adequate dielectric strength, a composite design of inner and outer braids sandwiching a central core of multiple layers of mica paper was chosen as the preferred approach.

Due to the inherently fragile nature of reconstituted mica paper as supplied, significant effort was expended to identify an impregnant for the paper which would result in a composite dielectric having good initial electrical, mechanical and environmental properties, with retention of a significant proportion of these properties after multiple exposures to 1250°F, while maintaining an insignificant level of smoke or fume evolution during initial high temperature exposure.

Three approaches were considered:

1. Impregnation with easily pyrolyzed organic polymers which would result in a dense layer of "clean" mica dielectric after thermal exposure;

2. Impregnation with inorganic salts which would revert to more refractory materials after high temperature exposure, and

3. Impregnation with inorganic polymers.

After considerable experimentation, the first and second approaches were abandoned and all efforts were concentrated on the development of a mica/inorganic polymer dielectric composite. This approach was ultimately successful as a novel dielectric paper was identified which used as the impregnant the highly thermally stable poly(carborane siloxane) organometallic polymer. Multiple layers of this dielectric, applied as tapes over an inner braid of refractory silica and protected in turn by an outer braided layer of the same refractory silica met or exceeded the design requirements of the Statement of Work when evaluated as a prototype hand made wire. To further enhance the resistance of the wire to mechanical damage by abrasion or blocking, an outer armour layer of knitted Inconel 600 fine gauge wire was applied.

Thus, at the end of Phase I, a prototype wire design had been established, as shown in Figure 1, which met or exceeded the design requirements as stipulated in the Statement of Work. The results of the evaluation of prototype lengths of wire may be found in Table 2.

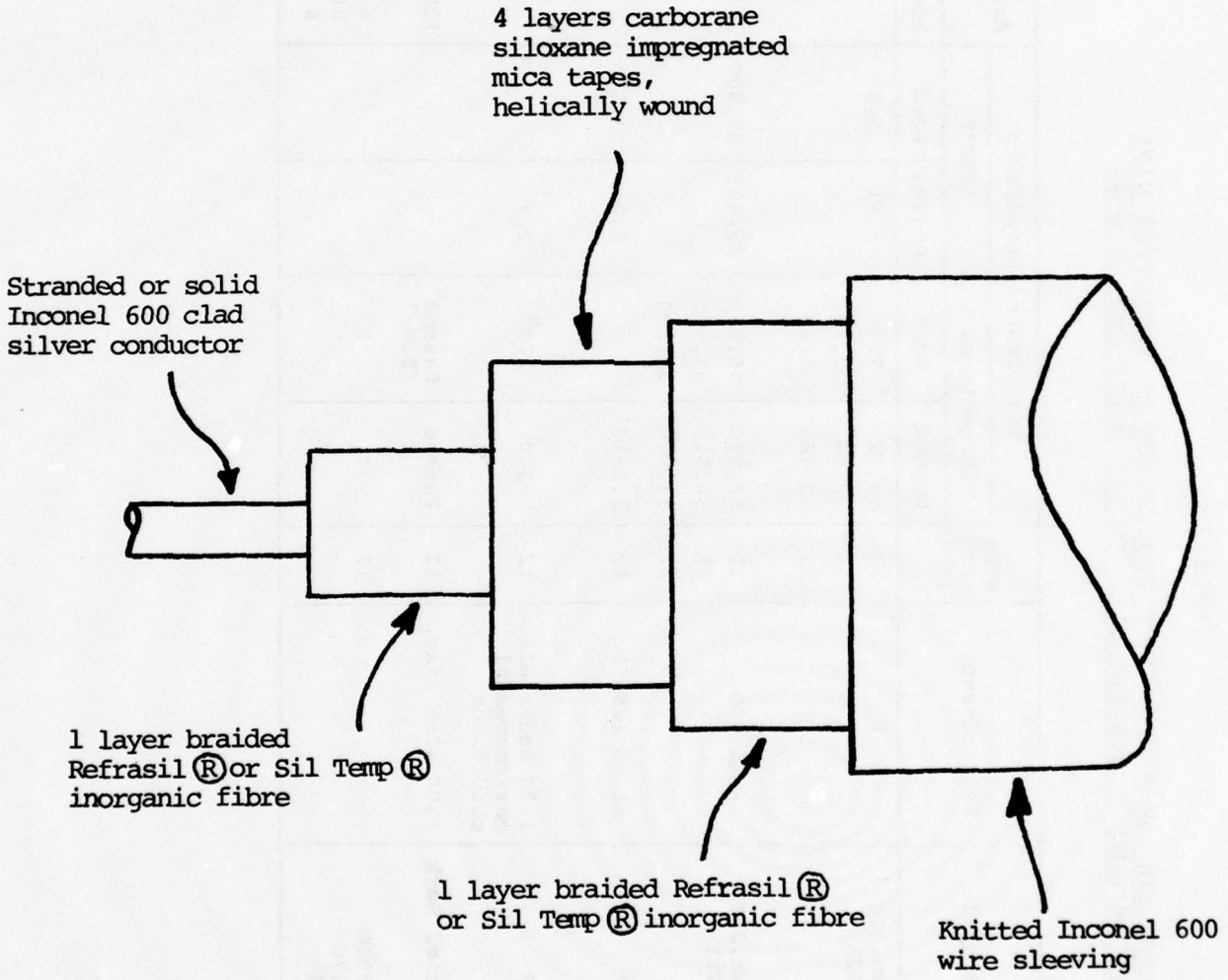


Figure 1. Prototype wire design, Phase I end.

TABLE 2

COMPARISON OF PERFORMANCE PROPERTIES OF PROTOTYPE WIRE WITH REQUIREMENTS DETAILED IN THE STATEMENT OF WORK

Test	Test method	Requirement[1]	Wire size (AWG)	Performance properties				Recommended performance standards	
				Room temp.		1250°F			
				Unaged	Aged[2]	Unaged	Aged[2]	Room temp.	1250°F
Conductor resistivity (microhm-cm)	Fed.Std.No.228 method 6021	<6	12	2.72	2.71	9.61	9.64	<4	<12
			18	2.84	2.78			"	"
			22	2.76	2.89			"	"
Tensile strength (psi)	Fed.Std.No.228 method 3211	35,000	12	58,900	56,000	38,800	30,300	>45,000	>25,000
			18	59,500	53,300			"	"
			22	54,000	51,200			"	"
Insulation resistance (megohms/ft.)	ASTM D257	>1 at 1250°F	12	1.3×10^7		8.0	3.5[a]	>1	>1
Dielectric strength (kilovolts/ac)	ASTM D149	1.20 under all environmental conditions	12	>5.0	2.0[b]	2.4		2.2	>1.2
Mandrel	Statement of Work	∤20X wire diam.	12	Passed	Passed[c] /D.W.[3]			∤20X wire diam.	∤20X wire diam.
Thermal shock resistance	MIL-STD-202D method 107C (modified)		12	"	"			5 cycles of -65°F & 1250°F	

TABLE 2 (continued)

Test	Test method	Requirement[1]	Wire size (AWG)	Performance properties				Recommended performance standards	
				Room temp.		1250°F			
				Unaged	Aged[2]	Unaged	Aged[2]	Room temp.	1250°F
Vibration resistance	MIL-STD-202D method 204B	10-2000 cps, 20 g's	12	Passed[d] /D.W.[3]	Passed[d] /D.W.[3]			20 g's/10-2000 cps	
Mechanical shock resistance	MIL-STD 202D method 213A	50 g's/11 ± 1 msec.	12	"	"			50 g's/11 ± 1 msec.	
Abrasion resistance (in. of garnet tape)	Fed.Std.No.228 method 2211		12	30				25 minimum	
Flame resistance	Fed.Std.No.228 method 5211		12	Passed /D.W.[3]					
Life cycle	Statement of Work	1) 1000 hr. at 1250°F 2) 600 volts at 1250°F for 1000 hr.		Planned for Phase II				See text	See text
Flexibility & flex fatigue	AF -TR-66-97	20X wire diam.	-	Planned for Phase II				20X	20X
Blocking	MI -27500A		-	Planned for Phase II					
Shrinkage (in.)	MIL-C-13777F	0.06	-	"				No test	No test
Sublimation	No test	At normal & elevated temperatures & altitudes	12	Minimal				-	Minimal

(1) As per the Statement of Work.
(2) Aged for 1000 hours at 1250°F (unless stated otherwise).
(3) After testing, passed dielectric withstand of 2.2 Kv/5 sec.

(a) After 180 hours at 1250°F
(b) After 800 hours at 1250°F
(c) After 5 hours at 1250°F
(d) After 25 hours at 1250°F

5 Connector Development

Connector development during Phase I is discussed in the Introduction to Section 4.

Based on the success achieved in Phase I of the program, Phase II, which was intended to fabricate and test experimental wire and connectors based on these designs, was approved by the United States Air Force Aero Propulsion Laboratories. The remainder of this report describes the work accomplished in this Phase.

It should be noted that, due to a cost overrun in Phase II, work was terminated before the program could be completed. Those objectives of the effort that were successfully achieved are noted in the report.

SECTION II

PHASE II MATERIALS DEVELOPMENT

A Conductor

1. Prime Conductor

The prime conductor design, established in Phase 1([1]), is an Inconel 600 sheathed silver core wire fabricated in 12 through 22 AWG. The fabrication procedures for this wire were developed in cooperation with the Reuter Stokes Company of Canada Ltd., Cambridge, Ontario, who undertook to supply the anticipated needs of the program, namely 1000 feet of 12 AWG conductor, and 100 feet of each of 14 through 22 AWG.

The order for the 12 AWG conductor was received very late in Phase I. Initial evaluation indicated([1]) that the performance would be entirely satisfactory. Upon being awarded Phase II, protracted negotations took place with Reuter Stokes, ultimately resulting in the placement of an order for the remaining 500 total feet of mixed gauge wire. Due to further delays because of extended delivery times for raw materials and difficulties with equipment and scheduling, the complete order was not received until over one year after the commencement of the program.

Testing of the long term physical and electrical characteristics of the 12 through 22 AWG prime conductor continued throughout the program. Table 3 lists the comparative physical performance characteristics of the various gauges of wire measured at room temperature, both before and after exposure to 1250°F for various times as noted. Samples were aged in a TEMCO® electric muffle furnace using a Barber Colman® Model 293C controller. Tensile strengths and elongations were determined using an Instron® Model 1122 with pneumatic action wire grips. A standard crosshead speed of 50 mm/minute (∿2 inches/minute) was employed. From the table it can be seen that all conductors easily exceeded the required performance of 35,000 psi, measured at room temperature.

Table 4 lists the performance of the various gauges of wire after selected periods of exposure to 1250°F, as measured at 1250°F. A tubular heating chamber sealed with asbestos plugs maintained the performance temperature around the wire during test. Temperature was monitored near the center of the 10-inch long specimen using a platinum rhodium thermocouple

From the table it can be seen that all conductors significantly exceeded the suggested([1]) specification of 25,000 psi tensile strength as measured at the upper service temperature.

TABLE 3

COMPARATIVE PHYSICAL PERFORMANCE CHARACTERISTICS
OF THE VARIOUS GAUGES OF PRIME CONDUCTOR (I AT 77°F)

(All tests performed at room temperature)

Wire Gauge (AWG)	Thermal Exposure (hr at 1250°F)	Wire Diameter (inches, averaged)	Tensile Strength (psia)	Elongation (%)
12	0	0.0805	56,200	21.5
12	250	0.081	54,700	-
12	500	-	55,300	-
12	750	0.0808	55,400	-
12	1000	0.0803	56,100	21.0
14	0	0.0639	58,600	-
14	1000	0.0638	58,700	-
16	0	0.0508	56,100	-
16	1000	0.051	56,100	-
18	0	0.0399	61,800	18.0
18	1000	0.0399	61,300	18.0
20	0	0.0320	57,900	17.5
20	1000	0.0324	57,100	18.0
22	0	0.0256	56,800	18.2
22	250	0.0259	54,300	-
22	500	-	53,500	-
22	1000	0.0257	56,200	18.5

TABLE 4

COMPARATIVE PHYSICAL PERFORMANCE CHARACTERISTICS
OF THE VARIOUS GAUGES OF PRIME CONDUCTOR (II AT 1250°F)

Wire Gauge (AWG)	Sample Exposure (hr at 1250°F)	Diameter (inches)	Ultimate Tensile Strength (psia)	Elongation (%)
12	0	0.804	38,800	13.5
	0	0.804	37,100	12.0
	0	0.803	37,200	12.5
	1000	0.805	32,600	10.3
	1000	0.805	33,200	10.0
14	0	0.0639	40,000	12.1
	0	0.0639	40,900	12.6
	0	0.0639	38,100	11.5
	1000	0.0639	35,600	11.5
	1000	0.0639	37,500	11.8
16	0	0.0507	41,100	11.7
	0	0.0507	39,300	11.2
	0	0.0507	40,500	12.0
	1000	0.0507	36,100	10.8
	1000	0.0507	33,800	10.0
18	0	0.0398	43,700	10.5
	0	0.0398	46,600	11.4
	1000	0.0398	36,000	8.8
	1000	0.0398	36,300	9.0
20	0	0.0324	39,400	-
	0	0.0324	37,000	10.0
	0	0.0324	38,800	12.0
	1000	0.0324	35,500	8.9
	1000	0.0323	35,100	9.0
22	0	0.0257	39,600	-
	0	0.0256	38,500	11.0
	1000	0.0256	34,600	9.5
	1000	0.0256	32,700	9.3

Electrical performance characteristics of the prime conductor were determined, using the test equipment and procedure described in the Phase I final report([1]), (Appendix A), with the following modifications:

a) An improved furnace which allowed a controlled 1250°F exposure zone for 16 inches of sample length;

b) The use of castable ceramic support jigs in place of the original mica spacers so as to evaluate a larger number of wires per test.

The D.C. resistance of the conductor was determined in accordance with Fed. Std. 228, Method 6021, using a Croydon four-terminal bridge, accuracy ± 0.01%. A direct constant current of 1.0 amps was maintained across the samples. Nickel potential leads were attached to the samples using Handy & Harman braze #541 with borax flux. Stray emf was equalized using a microvolt power source to zero the detector while there was no current in the bridge.

The resistivity of each conductor, in ohm cm, was calculated by:

$$\text{Resistivity (ohm cm)} = \text{resistance (ohm/cm)} \times \text{cross section area } (cm^2)$$

$$= \frac{R\pi d^2}{L4}$$

The results of the initial experiments are shown in Table 5. All conductors initially tested met the specification of $<4 \times 10^{-6}$ ohm cm at 77°F, and $<12 \times 10^{-6}$ ohm cm at 1250°F, both before and after exposure to 1250°F for 1000 hours. Also noted in Table V is the relative conductivity (in %) of the prime conductor versus a standard OFHC copper control.

As noted in Table 5, connector lead failure occurred on certain of the samples during exposure, necessitating repeating the test. On removing these samples from the oven, severe surface damage and embrittlement were noted. Metallographic examination of the conductors (Figures 2-5) showed evidence of significant deterioration of the Inconel 600 sheath, a phenomenon not experienced in Phase I studies, and not duplicated on samples under concurrent long term thermal aging in another furnace.

TABLE 5

COMPARATIVE ELECTRICAL CHARACTERISTICS OF THE VARIOUS GAUGES OF PRIME CONDUCTOR

Wire Gauge (AWG)	Wire Dimensions (mm)			Exposure Temperature (°F)	Resistivity (Ω cm x 10^{-6})	Conductivity (% of OFHC Copper Standard)
	Diameter	Area	Length			
12	2.047	3.291	349.0	63	2.784	61.9
				250	3.791	
				500	5.275	
				750	6.733	
				1000	8.366	
				1250	9.972	
				1250*	10.02**	
				91*	2.779**	
12	2.047	3.291	349.0	79	2.907	59.3
				250	3.857	
				500	5.297	
				750	6.870	
				1000	8.426	
				1250	10.07	
				1250*	10.09	
				63	2.866	
14	1.629	2.084	355.5	72	2.963	58.2
				250	3.878	
				500	5.366	
				750	6.460	
				1000	7.902	
				1250	9.132	
				1250*	10.02	
				66*	2.947	

TABLE 5 (continued)

Wire Gauge (AWG)	Wire Dimensions (mm)			Exposure Temperature (°F)	Resistivity (Ω cm x 10^{-6})	Conductivity (% of OFHC Copper Standard)
	Diameter	Area	Length			
16	1.298	1.323	355.5	72	3.196	53.9
				250	4.137	
				500	5.558	
				750	6.991	
				1000	8.529	
				1250	9.940	
				1250*	9.959	
				66*	2.888	
18	1.026	0.827	349.5	63	3.138	54.9
				250	4.290	
				500	5.954	
				750	7.579	
				1000	9.386	
				1250	11.18	
				1250*	11.33	
				91*	3.368	
20	0.828	0.538	338.5	63	2.875	59.9
				250	3.996	
				500	5.534	
				750	7.034	
				1000	8.719	
				1250	10.35	
				1250*	Connector failed	
				91*	"	
22	0.657	0.339	351.5	63	2.802	61.5
				250	3.839	
				500	5.327	
				750	6.783	
				1000	8.424	
				1250	9.990	
				1250*	Connector failed	
				91*	"	

TABLE 5 (continued)

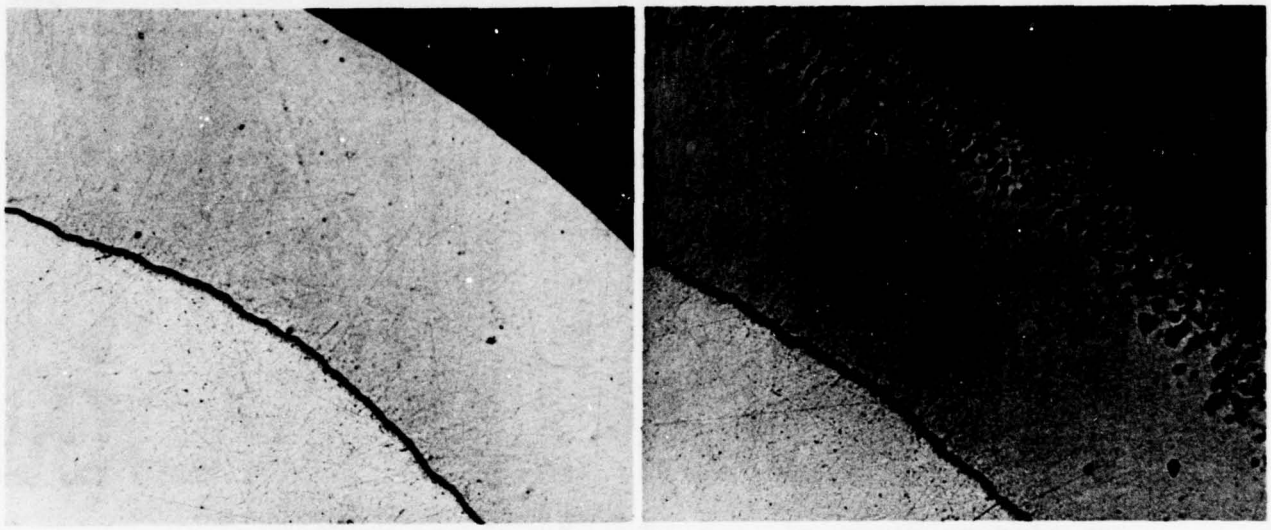
Wire Gauge (AWG)	Wire Dimensions (mm)			Exposure Temperature (°F)	Resistivity (Ω cm x 10^{-6})	Conductivity (% of OFHC Copper Standard)
	Diameter	Area	Length			
22 (Rept. #1)	0.657	0.339	355.5	77	2.799	61.6
				250	Shorted	
				500	"	
				750	"	
				1000	7.858	
				1250	10.15	
				1250*	Connector failed	
				91*	"	
20 (Rept. #1)	0.828	0.538	340.0	63	2.881	59.8
				250	3.995	
				500	5.537	
				750	7.041	
				1000	8.825	
				1250	10.35	
				1250*	10.68	
				91*	2.971	
22 (Rept. #2)	0.657	0.339	352.5	79	2.854	60.4
				250	3.781	
				500	5.237	
				750	6.770	
				1000	8.312	
				1250	9.982	
				1250*	10.04	
				63*	2.848	

TABLE 5 (continued)

Wire Gauge (AWG)	Wire Dimensions (mm)			Exposure Temperature (°F)	Resistivity (Ω cm x 10^{-6})	Conductivity (% of OFHC Copper Standard)
	Diameter	Area	Length			
22	0.657	0.339	349.0	79	2.861	60.3
				250	3.789	
				500	5.235	
				750	6.773	
				1000	8.316	
				1250	9.981	
				1250*	10.04	
				63*	2.862	

* After 1000 hours at 1250°F.

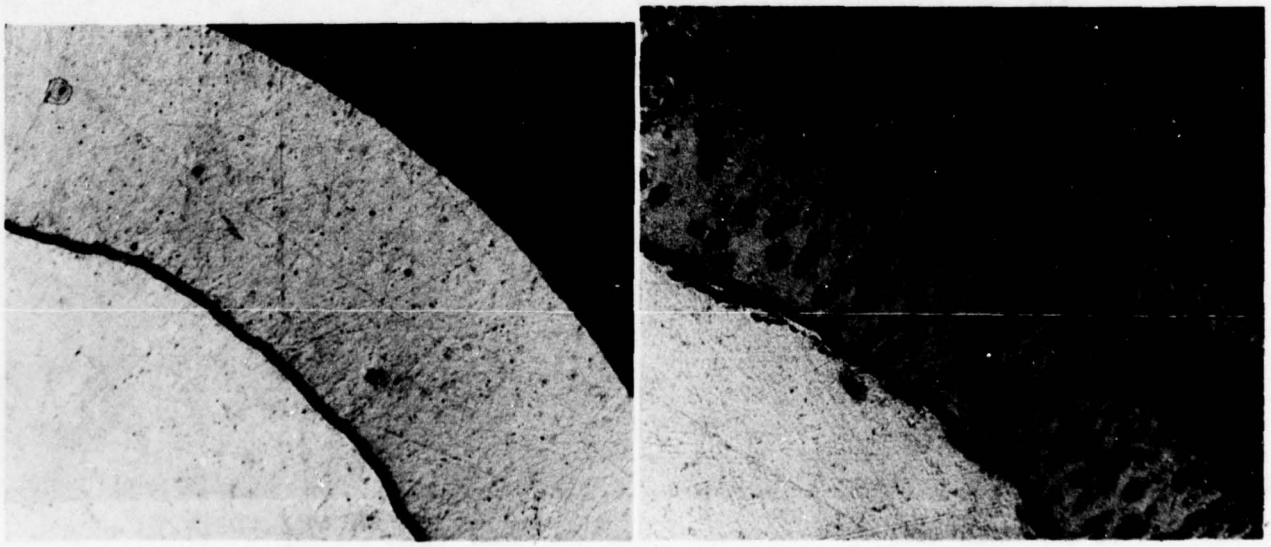
** Doubtful connection.



#12 AWG UNAGED 200X

#12 AWG 200X
AGED 1000hr @ 1250F

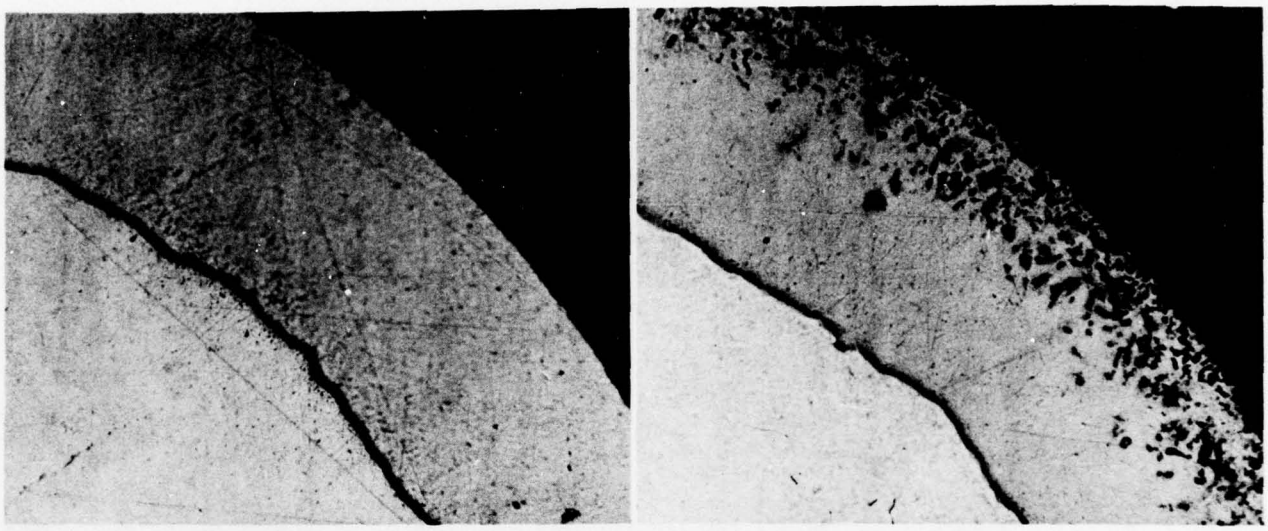
Figure 2. Metallographic sections of selected conductors.



#18 AWG UNAGED 300X

#18 AWG 300X
AGED 1000hr @ 1250F

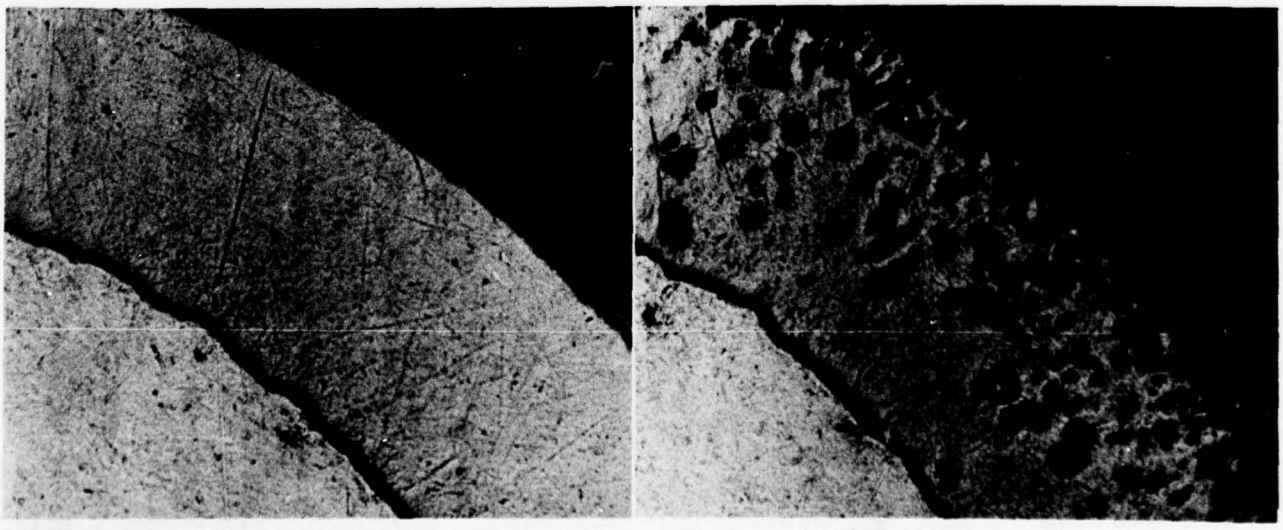
Figure 3. Metallographic sections of selected conductors.



#20 AWG UNAGED 400X

#20 AWG 400X
AGED 1000hr @ 1250F

Figure 4. Metallographic sections of selected conductors.



#22 AWG UNAGED 600X

#22 AWG 600X
AGED 1000hr @ 1250F

Figure 5. Metallographic sections of selected conductors.

In an effort to establish the cause of the damage, discussions were held with the test facility, the conductor subcontractor, and the manufacturers of the components used in the furnace assembly. It was finally established that the use of the castable ceramic support, (Sauereisen #33, Sauereisen Co. Ltd.), resulted in the evolution of volatile silicic acid based degradation products in excess of 1000°F, which in turn accelerated oxidative degradation of the sheath.

To verify these findings, two samples of each of 12 and 22 AWG conductors were exposed concurrently at 1250°F in covered platinum crucibles, one of each in the presence of Sauereisen #33. As can be seen from Figures 6 and 7, exposure of the conductor under normal conditions for 500 hours at 1250°F effected no noticeable deterioration, while exposure in the presence of the cast plug for only 235 hours resulted in significant damage. All subsequent experiments employed the original mica spacers.

It should be noted that, even in these excessively severe conditions, conductor resistivity was hardly affected, suggesting that a significant degree of corrosive damage could be tolerated with little loss of electrical performance.

As a result of these studies, it was concluded that the Inconel 600 sheathed silver core conductor was entirely satisfactory as a conductor for service at 1250°F, in all gauges from 12 AWG to 22 AWG inclusive.

2. Experimental Conductor

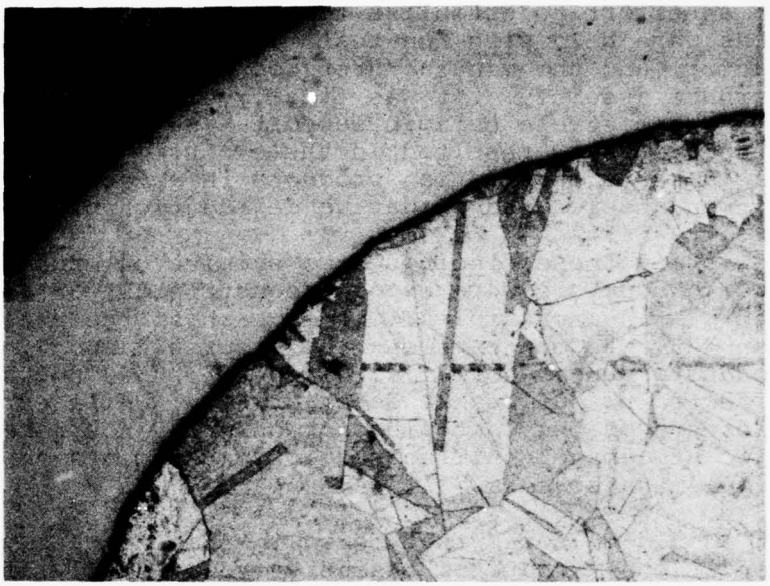
Although Phase I studies[1] established that the Inconel® 600 clad silver core conductor would fulfill the performance requirements of the program, it was recognized that this configuration was an excessively expensive (>$10/linear foot) solution. Various alternative conductor configurations were considered. One of the more promising was the development of a technique by a scientist at the University of Western Ontario, London, Ontario, to provide a protective and stable high temperature coating for copper by means of multiple layer solid state diffusion techniques.

A contract was awarded early in Phase II for further studies of this approach. Details of the effort may be found in Appendix A.

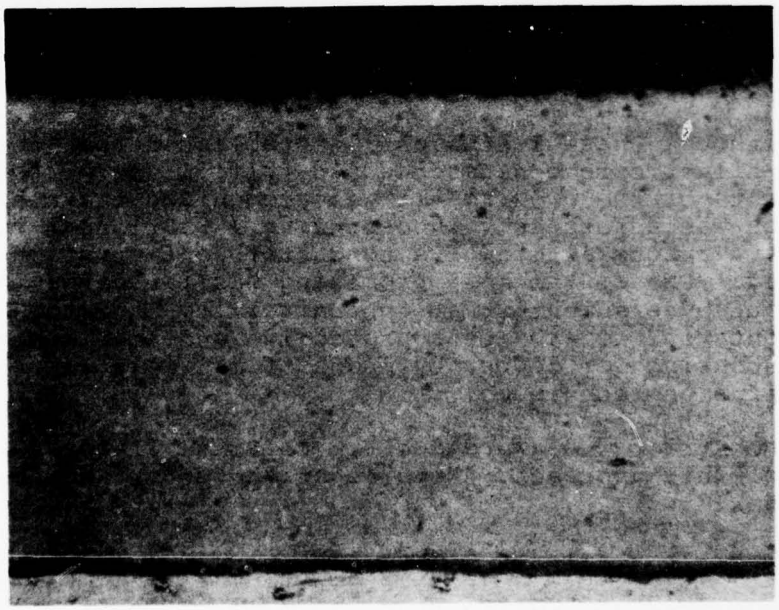
B Primary Insulation

1. Introduction

The preferred primary insulation developed in Phase I was established as reconstituted mica paper impregnated with poly(carborane siloxane)s, applied to the wire in tape form[1]. Phase II studies continued the optimization and characterization of this novel insulation.

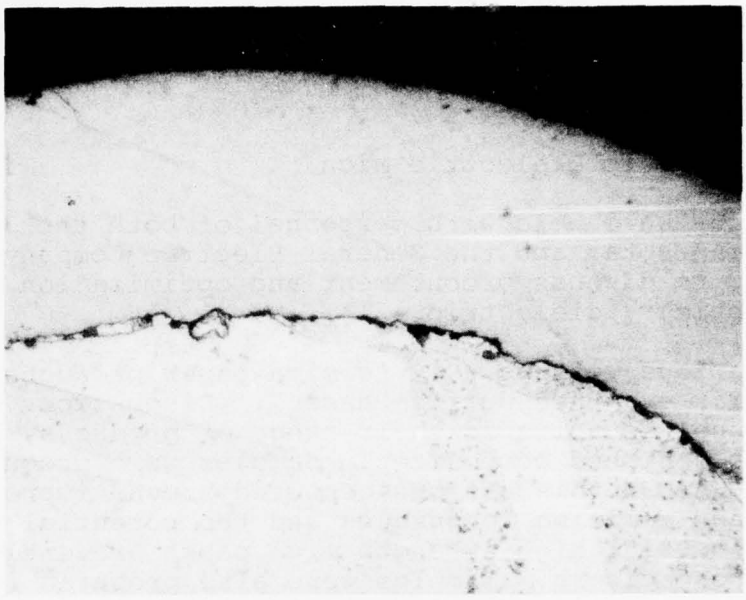


a) 12 AWG Inconel 600/Ag after 500 hours at 1250°F in air.

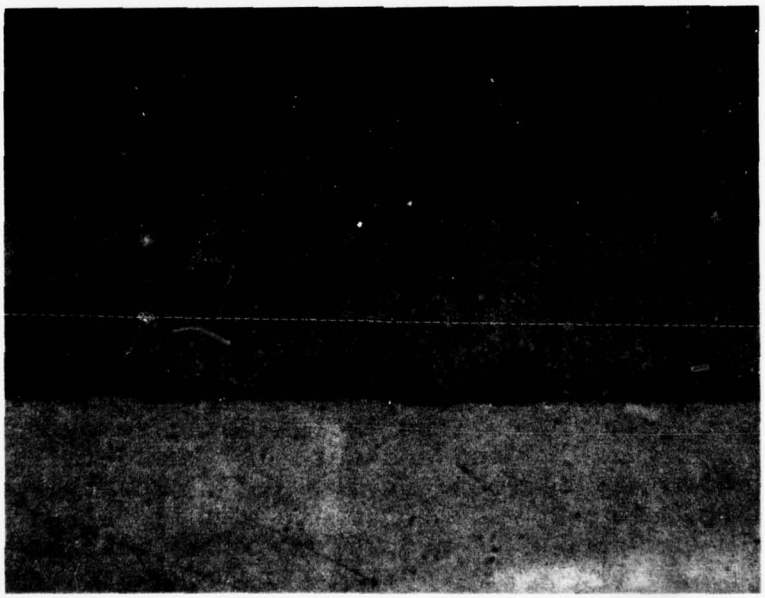


b) 12 AWG Inconel 600/Ag after 235 hours at 1250°F in air and the presence of Sauereisen #33.

Figure 6. Resistance of prime conductor to corrosion damage, #12 AWG.



a) 22 AWG Inconel 600/Ag after 500 hours at 1250°F in air.



b) 22 AWG Inconel 600/Ag after 235 hours at 1250°F in air and the presence of Sauereisen #33.

Figure 7. Resistance of conductor to corrosion damage, #22 AWG.

2. Primary Dielectric Mica

Meetings were held with personnel of both the 3M Company, St. Paul, Minnesota, and the General Electric Company, Schenectady, N.Y. to discuss procurement and optimization of the mica paper primary dielectric.

For various reasons, G.E.'s mica paper products were only cursorily examined during Phase I. Discussions with their personnel established their range of products, from raw paper to finished composites. Samples were procured for evaluation. At 3M, discussions centered around improved impregnating and splicing techniques and the potential value of ultrathin (<0.001") high strength mica paper presently used for winding capacitors. Samples were also procured for examination.

Referring to Table 6, 3M's 4200 grade is the paper which was used in the development of the prototype electrical insulation tapes in Phase I. The 4100 grades are described as high quality, high performance flexible papers primarily designed for capacitor winding([2]). The 0.002" thick paper was procured for direct performance comparisons with 4200; the 0.0009" thick paper offers potential for reduction of creasing damage during taping. The G.E. 77002 paper is a standard quality, equivalent to 3M 4200. G.E. 78472 is an experimental paper developed for use in an experimental "flame resistant" cable designed by Rome Cable([3]).

These papers were impregnated both with Dexsil®300 and preheataged Dexsil 300, made as previously described in the Phase I final report([1]). The impregnation technique was as previously described, viz. mica paper squares, prewashed and dried, were weighed, impregnated with a 30% xylene solution of the appropriate polymer (the excess being removed by scraping and padding), cured and tested. The details of the impregnation may be found in Table 7. For the purpose of this report, testing was limited to tensile strength and flexibility when wrapped as a 0.25" unbacked tape around a 12 AWG bare conductor. The tensile strengths are reported both as pounds per inch width and pounds per square inch. This latter is derived by the general formula:

$$\text{Tensile strength}_{\text{psi}} = \frac{\text{pounds/inch width} \times 1000}{\text{thickness in inches}}$$

The results may be found in Table 8.

TABLE 6

POTENTIAL CANDIDATE MICA PAPERS

Code	Commercial Identification	Supplier	Paper Thickness inches	Base weight gm/sq.ft.	Comments
M-A	4200	3M	0.002	7.52	relatively coarse flake, pale brown
M-B	4100	3M	0.002	7.82	very fine flake, lustrous
M-C	4100	3M	0.0009	3.27	very fine flake, translucent
M-D	77002	GE	0.0018	6.72	very fine flake, lustrous
M-E	78472	GE	0.002	8.43	coarse flake, lustrous, translucent

TABLE 7

IMPREGNATION AND CURE CONDITIONS FOR POLY(CARBORANE SILOXANE) IMPREGNATED MICA SHEETS

Sample Code	Material	Impregnant Solution Constituent Ratio % (w/w)		% Active Ingredient (w/w)	Cure Cycle Min./°C.		
		Active Ingredient	Carrier Solvent Xylene				
II-1	M-A	-					
II-6	D300/M-A	30.8	69.2	14.5	15/100	30/200	30/300
II-7	D300/M-A	30.8	69.2	13.8	15/100		30/300
II-8	PHA D300/M-A	36.5	63.5	16.5	15/100	30/200	30/300
II-9	PHA D300/M-A	36.5	63.5	17.4	15/100		30/300
II-2	M-B	-					
II-10	D300/M-B	30.8	69.2	11.0	15/100	30/200	30/300
II-11	D300/M-B	30.8	69.2	11.5	15/100		30/300
II-12	PHA D300/M-B	36.5	63.5	11.9	15/100	30/200	30/300
II-13	PHA D300/M-B	36.5	63.5	12.7	15/100		30/300
II-3	M-C	-					
II-14	D300/M-C	30	70	14.3	15/100	30/200	30/300
II-15	D300/M-C	30	70	13.4	15/100		30/300
II-16	PHA D300/M-C	30	70	14.7	15/100	30/200	30/300
II-17	PHA D300/M-C	30	70	13.7	15/100		30/300
II-4	M-D	-					
II-18	D300/M-D	30	70	15.7	15/100	30/200	30/300
II-19	D300/M-D	30	70	17.2	15/100		30/300
II-20	PHA D300/M-D	30	70	15.3	15/100	30/200	30/300
II-21	PHA D300/M-D	30	70	18.6	15/100		30/300
II-5	M-E	-					
II-22	D300/M-E	30	70	6.0	15/100	30/200	30/300
II-23	D300/M-E	30	70	6.0	15/100		30/300
II-24	PHA D300/M-E	30	70	5.8	15/100	30/200	30/300
II-25	PHA D300/M-E	30	70	6.3	15/100		30/300

TABLE 7 (continued)

IMPREGNATION AND CURE CONDITIONS FOR POLY(CARBORANE SILOXANE) IMPREGNATED MICA SHEETS

Sample Code	Material	Impregnant Solution Constituent Ratio % (w/w)		% Active Ingredient (w/w)	Cure Cycle Minutes/°C
		Active Ingredient	Carrier Solvent Xylene		
II-26	M-B/D300*	30	70	16.1	15/100, 30/200, 30/300
II-27	M-B/PHA D300	30	70	17.8	15/100, 30/200, 30/300
II-28	M-B/D300	30	70	17.2	15/100, 30/300
II-29	M-B/PHA D300	30	70	17.6	15/100, 30/300
II-30	M-C/D300	30	70	19.7	15/100, 30/200, 30/300
II-31	M-C/PHA D300	30	70	18.3	15/100, 30/200, 30/300
II-32	M-C/D300	30	70	16.9	15/100, 30/300
II-33	M-C/PHA D300	30	70	20.5	15/100, 30/300
II-34	M-D/D300	30	70	18.6	15/100, 30/200, 30/300
II-35	M-D/PHA D300	30	70	17.0	15/100, 30/200, 30/300
II-36	M-D/D300	30	70	17.2	15/100, 30/300
II-37	M-D/PHA D300	30	70	19.0	15/100, 30/300
II-38	M-B/D300	15	85	6.5	15/100, 30/200, 30/300
II-39	M-B/PHA D300	15	85	6.1	15/100, 30/200, 30/300
II-40	M-B/D300	15	85	6.8	15/100, 30/300
II-41	M-B/PHA D300	15	85	6.6	15/100, 30/300
II-42	M-C/D300	15	85	7.2	15/100, 30/200, 30/300
II-43	M-C/PHA D300	15	85	5.4	15/100, 30/200, 30/300
II-44	M-D/D300	15	85	5.7	15/100, 30/300
II-45	M-C/PHA D300	15	85	6.4	15/100, 30/300

* Where D300 refers to Dexsil 300 and PHA D300 refers to preheataged Dexsil 300, as described in the Phase I final report([1]).

TABLE 8

TENSILE STRENGTH AND FLEXIBILITY OF IMPREGNATED MICA SHEETS

Code	Tensile Strength Pounds/inch width (absolute psi)		Flexibility (0.25 inch tape diagonally wrapped around a bare 12 AWG Conductor)
II-1	~ 1.0	(~ 500)	heavy creasing, flaking and delamination
II-6	15	(7,500)	heavy creasing
II-7	16	(8,000)	medium creasing
II-8	8	(4,000)	medium creasing, slight flaking
II-9	8	(4,000)	medium creasing, slight flaking
II-2	~ 1.5	(~ 750)	heavy creasing, flaking and delamination
II-10	36	(18,000)	medium creasing
II-11	25	(12,500)	medium creasing
II-12	12	(6,000)	medium creasing
II-13	10	(5,000)	medium creasing
II-3	1.5	(1,700)	creasing, very little flaking, no delamination
II-14	7	(7,800)	very light creasing
II-15	8	(8,800)	very light creasing
II-16	8	(8,800)	very light creasing
II-17	8	(8,800)	very light creasing
II-4	~ .5	(250)	heavy creasing, flaking and delamination
II-18	8	(4,000)	medium creasing, very slight flaking
II-19	17	(8,500)	light-medium creasing
II-20	8	(4,000)	light-medium creasing
II-21	13	(6,500)	light-medium creasing
II-5	1	(500)	medium creasing, heavy flaking, some delamination
II-22	24	(12,000)	medium-heavy creasing, slight flaking
II-23	22	(11,000)	medium-heavy creasing, slight flaking
II-24	22	(11,000)	medium-heavy creasing, slight flaking
II-25	6	(3,000)	medium-heavy creasing, slight flaking

In general, 0.002" 4100 grade capacitor paper offered significant performance improvements over the equivalent thickness 4200 grade. The G.E. 77002 paper, although somewhat more absorptive, showed equivalent tensiles and superior flexibility. The relatively large flake size and low penetration rate of G.E. 78472 resulted in excellent tensiles, but at the expense of flexibility. The 0.0009" thick 4100 grade capacitor paper from 3M showed an excellent combination of impregnant pickup, tensile strength and high flexibility. It was decided to substitute the 4100 grade of mica paper from 3M for 0.002" 4200 quality in all future development. Further experiments combined the evaluation of selected papers with studies to optimize impregnation techniques and loadings.

3. Primary Impregnant

The supplier for the poly(carborane siloxane) impregnant, Dexsil 300, during Phase I was Olin Mathieson, through their exclusive outlet, Analabs Inc. of Connecticut. Upon receiving approval to proceed with Phase II, Analabs was contacted to discuss the procurement of the anticipated volume of Dexsil 300 required for preparation of pilot quantities of primary dielectric tape. It was found, however, that Olin and Analabs were involved in protracted negotiations for the sale of the technology. This situation took some months to resolve; the negotiations between the two companies eventually collapsed and it was indicated that no further supplies of Dexsil 300 or any other of the Dexsil series would be available in the anticipated quantities ($\sim$400 grams) required for pilot impregnation studies.

During this period, a re-evaluation of the results of the cursory experiments to evaluate alternate poly(carborane siloxane)s during Phase I([1]) was made. It was hoped that one of the two other suppliers, Union Carbide Corporation, and Chemical Systems Inc., would have materials which offered an acceptable combination of properties for use as a Dexsil 300 substitute. It was decided that the Union Carbide D_2 (subsequently renamed UCARSIL®) polymers showed most promise.

Initial conversations with Union Carbide personnel stimulated great interest on their part and a visit was made to the Tarrytown, N.Y. Technical Center of Union Carbide Corporation to discuss the chemistry, uses and long-range marketing plans of their range of UCARSIL poly(carborane-siloxanes). (These polymers are being considered as potential alternatives to the Phase I primary mica Impregnant Dexsil 300, as, due to the recent sale of Dexsil materials and technology from Olin Mathieson to Analabs Inc., there is some doubt about the latter material's long-term availability).

As it was considered by Union Carbide that alternate methods of synthesis (i.e. Dexsil synthesis) resulted in polymer having excessive cross-linking([4,5]) which seriously affected fabrication and vulcanization, a new route was developed which resulted in a relatively simple chemical reaction yielding high molecular weight linear polymers in which structural modification can be readily achieved as required for vulcanization and specific property optimization. The basic equation for the synthesis is shown in Figure 8.

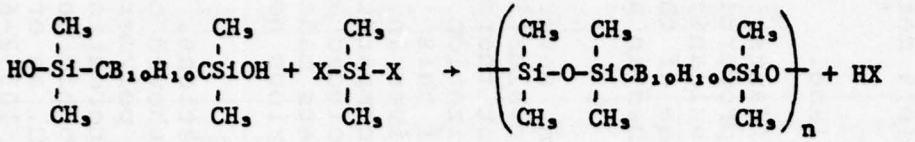
The preferred polymer for general elastomeric applications is a poly(carborane dimethylsiloxane) in which 33 mole % and 1 mole % of methyl phenyl siloxane and methyl vinyl siloxane, respectively, are substituted (Figure 9).

In-depth discussions elucidated the curing, cross-linking and thermal transformation mechanisms of the UCARSILS. As has been outlined in the many papers published describing poly-(carborane siloxane) chemistry([6]), derivation of the elastomeric behaviour of these polymers occurs through peroxide vulcanization of the vinylic unsaturation. Subsequent thermal cross-linking occurs primarily through abstraction of hydrogen from the methyl groups, resulting in bridging. The presence of methyl group stabilizing moieties on the side chains (i.e. phenyl groups), influences the thermal resistance to cross-linking. As well as effecting elastomeric properties, the presence of vinylic unsaturation might be used to improve the bond between the polymer and the mica substrate, through the use of commercial siloxane-based vinyl containing coupling agents.

An alternative route to bonding at the polymer/mica interface is through the use of UCARSIL polymer containing a silyl hydride reactive moiety which will potentially react directly with the hydroxyls available in the mica crystal lattice (Figure 9). The mechanism of cross-linking is postulated to be as follows:

$$\sim\!\!\sim Si\text{-}OH + H\text{-}\underset{\displaystyle CH_3}{\overset{|}{Si}}\text{-} \rightarrow \sim\!\!\sim Si\text{-}O\text{-}\underset{\displaystyle CH_3}{\overset{|}{Si}}\text{-} + 2H^+$$

It was agreed that Union Carbide would forward six samples of UCARSIL with relevant TGA and DTA data, having various structural variants, to allow evaluation as potential alternative impregnants for the primary dielectric mica tape. Referring to Figure 8, the structural variants are:



where X = $-N(CH_3)_2$, $-O-\overset{O}{\overset{\|}{C}}-NR_2$, $-N(Ph)-\overset{O}{\overset{\|}{C}}-N(CH_3)_2$ OR $-N(Ph)-\overset{O}{\overset{\|}{C}}-N$(cyclic)

FIGURE 8

POLYMERIZATION PROCESS FOR THE SYNTHESIS OF UCARSIL® POLY(CARBORANE SILOXANES)

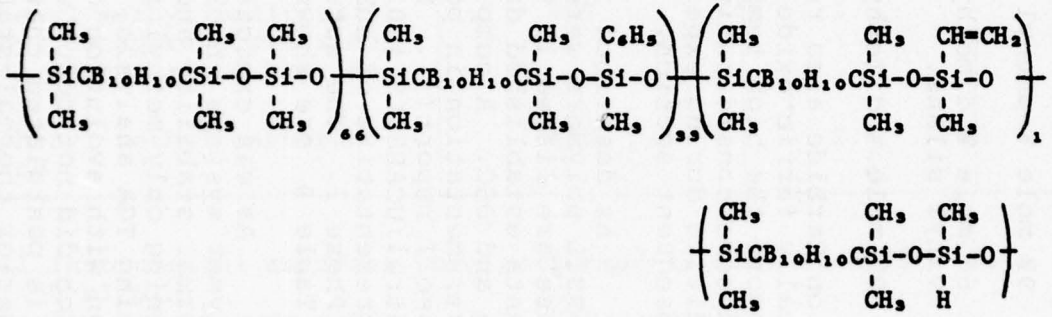


FIGURE 9

SCHEMATIC STRUCTURE OF PREFERRED UCARSIL® ELASTOMERIC BASE STOCK AND THE SILYL HYDRIDE VARIANT

1. 66 mole % dimethyl, 33 mole % phenyl methyl, 1 mole % methyl silyl hydride.

2. 66 mole % dimethyl, 33 mole % phenyl methyl, 1 mole % methyl vinyl silane.

3. 100 mole % phenyl methyl.

4. 99 mole % phenyl methyl, 1 mole % methyl vinyl silane.

5. 66 mole % dimethyl, 33 mole % diphenyl, 1 mole % methyl vinyl silane.

6. 99 mole % dimethyl, 1 mole % methyl vinyl silane.

Union Carbide also recommended the use of antioxidants, specifically ferric oxide, to improve the mechanical properties and promote the high temperature stability of their vulcanizates, and some consideration was given to the advantages of this additive during experiments which will be described in a subsequent section.

As described in detail in the next section, several UCARSIL polymers were subsequently received for evaluation; these are listed in Table 9 , as are two pertinent thermal events established during instrumental characterization using TGA and DSC. A complete discussion of the use of this instrumentation in polymer characterization is given in the Phase I report([1]). Dexsil 300 is included as a comparative control;UCARSILS #10352-61 and #10352-40 were included as representative of submissions of prototype polymers made late in Phase I. The actual TGA/DSC scans for the various polymers of Table 9 are shown in Figures 10-16.

As was expected from theoretical considerations, those polymer systems which contained phenyl moieties showed better thermal stability and lower weight loss than the polymers containing only methyl side chain substitution. Interestingly, during TGA analysis, Dexsil 300 showed a catastrophic degradation with evolution of silica fume at about 550°C, an effect which did not occur with the UCARSIL equivalent, 10352-61. It is postulated that this effect is confirmation of the superior thermal stability of the 10SiB2 configuration of the UCARSILS.

Visually, it was noted that, as the level of phenolic substitution increased, the postfired samples became darker until, at 100% substitution, the surface was black and covered with a brittle "glass". Evidence of this deterioration was also seen in the degradation of both handling and dielectrics after 1250°F exposure.

TABLE 9

COMPARATIVE ANALYSIS OF UCARSIL POLYMERS SUBMITTED

AS POTENTIAL CANDIDATE PRIMARY IMPREGNANTS

Code	Molecular Configuration	Peak of Oxidative Rearrangement, °C*	% Weight Loss at 650°C*	Internal Experimental Code Numbers**
Dexsil 300	100% dimethyl decaborane siloxane	390	15.0	C1-2
10352-61	100% dimethyl decaborane siloxane	390	5.0	C3-4
10352-40	x% phenyl methyl modified dimethyl decaborane siloxane	470	2.5	C5-6
10352-127	100% diphenyl decaborane siloxane containing 4% methyl vinyl unsaturation	500	3.5	C7-8
10615-6	33% phenyl methyl modified dimethyl decaborane siloxane with 4% methyl vinyl unsaturation	470	2.5	C9-10, C15-16
10615-22	33% phenyl methyl modified dimethyl decaborane siloxane with 4% methyl silyl hydride reactivity	465	3.0	C11-12
10615-50	100% phenyl methyl carborane siloxane	485	2.5	C13-14

* All instrumental analyses were performed in air, at a flow rate of 50 ml/min.
Program temperature rate of rise 10°C/minute.

** Code numbers used in text and Table II.

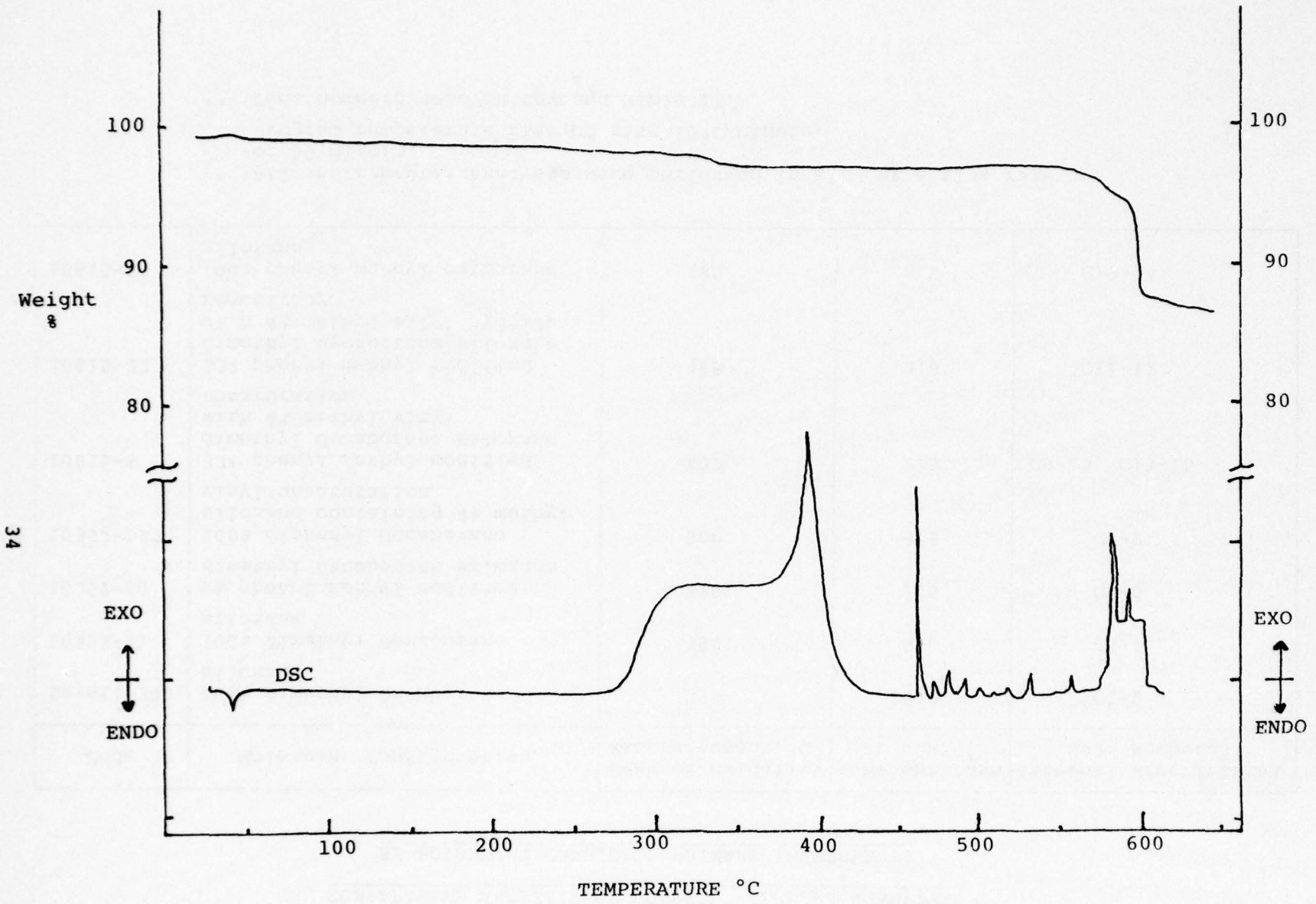


Figure 10. Thermal stability of Dexsil 300.

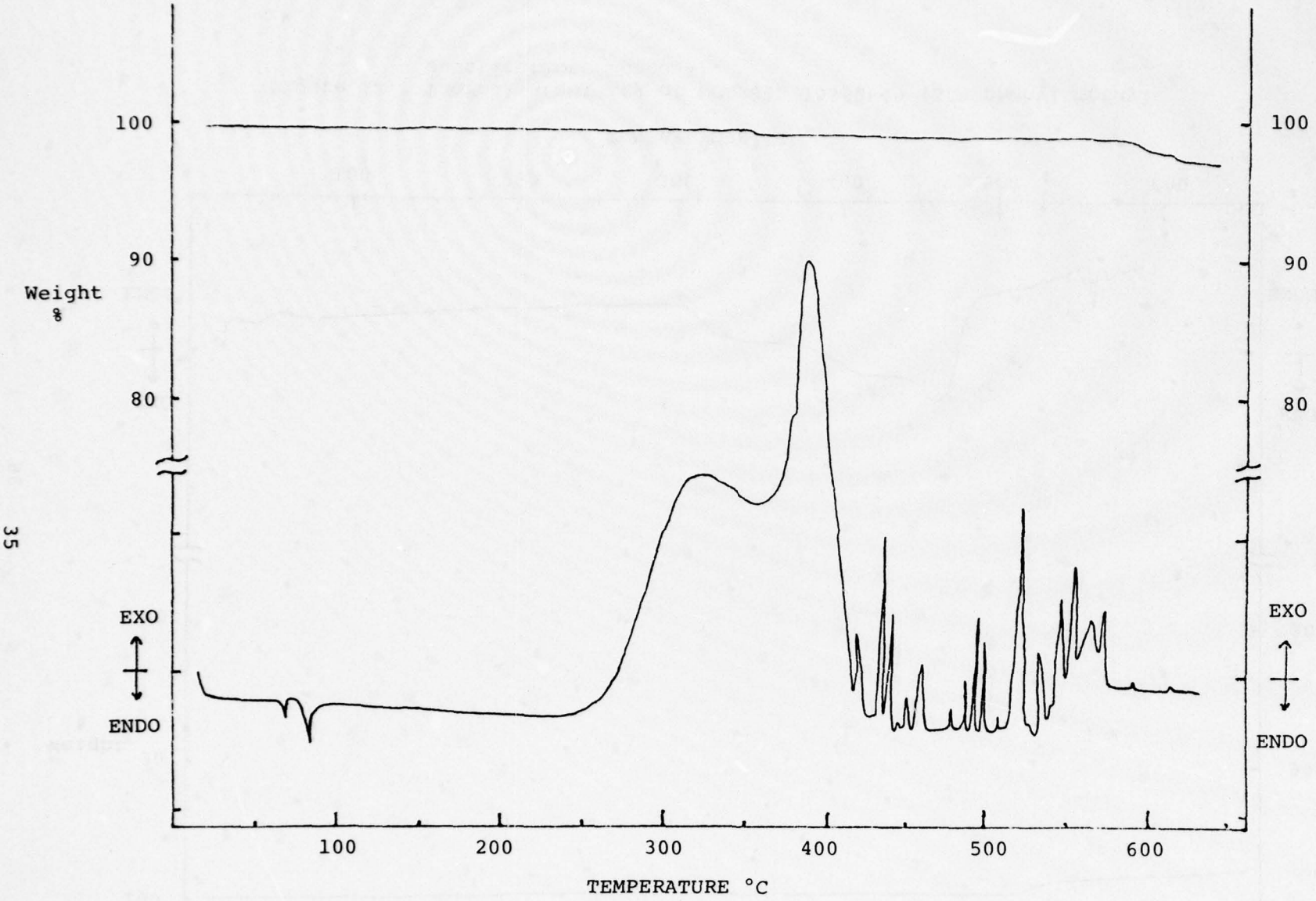


Figure 11. Thermal stability of UCARSIL 10352-61 (all methyl substitution, Phase I).

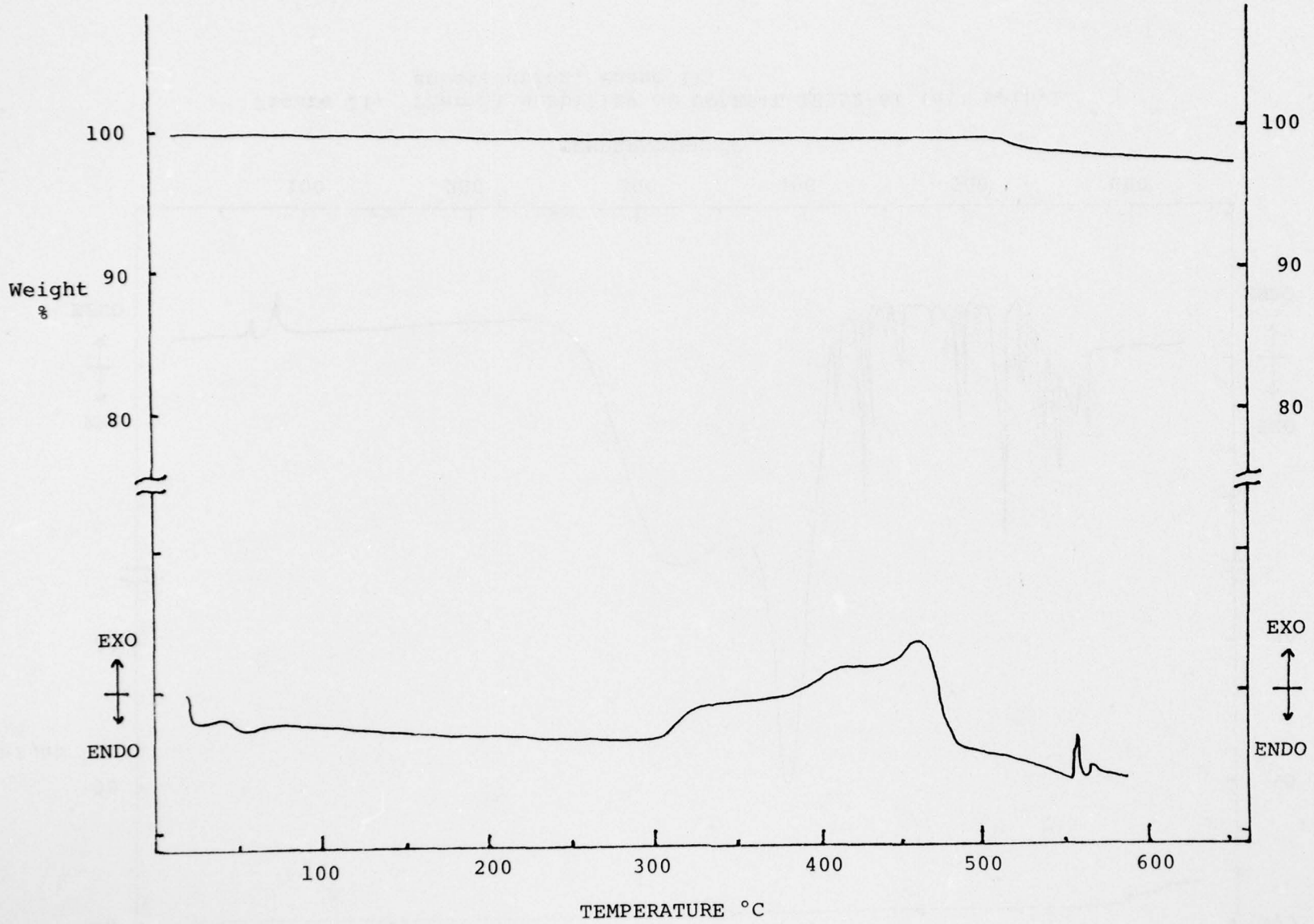


Figure 12. Thermal stability of UCARSIL 10352-40 (33% phenyl methyl substitution, Phase I).

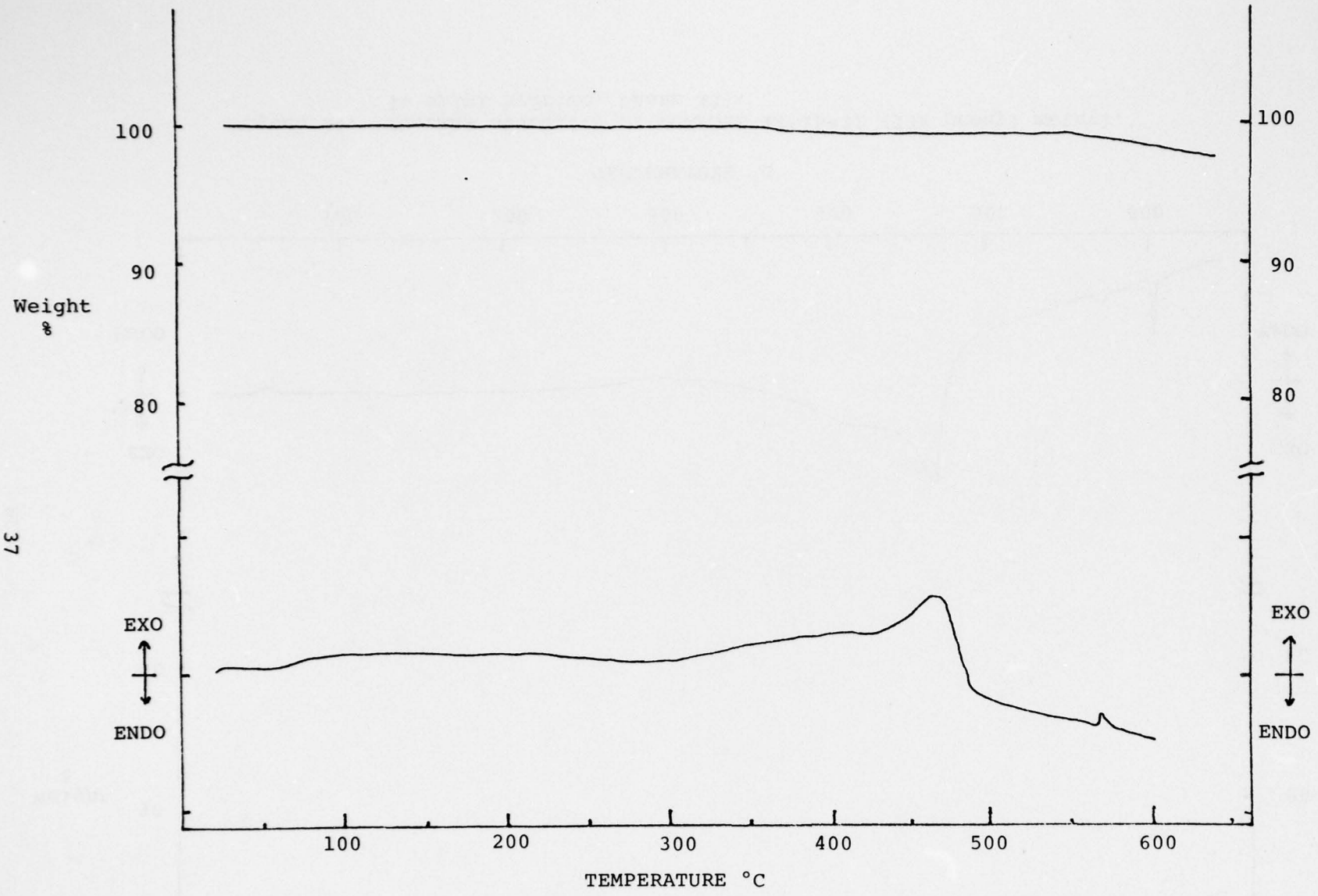


Figure 13. Thermal stability of UCARSIL 10615-6 (33% phenyl methyl, 4% methyl vinyl, Phase II).

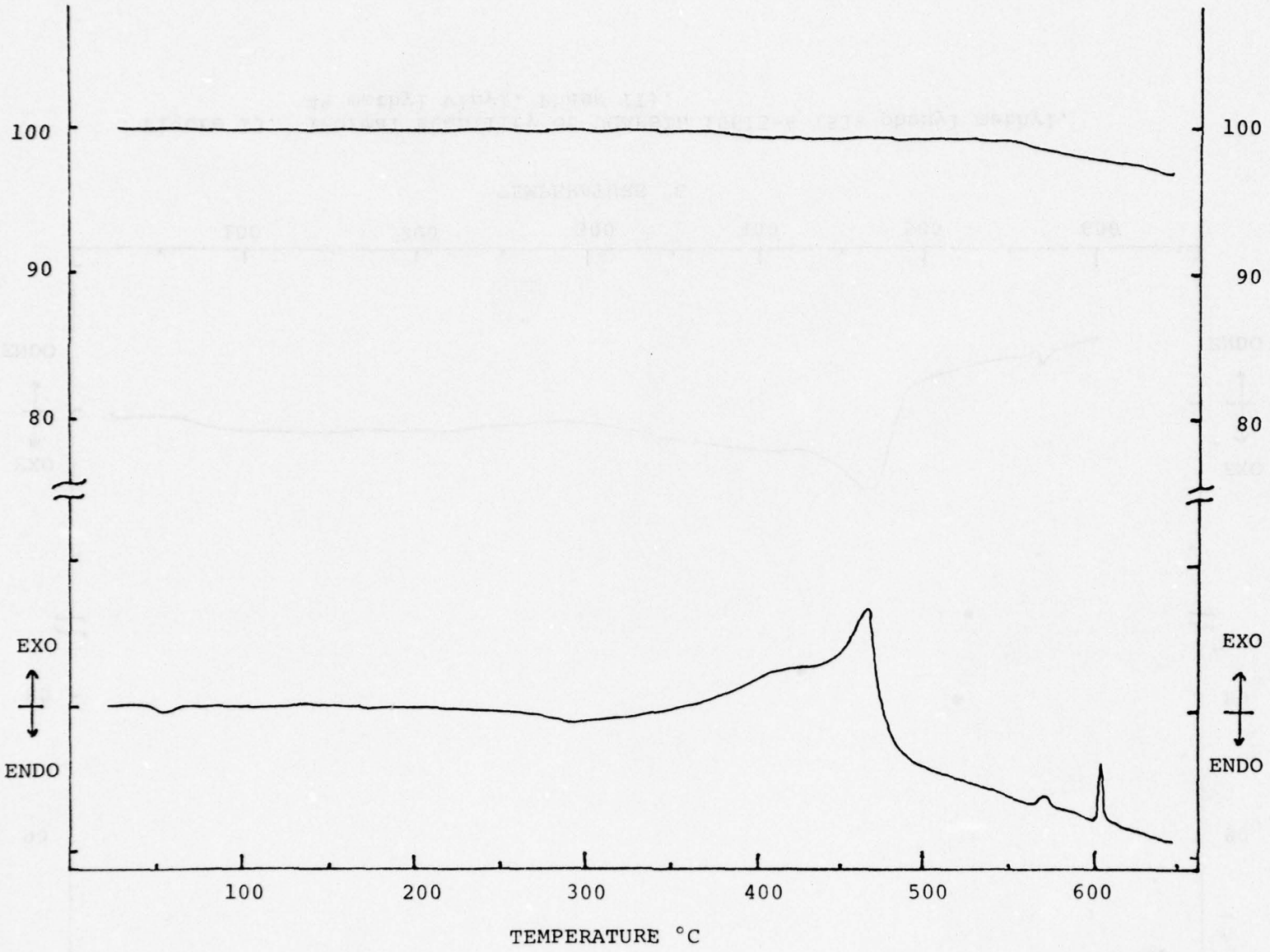


Figure 14. Thermal stability of UCARSIL 10615-22 (33% phenyl methyl, 4% silyl hydride, Phase II).

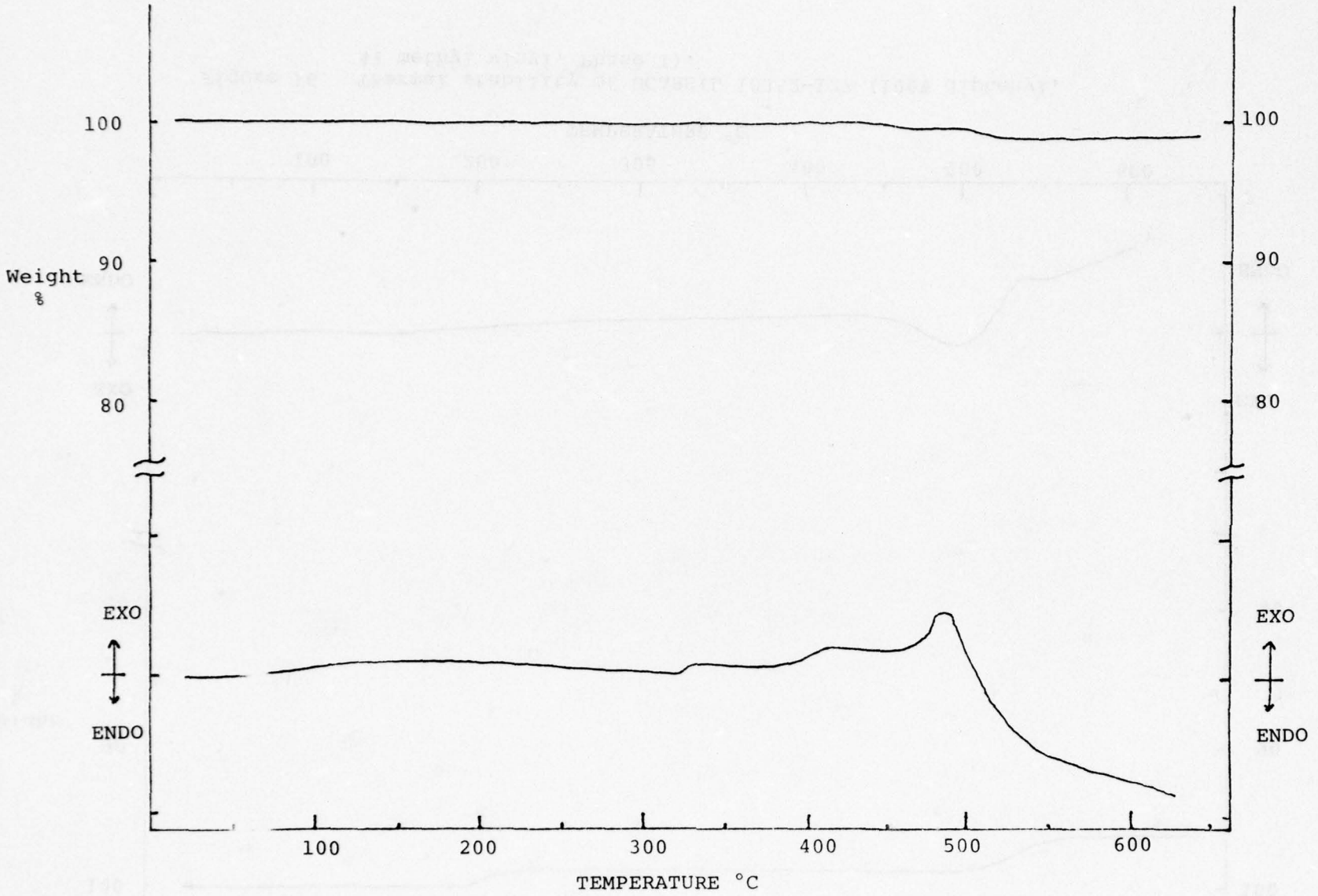


Figure 15. Thermal stability of UCARSIL 10615-50 (100% phenyl methyl, Phase II).

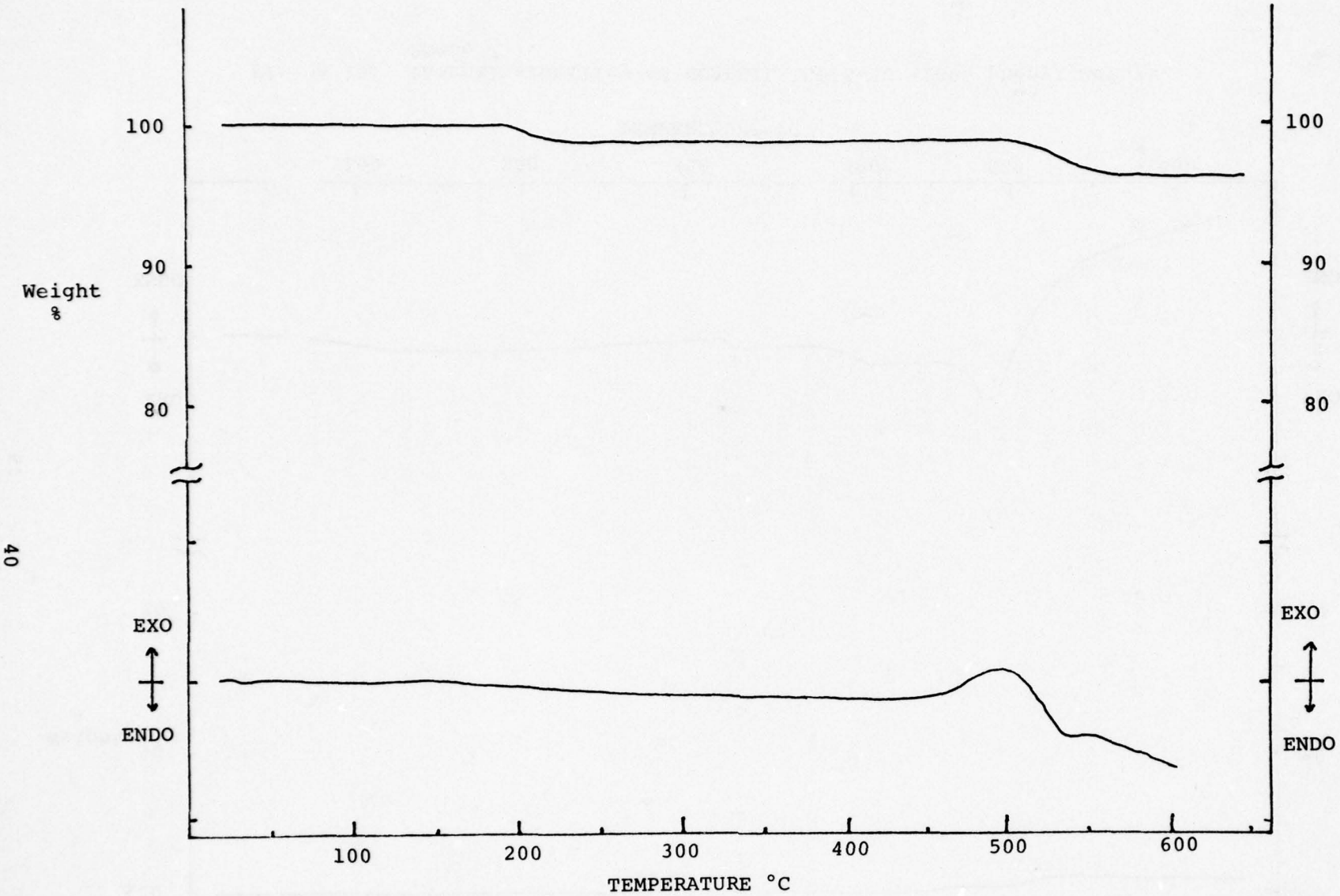


Figure 16. Thermal stability of UCARSIL 10352-127 (100% diphenyl, 4% methyl vinyl, Phase I).

In summary, selected UCARSILS were found to be satisfactory substitutes for Dexsil 300 as the impregnant in the primary dielectric mica paper.

Based on a further series of experiments, two polymers, #10615-6 and #10615-22, were chosen as candidates for use as primary impregnants. Both of these polymers are 33% phenyl methyl modified dimethyl decaborane siloxanes, with 4% methyl vinyl at 4% methyl silyl hydride reactivity respectively.

At the recommendation of Union Carbide, a verbal order for 300-500 grams of polymer was placed,to expedite delivery. A letter of intent to purchase was also forwarded, on request, to allow them to respond with a price quotation and a prediction of materials volume availability. Subsequent conversations with Union Carbide indicated that, due to precursor availability, only ∿300 grams of either of the two preferred polymers could be synthesized, but a greater volume of a mixture could be supplied if acceptable. As will be described in more detail in the next section, a 75:25 w/w blend of 10615-22/10615-6 was acceptable and an order was placed. A blended sample of 415 grams of polymer was finally received; the thermal stability of the blend as determined by DSC/TGA is illustrated in Figure 16A. Further evaluation is reported in the next section.

4. Optimization of the Composite Mica Primary Dielectric Tape

a. Evaluation of Various Impregnation Techniques

Due to the excessive cost of Dexsil 300 (∿$15/gram), laboratory evaluation of its efficacy as an impregnant during Phase I was limited to 4" x 4" squares of paper which were impregnated by using an eye dropper to apply a polymer solution, spreading with an applicator to achieve visual wetting and padding off the excess with a soft tissue. It was recognized at the time that this technique did not mirror industrial technique, however, it did offer good reproducibility and was fast.

A series of experiments was begun at the beginning of Phase II to study various impregnation techniques as part of the primary dielectric optimization function.

The initial series II-1 to II-25 were impregnated using the Phase I technique described above, and combined optimization of impregnant loading with polymer type, using both Dexsil 300 and preheataged Dexsil 300(1), and a further evaluation of the various mica samples described in Section 2. A description of the mica papers can be found in Table 6. The impregnation and cure conditions may be found in Table 7, with an initial evaluation of mechanical performance in Table 8.

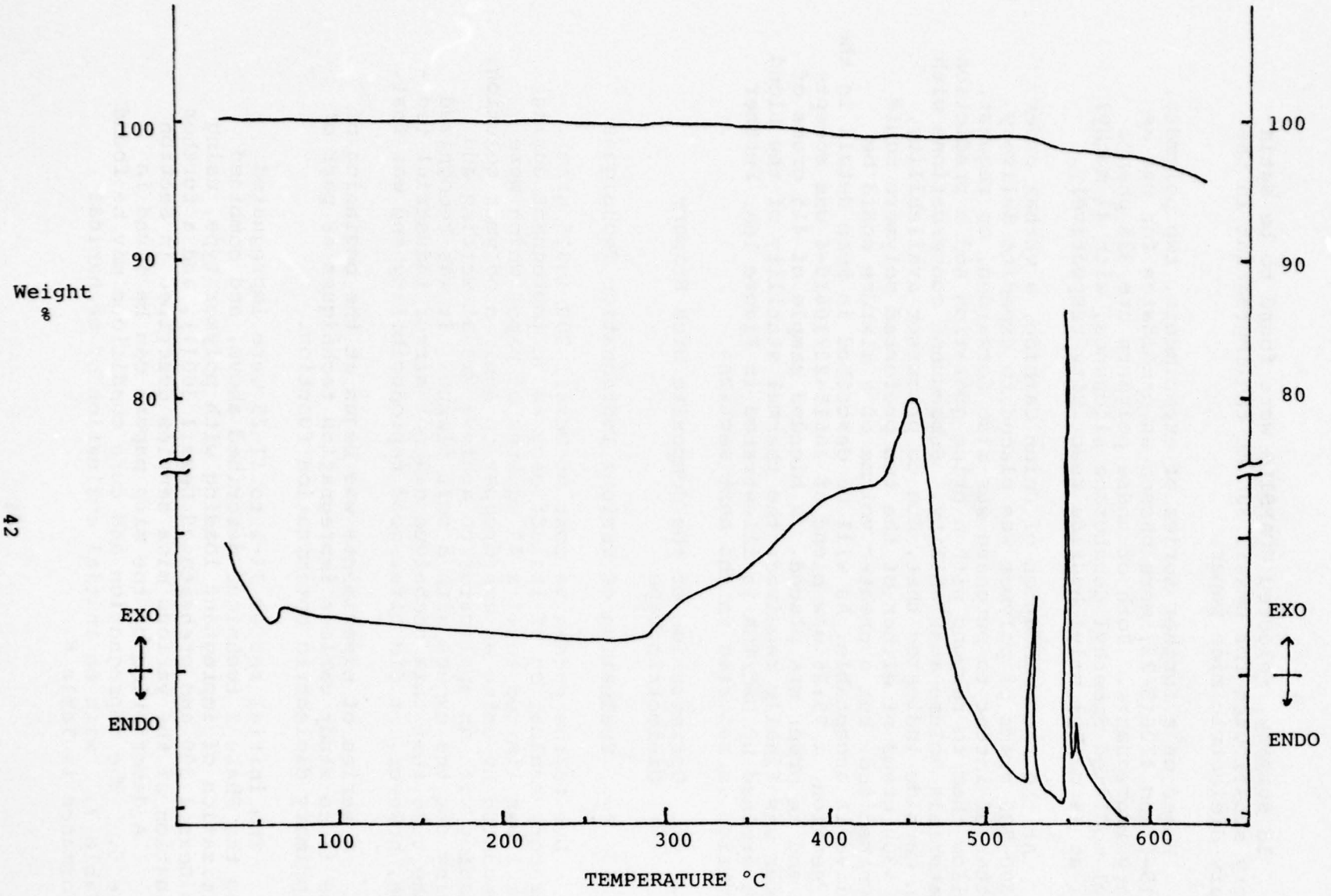


Figure 16. Thermal stability of UCARSIL blend in air.

Series II-26 to II-45, Table 7, were dip coated by immersing the mica sheets in solutions of the appropriate polymer for 1 minute, draining and padding off the excess with a soft tissue. Series II-26 to II-37 were dip coated using the standard 30% solids w/w solutions of either Dexsil 300 or preheataged Dexsil 300, as noted, and cured for either the Phase I standard three-step cure or the alternative two-step cure which was evolved late in Phase I. Initial results indicated that the method of impregnation was resulting in weight pickups in excess of that level considered optimum (∿12%), so a second series, (II-38 to II-45), was made to evaluate the effect of reducing the impregnant level to 15% solids w/w. As noted in Table 7, there was a significant drop in the percent active ingredient retained on the two mica papers reported, although in general, reproducibility of weight pickup was improved. A rough plot indicated that a solution of 23% solids content would yield pickups of approximately 12%.

These samples, and Samples II-1 to II-25, were evaluated for physical characteristics as outlined in the Phase I report (Appendix A, subsection 3)([1]). The results of these tests may be found in Table 10.

Considering the results of Table 10, it should be noted that both capacitor papers from 3M, II-2 and II-3, showed excellent tensile strength in the raw state. It is especially interesting that the 0.009" paper, (II-3), showed a significant increase in tensile strength after firing. This effect will be studied further.

Comparing the various samples, it was noted that the Samica®4200, 0.002" paper, (M-A), showed little improvement in performance when impregnated with preheataged Dexsil 300 compared to the regular material when cured in three steps, and had inferior tensile strength when impregnated with preheataged Dexsil 300 and cured in two steps.

Samica 4100, 0.002" thick, showed comparable handling characteristics and comparable tensile strengths to 4200 before 1250°F exposure, but significantly improved tensile strengths after exposure.

Samica 4100, 0.0009" thick, had slightly inferior handling characteristics to the 0.002" thick materials, but showed an excellent combination of tensile strengths for such a thin paper. G.E. 77002, 0.002" thick paper had handling characteristics slightly inferior to those of the 3M materials; although the pre- and post-fired tensiles were equal to or better than those of the 3M materials when using Dexsil 300 impregnant, they were generally poorer using preheataged

TABLE 10

PERFORMANCE EVALUATION OF POLY(CARBORANE-SILOXANE) IMPREGNATED MICA SHEETS

Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)		Comments
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	Before	After	Before	After	
II-1	-	0.05	0	-1.07	2	3	3	3	3	4	4	4	1500	1600	-	2.6	3D
II-2	-	0.10	0	-0.93	2	3	3	4	2	3	4	3	4500	2600	4.2	3.9	1D, 3D
II-3	-	0.05	0.17	-1.03	1	1	4	4	2	3	4	3	1600	4100	2.2	1.5	
II-4	-	0.06	0.12	-1.20	3	4	4	4	4	4	4	3	1500	1900	2.5	2.3	1E
II-5	-	0	0	-0.71	2	4	4	3	3	4	4	3	2800	2900	3.1	2.6	1C, 1D, 3D
II-6	14.5	0.05	2.4	-0.09	1	1	1	2	2	3	1	2	7800	10,800	3.8	4.5	
II-7	13.8	0.04	1.7	-0.22	1	2	1	2	1	3	1	2	7200	11,800	3.9	4.7	
II-8	16.5	0.10	3.4	+0.10	1	1	1	1	2	3	1	2	6100	12,300	3.7	4.6	3B
II-9	17.4	0.10	3.8	+0.48	1	1	2	2	3	4	1	2	4100	8800	4.7	4.7	
II-10	11.0	0	0.58	-0.05	1	1	2	1	1	4	2	3	5600	6400	4.8	5.6	
II-11	11.5	0	1.10	0	1	1	1	1	2	4	1	2	7700	11,600	5.1	5.6	
II-12	11.9	0	1.90	+0.34	1	2	1	2	2	4	1	2	5900	15,700	4.8	5.7	3D
II-13	12.7	0	1.91	+0.43	1	1	1	2	3	4	1	2	5400	13,900	4.7	5.0	
II-14	14.3				1	1	2	2					5800	6100	3.4		

TABLE 10 (CONTINUED)

Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)		Comments
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	Before	After	Before	After	
II-15	13.4				1	2	1	3					5600	7700	2.6		
II-16	14.7				1	1	2	2					6600	9000	2.0		
II-17	13.7				1	1	2	2					6900	9900	2.4		
II-18	15.7	0	0.71	-0.31	1	3	2	2	4	4	1	2	11,400	12,600	3.5	2.9	3C
II-19	17.2	0	2.16	-0.72	1	3	1	2	4	4	1	3	7700	11,700	3.4	3.4	3C
II-20	15.3	0	3.74	+0.86					4	4	1	3	3300	11,800	3.8	4.2	3C
II-21	18.6	0.06	5.88	+1.62					4	4	1	2	4600	8800	3.3	3.6	
II-22	6.0	0.05	0.76	-0.25	1	1	1	1	1	1	1	2	11,400	12,300	4.8	4.0	3D, 1S
II-23	6.0	0.05	0.74	-0.21	1	1	1	1	1	1	1	1	10,800	11,000	4.4	3.8	
II-24	5.8	0.05	0.87	-0.19	1	1	1	2	1	1	1	2	5500	9500	4.0	4.4	
II-25	6.3	0.05	0.98	-0.15	1	1	2	2	2	1	1	1	8800	8500	5.0	4.4	1D, 3D
II-26	16.1	0.08	4.62	+0.69	1	1	2	1	3	3	2	2	4800	11,500	6.0	5.1	3P
II-27	17.8	0.08	4.90	+1.30	1	2	1	2	2	4	1	2	4100	7000	5.4	4.7	3P
II-28	17.2	0.10	5.08	+0.86	1	1	2	2	3	3	1	2	5900	9700	6.1	5.8	3P

TABLE 10 (CONTINUED)

Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)		Comments
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	Before	After	Before	After	
II-29	17.6	0.05	4.83	+1.11	1	2	1	2	3	4	1	2	4800	9900	5.3	5.0	3P
II-30	19.7	0.09	6.7	+1.45	1	2	3	3	1	4	1	2	4200	6100	3.8	2.6	
II-31	18.3	0.13	7.0	+1.92	1	2	3	3	3	4	1	3	4500	8300	4.4	2.9	
II-32	16.9	0.17	6.6	+1.36	1	1	2	2	3	4	1	2	5500	7600	3.6	2.6	
II-33	20.5	0.15	7.6	+2.4	1	2	2	2	2	4	2	2	4300	2900	3.9	2.5	
II-34	18.6	0.13	3.1	-0.35	1	2	1	2	4	4	1	2	6900	12,000	3.5	3.7	3A
II-35	17.0	0.14	3.5	+0.81	1	1	2	2	4	4	1	2	3700	12,000	3.7	3.8	3A
II-36	17.2	0.06	2.58	-0.53	1	2	2	2	4	4	1	3	7900	11,000	3.3	3.7	3A
II-37	19.0	0.10	4.8	+1.20	1	3	2	3	4	4	1	3	2200	8000	2.8	3.7	3A
II-38	6.5	0.07	0.79	0	1	1	2	2	1	3	1	3	3100	9900	4.0	5.0	3D
II-39	6.1	0.05	0.43	-0.33	1	1	2	3	2	3	1	2	3700	7600	4.1	4.5	3D
II-40	6.8	0.05	0.45	-0.40	1	1	2	3	2	3	1	2	3800	6500	4.9	5.1	3D
II-41	6.6	0.05	0.46	-0.15	1	1	2	2	3	2	1	3	4300	9100	4.0	4.9	3D
II-42	7.2	0.44	1.31	-0.22	1	1	2	3	3	3	1	3	3800	8800	2.3	2.4	

TABLE 10 (CONTINUED)

Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)		Comments
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	Before	After	Before	After	
II-43	5.4	0	0.29	-0.58	1	1	2	3	2	4	2	3	2900	10,000	2.0	2.7	
II-44	5.7	0.10	0.51	+0.10	1	1	2	3	2	3	2	2	3300	6700	2.2	2.4	
II-45	6.4	0.20	0.30	-0.30	1	1	2	2	3	4	2	3	3900	8900	2.3	2.2	

Dexsil 300. The 78472 paper from G.E. showed generally better handling characteristics than the other papers, as would be expected from the larger flake size. The tensile strengths both before and after firing were excellent, however, they showed little improvement after firing, as has been seen with other samples. Impregnating with preheataged Dexsil 300 caused a significant deterioration in tensile strength.

Comparing the three approaches to impregnating Samica 4100, 0.002" thick paper, (M-B), with Dexsil 300 (II-10, 26, 38), dipping resulted in a slight improvement in handling characteristics, slightly poorer pre-fired tensiles and improved post-fired tensiles. This latter is especially interesting when it is noted that even at 6.5% impregnant pickup, (II-38), the post-fired tensile strength was 50% greater than that of the Phase I technique loaded at 11%, (II-10). Considering the samples made with Dexsil 300 and cured using the two-step technique, (II-11, 28, 40), the handling characteristics were marginally superior to those samples cured in three steps. Pre- and post-fired tensile strengths on the blade applicated sample were superior to that of the three-step cured sample; while pre-fired tensiles on the dipped materials were superior, post-fired values were inferior.

Comparing the samples made with preheataged Dexsil 300 to those with Dexsil 300, (II-12, 27, 39), handling was slightly inferior while pre-fired tensiles were generally comparable. The post-fired tensile strength of the blade applicated sample was significantly superior, while those of the dipped samples were poorer. Examining the performance of the mica sheets impregnated with preheataged Dexsil 300 and cured in two steps rather than three (II-13, 29, 41), handling characteristics were slightly inferior. The tensile strengths of the blade applicated samples were slightly poorer before firing and slightly superior after 1250°F exposure. The pre-fired tensiles of the dipped samples were comparable to slightly inferior while post-fired tensiles were slightly superior.

Evaluating the Samica 4100, 0.0009" impregnated papers made with Dexsil 300 and cured with either the three-step (II-14, 30, 42), or two-step technique, (II-15, 36, 44), pre-fired handling characteristics were comparable overall while there was no positive comparison possible of post-fired handling performance. Pre-fired tensile strengths were generally comparable with the exception of the 30% solids dipped sample having a two-step cure which was significantly superior. Comparing the post-fired values, those from the samples having a three-step cure were generally very good, and superior to the values found for the two-step cure samples, again with the exception of the single sample (II-36) which showed significantly higher performance. It should be noted that the performance of this set of samples was only slightly inferior,

in general, to the performance previously reported for the 4100 0.002" papers.

Considering those samples made with preheataged Dexsil 300 and cured either with the three-step technique, (II-16, 31, 43), or the two-step technique, (II-17, 33, 45), handling characteristics were in general equivalent to the series made using Dexsil 300. Pre-fired tensile strengths were generally equivalent to the previously described series; post-fired tensiles were generally superior.

The next series of experiments evaluated two more techniques using a 15% solids solution in xylene of Dow Corning Silicone resin DC 994 as the impregnant to minimize wastage of Dexsil 300, with the standard mica paper substrate being 0.002 inch thick Samica 4100 from 3M. All tests were made in quadruplicate.

The first approach was a padding technique and consisted of dipping a previously xylene washed and dried section of mica paper into the polymer solution for three minutes to effect total impregnation, draining vertically for thirty seconds and then absorbing the excess surface resin between two layers of paper tissue, for thirty seconds. The sheet was then cured using the recommended schedule.

The second approach was to dip and drain the mica sheet as previously described and then remove the excess surface coating by running the sample through a pair of smooth, vulcanized rubber rollers. In the initial experiment with the rollers, they were used with the upper roller resting directly on the lower, resulting in an applied load equal to the roller weight, or approximately 1300 grams. This resulted in damage to the sheet coupled with delamination of the paper through adhesion of the tacky polymer to the rollers. A further series of samples was then prepared using a modified pair of rollers which had been given an artificial gap of either 0.004 inches or 0.006 inches by using metal shim stock wrapped around the lower roller. After completing the required cure, four 0.5 inch wide strips from each of the four samples prepared under a specified condition were tested for tensile strength. The results of the first series may be found in Table VI; it should be noted that the tensile strength is the average of the four values, and the average deviation indicates the spread between the lowest and highest values derived. Techniques a) and d) were repeated to evaluate reproducibility of the two most promising techniques found in this initial set, and are reported as e) and f) in Table 11.

TABLE 11

EVALUATION OF IMPREGNATION OF SAMICA 4100 MICA PAPER WITH DC994

	Loading, % by Weight	Thickness of Paper After Cure Average, Inches	Average Tensile Strength, psi	Average Deviation, ±, psi
a) Padding	5.63	.0025	11,800	720
	5.33	.0024-.0025	13,235	515
	5.37	.0024-.0026	13,102	832
	5.37	.0024-.0026	12,744	1044
b) Rollers, under own weight	3.00	.0022	10,091	682
	3.02	.0021-.0022	11,289	736
	3.01	.0022	13,591	955
	3.14	.0021-.0022	11,124	1149
c) Rollers, .006 inch shim	6.36	.0025	10,600	600
	7.15	.0024-.0025	11,615	907
	6.63	.0025	9,327	480
	5.75	.0023-.0025	9,004	518
d) Rollers, .004 inch shim	5.03	.0023-.0024	10,213	127
	4.73	.0022-.0024	8,216	560
	5.15	.0023-.0024	10,995	314
	5.01	.0023-.0024	10,537	259
e) Padding, for reproducibility	5.37	.0024	9,503	1085
	5.18	.0023-.0025	10,753	1932
	5.16	.0024-.0025	8,840	481
	5.07	.0024-.0025	8,812	240
f) Rollers, .004 inch shim, for reproducibility	5.09	.0024-.0025	10,125	641
	5.48	.0023-.0024	7,852	356
	5.81	.0024-.0025	9,043	1002
	5.47	.0024	7,328	336

TABLE 11 (continued)

EVALUATION OF IMPREGNATION OF SAMICA 4100 MICA PAPER WITH DC994 (CONT'D)

Technique	Loading, % by Weight	Thickness of Paper After Cure Average, Inches	Average Tensile Strength, psi	Average Deviation, ±, psi
g) Dip & padding	4.9	.0025	13,900	30
	4.9	.0023-.0024	16.700	800
	5.0	.0025-.0026	12,600	620
	5.4	.0025-.0026	14,100	600
h) Vac. dip & padding	6.5	.0025-..0026	14,700	110
	7.3	.0025	14,700	1430
	7.5	.0025-.0026	12,000	270
	7.2	.0025-.0026	14,300	550
i) 20% solution, dip & padding	7.6	.0024-.0027	13,400	360
	8.0	.0025-.0028	12,300	730
j) Dropwise coating & padding	7.4	.0025-.0027	14,100	670
	7.0	.0025-.0026	13,500	310
k) Dip & 5.0 mil nip steel rollers	8.9	.0025-.0027	12,700	840
	9.0	.0025-.0026	13,300	1460
l) Dip & 2.5 mil nip steel rollers	5.0	.0023-.0025	14,200	520
	4.8	.0024-.0025	14,200	1090
m) Dip & 2.0 mil nip steel rollers	4.9	.0024-.0025	15,600	330
	4.7	.0024	13,600	1100
n) Vacuum dip & 2.0 mil nip steel rollers	5.5	.0024-.0025	13,900	1300
	6.4	.0024	15,000	1100

TABLE 11 (continued)

Technique	Loading % by Weight	Thickness of Paper After Cure Average, Inches	Average Tensile Strength, psi	Average Deviation, ±, psi
o) Passing dry paper through wet 2 mil nip steel rollers	3.4	.0023	7,040	220
	4.1	.0023	11,900	1430
p) Multiple pass of (q)	6.6	.0024	11,000	1760
q) Passing dry paper through wet 4 mil nip steel rollers	12.6	.0028	12,800	110

It may be seen from Table 11 that padding yielded very consistent weight pickups but the roller technique using 0.004 inch shims gave slightly superior reproducibility of tensile strength. Balanced against this, however, is the fact that the average tensile strength is higher using the padding technique. Both techniques gave poorer reproducibility than expected which might be explained in part by the relatively poor uniformity inherent in the base paper stock.

Continuing the series, g) was a repeat of the previously reported series a) and e), consisting of a 3-minute dip of the paper in impregnant, draining and absorbing the surface excess between two layers of tissue.

Series h) was a vacuum impregnation technique which it was felt would effect more efficient impregnation through the paper. The mica sheet, immersed in impregnant, was placed in a desiccator and the container evacuated for thirty seconds. After breaking the vacuum, the sheet was padded with tissue to remove surface excess and then cured.

Series i) was impregnated from a 20% solids solution of DC 994 in lieu of the usual 15% solution, to evaluate the effect of higher solution concentration on weight pickup and performance. The application technique was a 3-minute dip followed by padding.

Series j) re-evaluated the Phase I technique which was to hand spread a surface layer of impregnant on the paper and remove the excess mechanically after visual wetting had occurred.

Series k), l) and m) continued the investigation of impregnation by roller coating applicators. The earlier studies employed vulcanized rubber rollers. Due to the deficiencies cited at that time, and to more closely reproduce industrial practice, this series was made using a steel two-roll mill primarily employed for plastics compounding. Recognizing the fact that the speed of the individual rollers is not synchronized, thus effecting a desirable shearing action in compounding, but a potentially undesirable shear in impregnation, the gap between the rollers was never reduced below the nominal 0.002 inch thickness of the paper stock. Thus, only minimal compaction could be effected. In this series, the paper was predipped for 3 minutes and then passed through a preset gap of 0.005 inches (Series k), 0.0025 inches (Series l), and 0.002 inches (Series m).

The samples of Series n) were first vacuum dipped as in Series h), and then passed through the roller pair set at a gap of 0.002 inches.

Series o), p) and q) were attempts to impregnate the mica paper by passing it through resin solution held in the nip of the rollers. In Series o), the roller gap was set at 0.002 inches, and the paper passed through once; in Series p), the roller gap was 0.002 inches and the paper was passed through 3 times; in Series q), the roller gap was set at 0.004 inches and the paper was passed through once.

Considering the various series, compiled in Table 11 the following conclusions may be drawn. Series g) effected a significant improvement in tensile strength, which can only be ascribed to improved operator technique. Although in Series h), vacuum impregnation resulted in the loss of large volumes of gas from the paper, and improved visual wetting, the net result was an approximately 50% increase in loading with no concurrent improvement in tensile values. Raising the solution solids, Series i) et seq., generally increased the loading, but had no significant effect on tensiles. Surprisingly, the very crude mechanical coating of Series j) resulted in comparatively acceptable strength, substantiating the values reported in Phase I, by a second operator.

Passing the preimpregnated sheets through rollers set at various gaps (Series k, l and m) demonstrated the relative merit of reducing the gap in the latter two sets, but effected no significant improvement in strength. Preimpregnating under vacuum (Series n), and passing through prewetted rollers (Series o-q) resulted, respectively, in equivalent performance in Series n) and reductions in strength in Series o), p) and q).

Further performance results characterizing the various methods of impregnating mica sheet are reported in Table 12. The overall conclusion drawn from the study was that no method evaluated offered a clear-cut advantage in performance, however, dipping and padding or vacuum dipping and padding were marginally superior. As the dipping and padding technique is similar to industrial practice, significant adjustments in commercial or pilot scale equipment are not anticipated.

On the basis of this study, two techniques, (a) dipping and padding, and (b) dipping followed by passing through rubber rolls gapped to 0.004" were repeated, using Samica 4100 paper and a 15% w/w Dexsil 300 solution in xylene, Tables 13 and 14 Technique (a) gave better reproducibility of loading pickup, higher initial tensile strength, and slightly better initial dielectrics. Initial handling characteristics were equivalent. Post-fired handling characteristics were equivalent for the two methods, while technique (a) gave marginally superior dielectric strength. Post-fired tensile strengths were reasonably equivalent.

TABLE 12

PERFORMANCE CHARACTERISTICS OF SAMICA4100 MICA PAPER

IMPREGNATED WITH DOW CORNING 994 SILICONE RESIN

Technique	% Loading W/W *	Handling Properties**				Dielectric Strength (kV)	Tensile Strength (psia)
		180° Wrap	360° Wrap	Abrasion Resist.	Pull Strength		
a) Dip and pad	5.63	1	1	2	1	4.5	11,800
	5.33	1	1	2	2	4.5	13,235
	5.37	1	1	2	2	4.2	13,102
	5.37	1	1	2	2	4.2	12,700
b) Weighted rubber rolls	3.00	1	1	2	1	3.9	10,100
	3.02	1	2	2	2	4.3	11,300
	3.01	1	1	2	1	4.5	13,600
	3.14	1	1	2	2	4.2	11,100
c) Rubber rolls, .006" shim	6.36	1	1	1	2	3.9	10,600
	7.15	1	1	2	2	4.3	11,600
	6.63	1	1	1	2	3.9	9,300
	5.75	1	1	2	2	4.4	9,000
d) Rubber rolls, .004" shim	5.03	1	1	1	2	4.3	10,200
	4.73	1	1	1	2	3.8	8,200
	5.15	1	1	1	2	4.0	11,000
	5.01	1	1	1	1	4.0	10,500
e) Dip and pad	5.37	1	1	1	1	3.9	9,500
	5.18	1	1	2	2	4.2	10,800
	5.16	1	1	1	2	4.0	8,800
	5.07	1	1	2	1	4.3	8,800
f) Rubber rolls, 0.004" shim	5.09	1	1	2	1	4.1	10,100
	5.48	1	1	2	1	3.8	7,900
	5.81	1	1	1	2	4.2	9,000
	5.47	1	1	1	1	4.5	7,300
g) Dip and pad	4.9	1	1	2	1	4.5	13,900
	4.9	1	1	2	2	4.1	16,700
	5.0	1	1	1	2	4.6	12,600
	5.4	1	1	1	2	4.5	14,100

TABLE 12 (CONTINUED)

Technique	% Loading W/W *	Handling Properties**				Dielectric Strength (kV)	Tensile Strength (psia)
		180° Wrap	360° Wrap	Abrasion Resist.	Pull Strength		
h) Vacuum dip and pad	6.5	1	1	2	1	4.2	14,700
	7.3	1	1	2	2	4.6	14,700
	7.5	1	1	1	2	4.7	12,000
	7.2	1	1	2	1	4.7	14,300
i) Dip and pad, 20% solution	7.6	1	1	1	2	4.4	13,400
	8.0	1	1	2	2	4.7	12,300
j) Dropwise coat and pad	7.4	1	1	2	1	4.3	14,100
	7.0	1	1	2	1	4.9	13,500
k) Dip and pass through 0.005" steel rolls	8.9	1	1	2	1	4.4	12,700
	9.0	1	1	1	1	4.3	13,300
l) Dip and pass through 0.0025" steel rolls	5.0	1	1	2	1	4.4	14,200
	4.8	1	1	1	1	4.4	14,200
m) Dip and pass through 0.002" steel rolls	4.9	1	1	2	2	4.5	15,600
	4.7	1	1	1	2	4.1	13,600
n) Vacuum dip and pass through 0.002" steel rolls	5.5	1	1	2	1	4.2	13,900
	6.4	1	1	2	1	4.4	15,000
o) Dry mica through wet 0.002" steel nip	3.4	1	1	2	3	4.3	7,000
	4.1	1	1	2	2	4.0	11,900
p) Repeat of o, multiple passes	6.6	1	1	2	3	4.1	11,000
q) Dry mica through wet 0.004" steel nip	12.6	1	1	2	2	4.7	12,800

* Cure schedule 60'/250°C

** Post fired performance characteristics not assessed.

TABLE 13

EVALUATION OF IMPREGNATION OF SAMICA 4100 MICA PAPER WITH DEXSIL 300

	Technique	Sample No.	Loading, % by Weight	Thickness of Paper After Cure Average, Inches	Average Tensile Strength, psi	Average Deviation, ±, psi
a)	Padding, (after cure)	1	4.77	0.0022	9,633	222
		2	4.86	0.0023	8,034	127
		3	4.22	0.0023-0.0024	11,125	1422
		4	4.88	0.0022	9,067	1111
b)	Padding (after exposure to 1250°F/60 min)	1		0.0026-0.0027	9,456	649
		2		0.0026-0.0027	12,868	109
		3		0.0026-0.0027	13,041	896
		4		0.0026-0.0027	11,817	488
c)	Rollers, 0.004 inch shim, (after cure)	1	3.91	0.0022	6,867	489
		2	5.07	0.0022-0.0025	7,745	235
		3	4.30	0.0022-0.0023	7,258	503
		4	5.82	0.0023-0.0025	7,589	297
d)	Rollers, 0.004 inch shim, (after exposure to 1250°C/60 min)	1		0.0025-0.0027	10,018	695
		2		0.0026-0.0027	10,843	710
		3		0.0025	11,029	782
		4		0.0026-0.0029	12,584	604

TABLE 14

EVALUATION OF SELECTED IMPREGNATION TECHNIQUES USING DEXSIL 300 AND SAMICA 4100 MICA PAPER

Sample Code	% Impreg. Load W/W *	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)		Comments
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	Before	After	Before	After	
Dip & pad	4.77				1	1	1	2	1	3	1	2	9600	9500	3.8	4.5	
	4.86				1	1	1	2	2	3	1	2	8000	12,900	4.8	4.8	
	4.22				1	1	1	2	2	4	1	1	11,100	13,000	4.5	5.0	
	4.88				1	1	1	2	1	3	1	2	9100	11,800	4.4	4.9	
Rollers 0.004" Shim	3.91				1	1	1	2	2	3	1	2	6900	10,000	3.9	4.6	
	5.07				1	1	1	1	2	3	1	2	7700	10,800	3.7	-	
	4.30				1	1	1	2	3	3	1	2	7300	11,000	3.8	4.4	
	5.82				1	1	1	2	2	4	1	2	7600	12,600	4.1	4.5	
Brush & pad	10.9				1	1	1	1	3	4	1	2	8300	10,700	4.6	4.6	
	12.7				1	1	1	1	3	4	1	1	9300	11,900	4.8	5.5	

* Cure Schedule: 15'/100°C 30'/200°C 30'/300°C

In conclusion, the impregnation of Samica 4100 with Dexsil 300 seems to be relatively independent of technique and responds favourably to the dipping and padding approach. No difficulties are anticipated in employing Dexsil 300 solutions on a pilot scale.

It may also be noted in Table 14 that another approach to impregnating Samica 4100 with Dexsil 300 was evaluated. In this method the impregnant is applied to the substrate with a soft artist's brush, and is intended to be used exclusively for evaluation of polymers where there is such a small volume available for study that dipping and padding is impractical. The method is feasible, resulting in performance properties comparable to other techniques at loading levels which are close to optimal.

b. Evaluation of UCARSIL Polymers as Alternative Candidate Impregnants

As noted in Section 3 , samples of several UCARSIL polymers were received from Union Carbide for evaluation as potential alternative impregnants for the primary dielectric mica. Details of the basic structure and variants are listed in Table 9, as are two pertinent thermal events established during instrumental characterization using TGA and DSC. Dexsil 300 is included as a comparative control; UCARSILS #10352-61 and #10352-40 were included as representative of submissions made late in Phase I.

A series of mica composites were prepared using solutions of the polymers listed in Table 9 at two concentrations, 10% and 15% w/w respectively, in toluene. All experiments used the standard Samica 4100 0.002 inch thick mica paper substrate. All the solutions were applied using the brushing technique previously described, followed by a light padding to remove surface excess. The impregnated sheets were cured in a forced draft oven at a standard schedule of 15 minutes/100°C, 30 minutes/200°C, and 30 minutes/peak of oxidative rearrangement less $\sim$ 80°C. This latter temperature was derived from work in Phase I when it was shown that superior performance accrued if the upper limit of the cure schedule was at or near the temperature of onset of oxidative rearrangement, rather than at the peak. The impregnation loadings and cure schedules are listed in Table 15 performance results are listed in Table 16 Samples C15 and C16 were repeats of C9 and C10, using 2% w/w of benzoyl peroxide on weight of resin to effect cure catalytically through the vinylic unsaturation.

TABLE 15

IMPREGNATION AND CURE CONDITIONS FOR UCARSIL® IMPREGNATED MICA SHEETS

Sample Code	Material	Impregnant Solution Constituent Ratio % (w/w)		Active Ingredient Absorption % (w/w)	Cure Cycle min./°C
		Active Ingredient	Carrier Solvent Toluene		
C1	Dexsil 300	10	90	10.9	15/100, 30/300, 30/300
C2	Dexsil 300	15	85	12.7	" " "
C3	10352-61	10	90	6.9	" " "
C4	10352-61	15	85	19.9	" " "
C5	10352-40	10	90	6.9	" " 30/390
C6	10352-40	15	85	12.5	" " "
C7	10352-127	10	90	7.6	" " 30/420
C8	10352-127	15	85	13.1	" " "
C9	10615-6	10	90	7.9	" " 30/390
C10	10615-6	15	85	15.2	" " "
C11	10615-22	10	90	7.7	" " "
C12	10615-22	15	85	13.7	" " "
C13	10615-50	10	90	8.3	" " "
C14	10615-50	15	85	9.3	" " "
C15	10615-16	10	90	10.2	" " "
C16	10615-16	10	90	9.5	" " "

TABLE 16

EVALUATION OF UCARSILS® AS PRIMARY IMPREGNANTS FOR

SAMICA 4100 MICA PAPER

Sample Code	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength (psia)		Dielectric Strength (kV)	
				BEFORE				AFTER							
	% Moisture Gain		% Weight Change	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.	180° Wrap	360° Bend	Abra-sion Res.	Pull Str.				
	Before	After										Before	After	Before	After
C1	0.12	2.70	-0.25	1	1	1	1	3	4	1	2	8,300	10,700	4.6	4.6
C2	0.06	2.76	-0.18	1	1	1	1	3	4	1	2	9,300	11,900	4.8	5.5
C3	0.22	1.19	-0.15	1	1	1	1	2	3	1	2	10,200	10,600	4.3	4.7
C4	0.07	3.83	+0.92	1	2	1	1	3	3	2	1	9,400	11,400	4.7	4.6
C5	0.23	1.28	-0.23	1	1	1	1	1	3	1	2	15,200	10,100	4.6	4.5
C6	0.50	2.72	+0.21	1	2	1	1	2	3	1	1	13,300	10,900	4.7	4.8
C7	0.08	2.10	-0.16	1	2	2	1	2	3	2	2	16,800	9,800	4.7	3.4
C8	0.35	2.82	+0.14	1	3	1	1	2	4	2	2	14,700	8,700	4.4	3.0
C9	0.28	1.74	∿0	1	2	1	1	1	3	1	2	13,200	9,600	4.9	5.1
C10	0.35	3.26	+0.21	1	2	1	1	1	3	1	1	15,100	11,800	4.1	4.2
C11	0.23	1.59	-0.30	1	1	1	1	1	2	1	1	16,600	12,300	4.8	4.6
C12	0.76	3.11	∿0	1	2	1	1	2	3	1	2	11,500	8,500	4.4	4.3
C13	0.08	1.04	-0.24	1	1	1	1	1	3	1	1	12,600	10,400	5.0	3.4
C14	0.29	1.23	-0.22	1	2	1	1	2	4	1	2	9,100	8,400	4.3	3.5
C15	0.06	1.60	-0.44	1	1	1	2	1	3	1	2	8,700	12,400	5.2	5.3
C16	0.44	1.81	-0.06	1	1	1	1	1	3	1	2	14,200	10,300	5.0	4.6

In general, the best combinations of performance properties were developed using the 33% phenyl methyl modified UCARSILS, C9-12 and C15-16. The higher loadings of impregnant showed slightly poorer handling characteristics, generally poorer tensile strengths and lower dielectric strengths. An insignificant level of performance improvement occurred, for this end use application, by using catalysis for curing. This is probably due to an insignificant level of performance strength being derived from cross-linking versus that derived from oxidative rearrangement. Interestingly, outstanding performance values derived from using a low loading of UCARSIL containing silyl hydride reactivity, which tends to support the postulated mechanism of interaction directly between the polymer and hydroxyl sites on the mica, as previously discussed.

After analysis of the performance results of these composites, a further series of composites was prepared using the most promising polymers 10615-6 and 10615-22. This series evaluated the effects of polymer loading and variations in cure schedule on performance. The composites are described in Table 17 performance results may be found in Table 18. All composites were made using the brushing technique, on Samica 4100, 0.002 inch thick mica substrates. An average Dexsil 300 impregnated mica composite sheet is also listed for comparison.

Composites made using both polymers showed acceptable performance characteristics which were generally superior to the Dexsil control. Overall the most promising composite was PS-88, Samica 4100, 0.002 inch thick mica paper impregnated with UCARSIL 10615-22 at a loading of ∿8% by weight, cured following the schedule predicted by DSC (Figure 14). Based on these results an order was placed with Union Carbide for ∿400 grams of 10615-22 for pilot tape trials.

As was noted in Section 3, it subsequently became necessary to consider having to use a blend of 10615-22 with 10615-6. A study was made of the effects of admixtures of the two polymers on the performance of the primary dielectric composite. The polymers were blended at 50:50 and 75:25 ratios (w/w), of 10615-22 and 10615-6 respectively; impregnation of Samica 4100, 0.002 inch thick mica paper was effected by brush application using a 10% w/w solution of the appropriate polymer blend in toluene. Neither polymer blend showed any evidence of incompatibility in solution. A standard cure schedule of 15 minutes at 100°C, 30 minutes at 200°C and 30 minutes at 400°C was employed.

TABLE 17

IMPREGNATION AND CURE CONDITIONS FOR SELECTED

UCARSIL IMPREGNATED MICA SHEETS

(Part 2)

Sample Code	Material	Impregnant Solution Constituent Ratio, % (w/w)		Active Ingredient Absorption % (w/w)	Cure Cycle minutes/°C
		Active Ingredient	Carrier Solvent Toluene		
PS-100	10615-6	5	95	3.0	15/100,30/200,30/300
PS-110	"	5	95	3.2	20/100,30/200,60/300
PS-111	"	5	95	5.5	20/100,30/200,60/300
PS -87	"	10	90	7.9	15/100,30/200,30/400
PS -93	"	15	85	15.2	15/100,30/200,30/400
PS -82	"	10	90	10.2	15/100,30/200,30/300 1200/200
PS -83	"	10	90	9.5	15/100,30/200,30/400
PS -96	10615-22	5	95	4.8	30/100,960/200
PS -97		5	95	5.1	30/100,960/200
PS -88	"	10	90	7.7	15/100,30/200,30/400
PS -94	"	15	85	13.7	15/100,30/200,30/400
PS -78	Dexsil 300	15	85	10.9	15/100,30/200,30/300

TABLE 18

EVALUATION OF SELECTED UCARSILS AS IMPREGNANTS

FOR SAMICA 4100 MICA PAPER
(Part 2)

Internal Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength psia		Dielectric Strength kV		Comment
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abrasion Res.	Pull Str.	180° Wrap	360° Bend	Abrasion Res.	Pull Str.	Before	After	Before	After	
PS-100	3.02	∿0	0.48	-0.71	1	1	1	2	2	2	1	2	6930	12,400	4.55	4.00	
PS-110	3.15	0.23	0.54	-0.46	1	1	1	2	2	3	1	2	7620	8160	4.91	3.93	
PS-111	5.49	0.15	0.45	-0.45	1	1	1	2	1	2	1	1	7460	8300	4.43	4.42	
PS-87	7.9	0.28	1.74	∿0	1	2	1	1	1	3	1	2	13,200	9600	4.9	5.1	
PS-93	15.2	0.35	3.26	+0.21	1	2	1	1	1	3	1	1	15,100	11,800	4.1	4.2	
PS-82	10.2	0.06	1.60	-0.44	1	1	1	2	1	3	1	2	8700	12,400	5.2	5.3	
PS-83	9.5	0.44	1.81	-0.06	1	1	1	1	1	3	1	2	14,200	10,300	5.0	4.6	
PS-96	4.8	0.06	0.31	-0.55	1	1	1	2	1	3	1	1	7540	11,500	4.7	4.44	
PS-97	5.1	0.07	∿0	-0.50	1	1	1	2	2	3	1	2	7300	9030	4.98	3.86	
PS-88	7.7	0.23	1.59	-0.30	1	1	1	1	1	2	1	1	16,600	12,300	4.8	4.6	
PS-94	13.7	0.76	3.11	∿0	1	2	1	1	2	3	1	2	11,500	8500	4.4	4.3	
PS-78	10.9	0.12	2.70	-0.25	1	1	1	1	3	4	1	2	8300	10,700	4.6	4.6	

Table 19 lists the performance characteristics of composites made using the two blends. Little difference was noted between the two; however, it was felt subjectively that the composites seemed stiff and "metallic" in feel, suggesting overcure. As noted in the previous section the preferred polymer was the silyl hydride variant 10615-22, thus based on these experiments, the 75:25 blend of 10615-22 and 10615-6 was chosen.

As excessive stiffening of the mica composite could detrimentally affect the application of cured mica tapes using commercial taping equipment, a brief study of the effects of temperature on the handling characteristics of the primary dielectric was made.

Impregnated mica samples made from either the 50:50 or 75:25 blends of polymer were cured for 30 minutes at one of 100°C, 200°C, 300°C or 400°C and evaluated for handling characteristics and tensile strength.

As shown in Table 20, handling characteristics of composites from the two blends were similar, with the onset of stiffening as evidenced by loss of 360° flexibility occurring above 300°C. Interestingly, the composite made using the preferred 75:25 blend of polymer developed full tensile strength after curing 100°C lower than the composite made using the 50:50 polymer blend.

Further evaluation of the blended UCARSIL was scheduled upon its receipt; however, the premature cessation of activities due to cost overruns precluded the completion of experimentation.

5. Mica Pretreatments and Polymer Stabilizers

A series of experiments was conducted to evaluate the usefulness of selected organometallic coupling agents in effecting improvements in the composite performance. Two general families of compounds were evaluated, silanes and organotitanates.

Silanes, $R'Si(OR)_3$ [7] are characterized by dual functionality. R' represents an organofunctional group usually bonded to the silica by a short alkyl chain, and OR represents a hydrolyzable alkoxy group attached to the silicon atom. The alkoxy groups hydrolyze to form silanols that react with or otherwise condense in the presence of silicas, silicates or metallic oxides. The organofunctional groups react with the

TABLE 19

PERFORMANCE PROPERTIES OF MICA COMPOSITE MADE USING BLENDS OF UCARSIL POLYMERS

Sample Code	Impregnant Loading (%/w)	S.T.T.C. % Moisture Gain Before 1250°F Exposure	S.T.T.C. % Moisture Gain After 1250°F Exposure	% Weight Change	Handling Properties Before 180° Wrap	Before 360° Bend	Before Abrasion Resist.	Before Pull Strength	After 180° Wrap	After 360° Bend	After Abrasion Resist.	After Strength	Tensile Strength (psia) Before	Tensile Strength (psia) After	Dielectric Strength (kV) Before	Dielectric Strength (kV) After
Series A*																
PS 124	7.11	0.15	1.39	-0.31	1	2	1	1	1	3	1	2	12,500	5,400	4.5	4.1
125	5.65	0.39	1.26	-0.16	1	2	1	1	1	3	1	2	13,700	9,300	4.4	3.8
126	6.02	0.23	1.22	-0.15	1	1	1	1	2	3	1	2	12,700	7,300	4.8	3.8
127	6.70	0.39	1.56	-0.23	1	1	1	1	1	2-3	1	2	9,800	7,200	4.2	4.2
132	6.10	0.23	1.33	-0.38	1	1	1	1	2	3	1	2	12,700	9,100	4.8	4.7
133	7.10	0.40	1.20	∿0	1	1-2	1	1	2	3	1	1-2	12,300	6,100	4.1	4.4
134	5.59	0.31	1.10	-0.16	1	1	1	1	1	3-4	1	2	13,100	9,000	4.4	5.2
135	6.74	0.23	1.28	∿0	1	2	1	1	1	3	1	1-2	12,500	7,000	4.7	5.2
Series B**																
PS 128	8.10	0.45	1.96	-0.08	1	2	1	1	2	3	1	2	9,800	6,100	4.9	4.7
129	8.08	0.39	1.79	-0.39	1	2	1	1	2	3	1	1	11,100	7,200	4.9	4.5
130	6.69	0.16	1.34	-0.24	1	2	1	1	1	3	1	2	14,000	8,300	4.3	4.0
131	7.56	0.24	1.41	-0.16	1	2	1	1	1-2	3	1	1-2	11,500	6,200	4.5	4.4

TABLE 20

EFFECT OF CURING TEMPERATURE ON THE PERFORMANCE OF MICA COMPOSITE MADE FROM UCARSIL BLENDS

Polymer Ratio (w/w)	Cure Temperature (°C)	Handling Characteristics				Tensile Strength (psia)
		180° Wrap	360° Bend	Abrasion Resist.	Pull Strength	
50:50	25	1	1	1	2	8,000
50:50	100	1	1	1	1-2	9,150
50:50	200	1	1	1	1-2	7,460
50:50	300	1*	1	1	1-2	9,310
50:50	400	1*	1-2	1	1	∿12,000
75:25	25	1	1	1	1-2	7,160
75:25	100	1	1	1	1-2	9,980
75:25	200	1	1	1	1-2	9,960
75:25	300	1	1	1	1-2	11,430
75:25	400	1*	1-2	1	1	∿11,500

polymeric organic matrix. In general, silanes offer one reactive site to the polymer and multiple reactive sites to the substrate.

Organotitanates are a relatively new family of coupling agents of the general structure $ROTi(OX'R^2Y)_n$.[8] R represents an organofunctional group which reacts with the substrate whereas Y^n represents the multiplicity of functional groups available in this molecule for reaction with the polymer. It is claimed that this offers a superior coupling function compared to the silanes.

Three silanes and two organotitanates were evaluated. The silanes were obtained from Dow Corning; the titanates were obtained from Kenrich Petrochemicals. Identification and descriptions of each are listed in Table 21.

All coupling agents were made up as 5% w/w solutions in toluene, applied to Samica 4100, 0.002 inch thick mica and dried at 100°C. The pretreated mica samples were then impregnated in the usual manner with selected poly(carborane siloxane)s. A description of the various composites may be found in Table 22, performance results are listed in Table 23.

TABLE 21

SELECTED COUPLING AGENTS EVALUATED IN CARBORANE-SILOXANE/MICA COMPOSITES

Coupling Agent Code	Molecular Configuration
Z-1211	Chloroalkyl functional silane
Z-6075	Vinyl functional silane
QZ-8-5069	Amino vinyl functional silane
TTS	Isopropyl triisostearoyl titanate
TSM2-7	Isopropyl, isostearoyl, dimethacryl titanate

The use of Z-1211 chlorosilane (PS-109) resulted in generally equivalent pre-fired performance characteristics, but a deterioration in post-fired tensile and dielectric strengths.

TABLE 22

IMPREGNATION AND CURE CONDITIONS FOR POLY(CARBORANE-SILOXANE) IMPREGNATED MICA SHEETS AFTER PRETREATMENT WITH SELECTED AGENTS

Sample Code	Pretreatment		Polymer Type	Polymer Impregnant		Cure Schedule for Polymer minutes/°C
	Type	Pickup wt. %		Concentration wt. % in Toluene	Active Ingredient Absorption,% (w/w)	
PS-100	-	-	10615-6	5	3.0	15/100,30/200,30/300
PS-111	-	-	10615-6	5	5.5	15/100,30/200,60/300
PS-109	Z-1211	0.2	10615-6	10	3.6	15/100,30/200,60/300
PS-108	Z-6075	1.5	10615-6	10	3.2	15/100,30/200,60/300
PS-103	QZ-8-5069	3.6	10615-6	10	4.6	15/100,30/200,30/300
PS-104	TTS	3.4	10615-6	10	1.6	15/100,30/200,60/300
PS-106	TSM2-7	1.7	10615-6	10	2.9	15/100,30/200,60/300
PS-78	-	-	Dexsil 300	15	10.9	15/100,30/200,30/300
PS-116	Ferric oxide	1.7	Dexsil 300	15	9.7	15/100,30/200,30/300
PS-118	Ferric oxide	0.2	Dexsil 300	15	11.9	15/100,30/200,30/300
PS-87	-	-	10615-6	15	7.9	15/100,30/200,30/400
PS-117	Ferric oxide	1.5	10615-6	15	9.5	15/100,30/200,30/300
PS-119	Ferric oxide	0.2	10615-6	15	10.5	15/100,30/200,30/300

TABLE 23

EVALUATION OF PRETREATMENTS FOR MICA IMPREGNATED WITH SELECTED POLY(CARBORANE SILOXANE)S

Internal Sample Code	% Impreg. Load W/W	S.T.T.C.			HANDLING PROPERTIES								Tensile Strength psia		Dielectric Strength kV		Comment
		% Moisture Gain		% Weight Change	BEFORE				AFTER								
		Before	After		180° Wrap	360° Bend	Abrasion Res.	Pull Str.	180° Wrap	360° Bend	Abrasion Res.	Pull Str.	Before	After	Before	After	
PS-100	3.0	∿0	0.48	-0.71	1	1	1	2	2	2	1	2	6900	12,400	4.6	4.0	
PS-111	5.5	0.15	0.45	-0.45	1	1	1	2	2	2	1	2	7500	8300	4.4	4.4	
PS-109	3.6	0.24	0.24	-0.72	1	1	1	2	2	3	1	2	7000	7200	4.7	3.8	
PS-108	3.2	0.08	0.79	-0.94	1	1	1	1	2	2	1	1	10,900	9300	5.2	4.9	
PS-103	4.6	0.38	1.43	-3.00	1	2	1	2	2	3	1	2	11,900	7600	5.0	3.3	3F
PS-104	1.6	∿0	1.19	-0.86	1	1	1	1	2	3	1	1	8500	10,600	4.9	5.0	3F
PS-106	2.9	∿0	0.24	-0.64	1	2	1	2	3	3	1	2	7900	8000	4.6	4.4	3F
PS-78	10.9	0.12	2.70	-0.25	1	1	1	1	3	4	1	2	8300	10,700	4.6	4.6	
PS-116	9.7	∿0	1.43	-2.93	1	2	1	1	3	4	1	1	9300	13,500	4.4	4.3	
PS-118	11.9	0.07	1.75	-3.18	1	2	1	2	2	4	1	2	7500	11,500	4.0	4.8	
PS-87	7.9	0.28	1.74	∿0	1	2	1	1	1	3	1	2	13,200	9600	4.9	5.1	
PS-117	9.5	∿0	1.35	-0.95	2	3	1	2	2	4	1	1	7400	11,600	4.3	3.3	
PS-119	10.5	∿0	0.78	-1.23	2	2	1	2	2	4	1	1	7200	12,600	4.0	3.9	

Sample PS-108, made using Z-6075 vinyl silane, showed an expected improvement in pre-fired performance characteristics, probably due to interaction between it and the vinyl unsaturation in the polymer. There was some loss of post-fired tensile strength and a general marginal improvement in dielectric strength.

Sample PS-103, made using the amino vinyl silane coupler, showed the severest loss of post-fired properties coupled with high moisture absorption and weight loss.

The use of titanate coupling agents generally effected little improvement in performance characteristics, either pre- or post-fired. It should be noted, however, that the use of TTS (sample PS-104), resulted in a composite which had performance characteristics approximately equivalent to the control, PS-100, but at only 50% of the polymer loading. Moisture pickup and weight loss on this sample were slightly poorer than the control.

No further experiments were made to evaluate silane coupling agents; further limited studies on titanate coupling agents is warranted.

Numerous references have been noted in the various reports of research on poly(carborane siloxane)s to the use of ferric oxide as an antioxidative stabilizer during high temperature exposure([9]). It was postulated that incorporation of colloidal iron oxide might effect significant improvements in retention of both pre- and post-fired handling and tensile strengths in the mica composite.

The following technique was used to form colloidal iron oxide in the substrate, Samica 4100, 0.002 inch thick mica paper. Ferric chloride hexahydrate was dissolved in acetone and used to saturate the mica. The mica was dried at 35°C, dehydrated at 200°C and then slowly heated to 540°C to oxidize the chloride. In all cases the mica sheet took on a rich red oxide colour. Samples monitored during the treatment showed quantitative conversion to the various forms, according to the following equation:

$$2FeCl_3 \cdot 6H_2O \xrightarrow{200°C} 2FeCl_3 + 12H_2O \xrightarrow{1000°C} Fe_2O_3 + 6HCl$$

A description of the composites may be found in Table 22 performance characteristics are listed in Table 23.

In composites using Dexsil 300, ferric oxide reduced moisture absorption but increased weight loss. Handling characteristics were essentially unchanged as was pre-fired tensile strength. Post-fired tensile strength retention was relatively better in the oxide protected samples. There was significant change in the dielectric strengths.

In the composition made using UCARSIL 10615-6, there was a significant increase in weight loss compared to the controls, and handling characteristics were generally poorer. Initial tensile strength was significantly poorer but there was a definite improvement in post-fired tensiles. Overall, dielectric strength was significantly deteriorated in the presence of ferric oxide. No further studies were made.

6. Braids and Tape Backing Yarns

a. Review of Available Refractory Yarns

The preferred Phase I secondary dielectric braid was based on the use of Refrasil®([1]), a refractory silica yarn manufactured by HITCO Corporation of Gardena, Calif. Although possessing excellent mechanical and thermal properties, this material tended to excessive moisture absorption. A literature update early in Phase II indicated that three new yarns introduced by the 3M Company, St. Paul, Minnesota, might have reduced moisture absorption, improved handling and better braidability. These materials were identified as AB312 alumina-boria-silica, ZS-11, zirconia silica, and AC-02 alumina chromia([10]).

Upon receipt of the samples, they, and other candidates from Phase I (Table 24) were evaluated, as shown in Table 25.

Considering the glassy materials, F1 and F7 showed relatively low moisture absorption and weight loss with generally poor retention of handling characteristics after 2 hours exposure to 1250°F as part of the modified short term test cycle. Significant deterioration in load to break values after heat exposure was also noted. F8, a modified fused silica similar to Refrasil, showed unacceptable moisture absorptivity and weight loss, relatively poor initial handling and tensile load to break properties, with a surprising improvement in both properties after firing. The Refrasils, F9 and F10, showed excessive absorptivity and weight loss, reasonable handling both before and after exposure and good retention of load to break strength. The chromia modified versions of Refrasil, F5 and F6, showed reduced absorptivity and weight change in comparison to the unmodified material

TABLE 24

DESCRIPTION OF FIBRE MATERIALS SUBJECTED TO INSULATION SCREENING TESTS

F-1	S glass, white high quartz yarn, twisted by Prodesco, 150 $^{1}/_{0}$ 12S.
F-2	ZS-11, zirconia-silica yarn, twisted 390 $^{1}/_{0}$, 3M Company.
F-3	AC-02, alumina-chromia yarn, twisted 390 $^{1}/_{0}$. 3M Company.
F-4	AB-312, alumina-boria-silica yarn, twisted 390 $^{1}/_{2}$, 3M Company.
F-5	C-1574-2, Irish Refrasil® cord, unknown twist, Hitco.
F-6	FYT-100-2T, Irish Refrasil® yarn, twisted $^{4}/_{5}$, Hitco.
F-7	Astroquartz yarn, twisted 300 $^{2}/_{8}$, J.P. Stevens.
F-8	Siltemp #1, unknown twist, Haveg Industries.
F-9	Refrasil® FYT-100Z, twisted 204 $^{4}/_{5}$, Hitco.
F-10	Refrasil® C100-2 cord, twisted 204 $^{4}/_{5}$, Hitco.

TABLE 25

EVALUATION OF POTENTIAL BRAIDING AND TAPE BACKING YARNS

Sample Code	% Moisture Gain		% Weight Change 2 hr @ 1250°F	Handling Properties								Load to Break, lb		Comment*
				Before				After						
	Before	After		180° Wrap	360° Bend	Abra-sion Res.	Pull Strength	180° Wrap	360° Bend	Abra-sion Res.	Pull Strength	Before	After	
F1	0.27	0.27	-.53	1	1	2	1	3	2	3	2	7.7	1.4	1I; 1.32%
F2	~0	0.24	-1.05	1	1	3	2	1	2	3	2	4.8	2.9	1I, 1J; 1.95%
F3	~0	0.27	-23.1	1	2	1	2	3	3	3	3	5.1	1.3	1I, 3J; 1.43%
F4	0.27	0.13	-.60	1	1	1	1	1	1	1	1	15.0	6.4	5.9%
F5	2.63	2.8	-1.52	1	1	2	1	1	1	3	2	12.8	10.8	3G; 1.94%
F6	5.8	4.7	-1.6	1	1	1	2	1	1	2	2	2.6	5.9	1I; 5.7%
F7	0.31	~0	-0.19	1	1	3	2	1	2	4	3	11.2	1.4	1I, 1J; .31%
F8	11.6	12.0	-7.38	1	1	3	3	1	2	2	2	0.8	4.7	3I; 6.5%
F9	11.0	7.0	-4.6	1	1	2	2	1	1	2	2	4.3	4.6	3I; 6.4%
F10	7.4	4.0	-2.8	1	1	2	1	1	1	2	2	11.8	10.9	3I; 7.0%

* Includes percentage weight loss on drying at 200°C to constant weight of virgin yarn material.

and equivalent or slightly better handling. Comparatively, load to break values for the yarn were poorer and for the cord were equivalent. As previously experienced, the chromia version showed excessive dusting and fraying.

Considering 3M's materials, the zirconia based yarn, F2, showed low absorptivity and weight change, relatively poor handling properties, low initial load to break strength and poor retention of strength. The chromia doped alumina, F3, showed low absorptivity but severe weight loss, quite good initial handling with poor retention of properties and poor load to break values. The boria doped alumina, F4, showed low moisture absorption, quite low weight loss, outstanding pre- and post-fired handling characteristics, the highest initial load to break strength and reasonable strength retention. Included in the comments column of Table XVI is a series of percentiles for each yarn. These are the percentage weight loss for each yarn during the initial step of the modified short term test cycle which conditions the sample to constant weight at 200°C. These weight losses are attributed to loss of lubricant (a fact confirmed for Sample F4 where it was established with 3M that AB312 is lubricated with Kraton oil and glycerine), and in the case of the Refrasils, lubricant and adsorbed moisture.

Based on these studies, AB312 was selected as the prime candidate for the primary dielectric braid and tape backing in Phase II.

b. Evaluation of Commercial Tape Backings

As part of the study to optimize the mica primary dielectric, various commercial mica tapes were evaluated to determine the nature and design of the backings used. This would allow a backing fabricated from a selected high temperature resistant yarn to be selected having an optimum yarn count. Thirteen tapes were examined, ranging from cloth backing, to scrim, to unidirectional filaments. In general, it was noted that the cloth employed multiple plys of yarn, heavily twisted, while scrim and filament backings tended to be one-ply and had little or no twist. These latter offer the advantage of allowing flatter, thinner cross-sections to be achieved which offer sufficient absorption of impregnant for efficient adhesion without the excessive pickups which might be expected from cloth weaves. The mica-based tapes examined all used scrim, at a warp/filling count of 60x36 per inch; cloth backings for other dielectrics ranged from 60x52 to 208x48. Filamentary backings ranged from 20 per inch to 68 per inch in the warp direction.

Two specimens of mica tape with glass scrim backing, and one sample with unidirectional filament backing were evaluated for tensile strength and elongation, to determine the relative performance of commercial materials. The results may be found in Table 26. The tensile strengths of these tapes range from ∿15,000 psi to ∿23,000 psi with elongations of from ∿3.5 to 8%. In comparison, the experimental tapes listed in Table 18 and impregnated with a poly(carborane siloxane) achieved tensiles of ∿15,000 psi unbacked, with elongations of ∿0.5%. This implies that the successful mating of a backing to the experimental tapes would, as expected, effect a significant increase in tensile strength, and improve the elongations, which would be advantageous in machine taping operations.

C Usefulness of AB312 for Braiding and Backing

To further quantify the use of AB 312 as a braiding yarn, discussions were held with 3M and with Bentley Harris of Lionville, Pennsylvania, who have considerable experience in braiding AB312, indicating that it would be necessary to employ a finer yarn than the AB312 390 $^{1}/_{2}$ submitted in order to braid <16 AWG. Accordingly, an order was placed for two 30-spool braider loads of AB312, sufficient 390 $^{1}/_{2}$ to braid 12 AWG-16 AWG, and sufficient 390 $^{1}/_{0}$ to braid 18 AWG-22 AWG.

On the recommendation of 3M, contact was also made with Woven Structures Inc., of Los Angeles, Calif., who have expertise in weaving AB312, in order to establish the usefulness of AB312 as a tape backing. Woven Structures advised that 390 $^{1}/_{0}$, the thinnest configuration of AB312 available, would weave into an unacceptably thick (∿0.007") backing at a cloth count expected to give good support, i.e. 60 x 36 nominal. They undertook to examine the feasibility of weaving at a much lower count, i.e. 20 x 20-30 x 30 to determine if a thinner backing can be achieved. Concurrently, they examined the feasibility of a composite weave having an AB312 390 $^{1}/_{0}$ warp and an Astroquartz® 500 $^{1}/_{0}$ fill which might significantly reduce the thickness. Unfortunately, on completion of their studies, they were unable to achieve a satisfactory woven product having a thickness of less than 0.007 inches, using any of their suggested combinations.

Due to the difficulties in procuring backing based on AB312, samples of other potentially useful backings were ordered. These were: (1) Astroquartz 503 from J.P. Stevens, having relatively good thermal stability of 1250°F, a count of 50 x 40, and a thickness of 0.005"; and (2) "E" glass based backing from Burlington Industries in two configurations,

TABLE 26

RELATIVE MECHANICAL PROPERTIES OF SELECTED COMMERCIAL MICA TAPES

Tape & Source	Backing Type & Count	Thickness x Width inches	Tensile Strength psia	Elongation %
#4351				
3M Company	Scrim, 60x36	0.004 x 1.00	22,000	6.8
			20,600	4.1
			22,900	7.9
#X4724	Scrim, 60x36	0.0075 x 1.00	15,000	3.9
3M Company			16,500	3.4
			18,000	3.4
#77925	Unidirectional fila-	0.0041 x 0.50	17,200	Sample continued to elongate
G.E. Co. Ltd.	ment 20/inch		18,400	after mica tape failed. No
			16,600	clear break.
			18,400	

#104, 60 x 52, and 0.0012" thick, and #108, 60 x 47, and 0.002" thick. Although backing based on the higher performance "S" glass fiber would have been preferable, it was established that "S" is available only in yarn of coarseness comparable to AB312.

Upon receipt of these materials, they were evaluated in the short-term test cycle and for handling characteristics (Table 27).

It may be noted that there was an excessive weight loss in the "E" glass scrim, both at 200°C and at 1250°F which can be ascribed to loss of the heavy loadings of lubricant present on the fine strands. Performance properties of the "E" glass deteriorated significantly after exposure for 200 hours at 1250°F; during the abrasion test the scrims powdered completely. Astroquartz being relatively thick and densely woven showed more resistance to abrasion but deteriorated in the same manner.

d. Mica/Scrim Bonding Studies

Several attempts were made to bond Samica 4100, 0.002 inch paper to Astroquartz 503 cloth by simultaneous impregnation using Dexsil 300. Due to excessive imbibement of the polymer, and the basic lack of "tack" common to the all methyl poly(carborane siloxane)s, little success was achieved. As alternative approaches, both catalytic and thermal curing as well as thermal/catalytic cure under pressure were employed, using Dexsil and UCARSIL 10352-61, with no success.

It had been noted, however, that the 33% phenyl methyl UCARSIL with a 4% vinylic unsaturation possessed a degree of "tack" during the cure schedule evaluating it as an impregnant. Using a 10% w/w toluene solution of UCARSIL 10615-6 containing 2% w/w benzoyl peroxide on polymer, two sheets of mica paper were impregnated and air dried. Mating surfaces of the sheets were then coated with a thin layer of polymer solution, air dried and contact bonded. The mated pair were then cured according to the standard schedule. After curing, peel tests showed that failure occurred by delamination of one of the mica sheets, rather than by glue line failure. After exposure to 1250°F, however, failure occurred, during peel, at the glue line. As these results were encouraging, attempts were made to impregnate and bond mica sheet to Astroquartz 503, using the same polymer. Although bonding was not wholly satisfactory, it was significantly superior to that achieved with Dexsil 300.

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DEVELOPMENT OF HIGH TEMPERATURE (1250 F) WIRE AND CONNECTORS.(U)
JUL 77 M A DUDLEY, F D BAYLES, P O SCADDING F33615-75-C-2040

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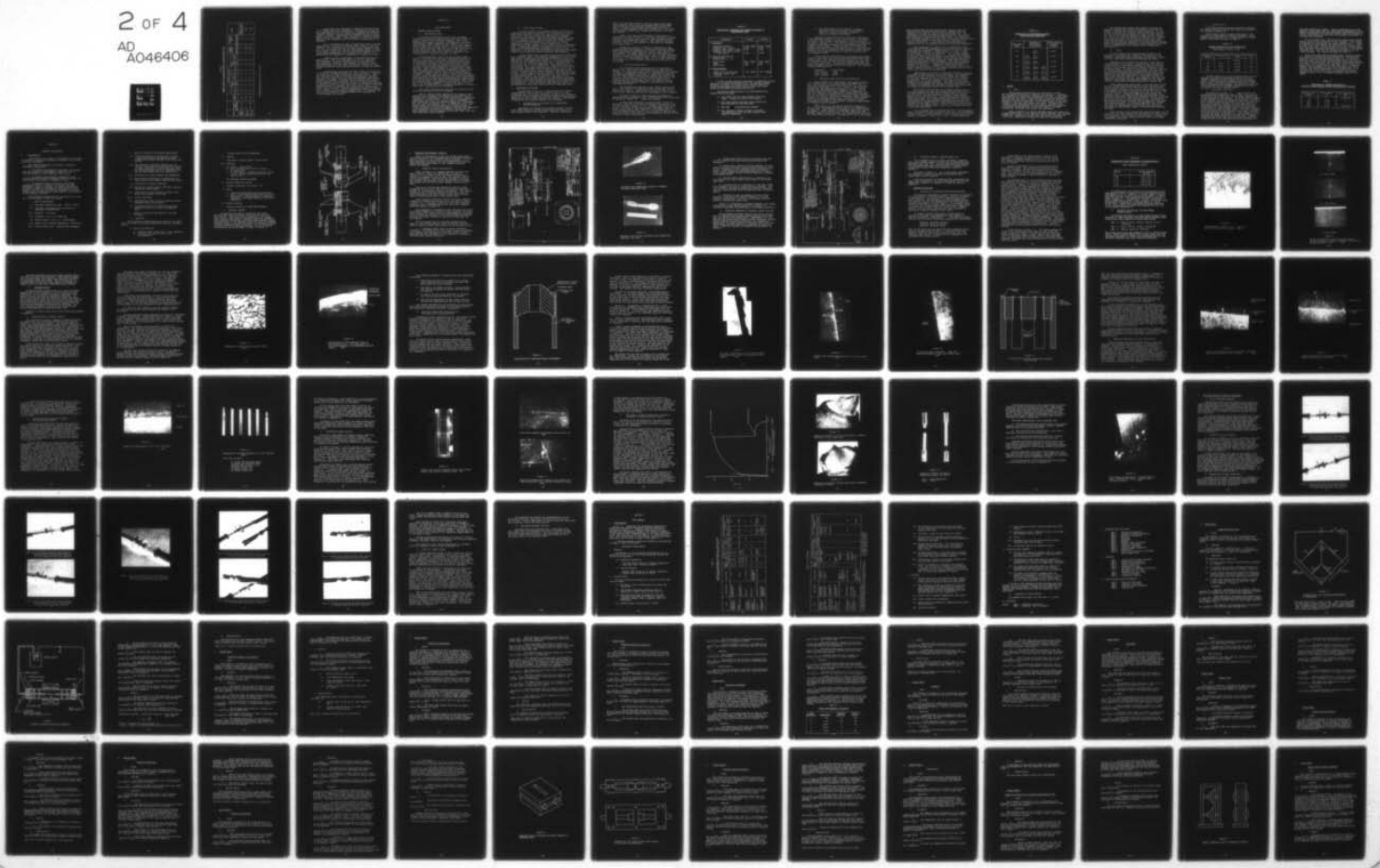

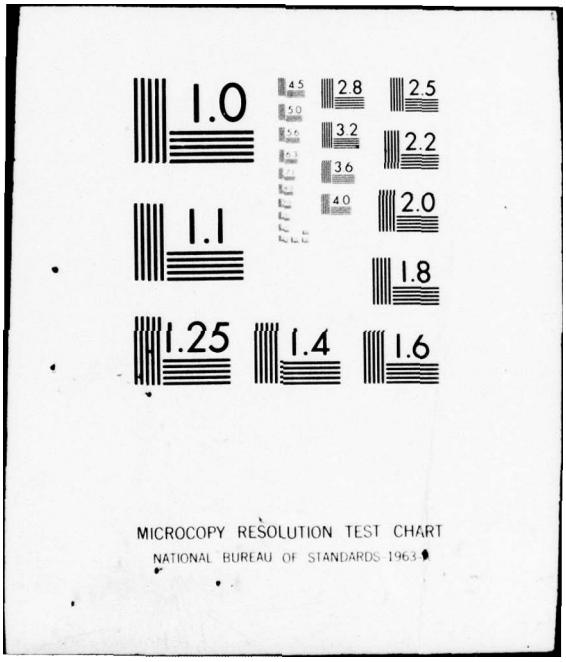
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3.2
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4.5
5.0
5.6
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963

TABLE 27

THERMAL STABILITY OF SELECTED BACKINGS IN SHORT TERM TEST CYCLE

Sample	% Moisture Gain		% Weight Change	Handling Properties*								Tensile Strength lb/inch		Dielectric Strength kV		Comment
				Before				After								
	Before	After		Flex	Stretch	Abrasion Res.	Tear Strength	Flex	Stretch	Abrasion Res.	Tear Strength	Before	After	Before	After	
Astroquartz 503	0.51	0.46	-0.32	1	2	2	1-2	4	4	4	4	-	-	-	-	Weight loss on drying @ 200°C -0.13
"E" 104-38	0.37	~0	-1.56	1	3	3	1-2	3	4	4	4	2.3	0.2	-	-	-1.80
"E" 108-38	0.13	0.62	-1.03	1	2	2	1-2	4	4	4	4	3.0	1.0	-	-	-0.85

* Handling determined after 200 hr at 1250°F.

Experiments were performed to determine the interaction of "E" glass scrims with impregnant. Both styles 104 and 108 were impregnated with a 15% solution of UCARSIL poly(carborane siloxane) and were found to absorb approximately 25% of their weight in polymer. Astroquartz 503, however, absorbed approximately 45% of its weight. After exposure to 1250°F, all three materials were extremely stiff and brittle, disintegrating on handling.

As it is desirable to have a backed mica tape of minimum cross section to effect the smallest space factor in the wire assembly, "E" glass scrim will be used as the supportive backing. It is not expected that any woven backing based on glass or refractory fibres will retain a significant degree of flexibility when impregnated with a polymer which converts to a glassy matrix on exposure to temperatures in excess of 1000°F.

A short series of experiments to evaluate adhesion of Samica 4100, 0.002 inch thick paper to the "E" glass scrim was completed. Using the UCARSIL 10615-6 vinyl modified poly(carborane siloxane), good adhesion was effected when a pre-impregnated mica sheet was contacted to a scrim and then bonded by imbibing resin through the glass, followed by thermal cure.

A preliminary evaluation was made of both the 50:50 and 75:25 blends of UCARSIL polymer for their potential in adhering the woven scrim to the mica primary dielectric sheet. Using the technique described in the previous paragraph, complete wetting of the paper and scrim could be effected using polymer dissolved in toluene. Although the evaluation of the cured, backed composite was subjective, using 75:25 blend resulted in an apparently superior interfacial bond as evidenced by handling characteristics and residual tack after cure at 200°C, with both blends retaining a good bond with significantly reduced tack after curing at 300°C. Further experiments planned to optimize cure, loading and performance characteristics of composite made from the pilot batch of blended UCARSIL were cancelled due to the program cost overrun.

SECTION III

WIRE FABRICATION

A Primary Dielectric Tape

1. Tape Manufacturing

A survey of manufacturers having pilot equipment potentially suitable for manufacturing continuous lengths of primary dielectric mica tape was completed. Two of the companies were chosen for further investigations, the Essex International Company, Newmarket, New Hampshire, and the U.S. Samica Company Ltd., Rutland, Vermont. This latter company was also chosen for the fact that it is now the sole source of the Samica 4100 mica paper which is the base stock for the primary dielectric tape. The 3M Company, from whom the original samples were obtained, sold its interests in mica technology to U.S. Samica on October 15, 1975.

A visit was made to the facilities of Essex International to discuss their ability to manufacture backed mica tape on a pilot scale. The overall conclusion drawn from the conversations was that they anticipated some difficulty in impregnating and heat treating the backed tapes, having to use at least two separate machines and having no means of achieving the highest temperature of the preferred curing schedule. Essex received samples of raw Samica 4100, 0.002 inch thick paper, a silicone resin dissolved in toluene which had a curing schedule similar to that of the preferred poly (carborane siloxane)s and "E" glass, style 108 scrim backing in order to conduct laboratory trials. After extensive experimentation they indicated that they would be able to manufacture pilot quantities of tape only by impregnating the composite and drying on one machine and effecting a preliminary cure at 200°C in a separate oven. The necessary final cure at $\geq$ 300°C would have to be effected elsewhere. As this would result in too high a risk of damage due to the excessive handling in a fragile green state, negotiations were terminated.

U.S. Samica indicated that they had no continuous pilot facilities and could not be of assistance.

Over 50 further custom impregnators and composite manufacturers were canvassed, with emphasis being placed on the availability of continuous run curing ovens having a temperature capability to at least 300°C. At the time of termination of experimental activities, two further potential candidates had been identified, the General Electric Company Ltd., Schenectady, New York, and Fiber Materials Inc., of Biddeford, Maine. The former company has extensive experience in manufacturing of continuous lengths of mica based composite and has high temperature curing equipment; the latter is primarily a manufacturer of custom continuous length composite using carbon fiber, also with high temperature curing equipment.

2. Pilot Taping Trials

A visit was made to the Simcoe, Ontario, facilities of Canada Wire and Cable to determine the feasibility of running 0.25" wide commercial backed mica tape on production tape applicators. The initial work was performed on a SYNCRO® vertical taping machine, using 3M 4351 glass backed mica tape, slit to a nominal 0.25" width and 12 AWG soft copper conductor overlaid with served cotton to bulk the O.D. of the copper to a nominal 0.1285", the anticipated O.D. of the experimental 12 AWG conductor with a single braid of refractory yarn. Although the severity of mechanical deformation of the tapes by the SYNCRO machines precluded its use as a tape applicator, the results were sufficiently encouraging to warrant further trials using horizontal planetary type equipment.

A second taping trial was then completed using horizontal Kraft planetary gear. The conductor employed was 12 and 22 AWG OFHC copper, overbraided with Refrasil® FYT-100Z silica yarn applied by a New England butt braider at 26.5 picks/inch to a nominal O.D. of 10 AWG (see Section 3.2.1). The tape used was U.S. Samica 4351 silicone impregnated mica backed with "E" glass scrim,slit to 0.25 inches wide. Some difficulties were encountered. The copper conductor was found to be too soft as it was easily deflected from the machine centre line by the pull of the taping heads, thus resulting in periodic erratic overlay. Within this limitation, however, ∿100 continuous feet of wire was successfully overlaid with both four and six layers of tapes respectively, at the machine's top speed of 100 feet/minute. Tape contact angles, butt separation and degree of overlay values and throughput gear drive ratios were determined which will allow setting the equipment for manufacturing prototype wire. It is anticipated that prototype wire will be successfully fabricated using horizontal tape applicators.

B Application of Braids

As mentioned in Section I, the preferred wire design incorporated an inner braid of refractory yarn as it was recognized that it would be impossible to apply mica composite tape below a certain substrate outside diameter without physical damage to the tape through creasing and cracking. Experiments to determine this outside diameter were undertaken.

1. Determination of Finished Wire Intermediate Outside Diameters

Experiments to determine the minimum diameter of bare wire around which the present impregnated mica tape can be wound without damage were completed. Unbacked, preheataged

Dexsil 300 impregnated Samica 4200 mica paper based tapes were prepared, 0.25" wide, and wrapped around bare copper wire in the butt edged helical configuration devised in Phase I. Starting with 8 AWG wire (0.1285" dia.), the gauge was gradually reduced until significant damage to the tape was noticed.

Using 0.25" tape it was possible to tape wrap to a diameter of 10 AWG (0.1019"). At 12 AWG, the angle of attack required to maintain butt edging was impracticably shallow, and there was severe edge flaking. Upon reducing the tape width to 0.125", it was possible to tape down to a diameter of 14 AWG (0.0641").

Examining a section of Refrasil overbraided 12 AWG conductor made during Phase I, it was found that the average braid thickness was approximately 0.015". Using this value, and assuming that three layers of unbacked tape would be 0.006"thick, a finished composite O.D. for a 22 AWG conductor, assuming 2 layers of braid and three layers of tape, was found to be 0.131" or approximately 8 AWG. For a 12 AWG conductor, the overall O.D. of a finished composite wire would be 0.153" or 6-8 AWG.

2. Pilot Braiding Trials

The overtaped 12 AWG experimental wire described in Section A-2 was further overbraided with a mixed load of Refrasil, "E" glass and cotton using a New England butt braider, in order to determine the ability to overbraid mica tape and the resultant diameters and material weights per linear foot of wire. The mixture of fibres was used as there was no full load of ceramic available at that time and there was no intention to fire the resultant wire; the glass and cotton both had the same nominal diameter as the Refrasil and the preferred AB312 yarn.

The overbraid was applied at pick counts ranging from 27.5 to 39.3/inch; 37.9 picks was chosen as optimum, giving a smooth 100% coverage. Approximately 40 feet of each of the four and six tape layered wire was overbraided in this fashion.

One foot of each of these wires was disassembled to determine nominal diameters, tape lengths/linear foot and weights of materials. Table 28 lists the findings.

It should be noted that the wire having 4 layers of dielectric tape had a dielectric breakdown strength of 5.2 KV and that having 6 layers, 9.7 KV. Although this latter value is well in excess of both the program requirements and the design safety factor (600 V RMS and 2.2 KV respectively), it is a wire design option that will be retained in case of significant dielectric deterioration during mechanical testing of the preferred 4 layer configuration.

TABLE 28

COMPARATIVE DIMENSIONS AND MATERIAL WEIGHTS OF EXPERIMENTAL WIRE

Parameters	4 Tapes	6 Tapes
a) Diameters		
Conductor diameter	0.081 inches	0.081 inches
Diameter over 1 braid	0.104 "	0.106 "
Diameter over braid & tapes	0.135 "	0.155 "
Diameter over braid, tapes & outer braid	0.165 "	0.186 "
b) Material Weights per Linear Foot		
Inner braid	0.97 grams	0.98 grams
Tapes	1.98 "	3.56 "
Outer braid	1.25 "	1.35 "
c) Miscellaneous		
Length of 0.25 inch wide tape per linear foot of wire	80 inches	135 inches

To gain further operator experience in mechanical braiding so as to evaluate modifications to the equipment deemed necessary to handle the ceramic yarns, further lengths of 12 and 20 AWG copper wire were overlaid with one or more layers of mixed Refrasil/glass/cotton yarn.

The machine modifications to the New England butt braider were as follows:

1) All bobbin carriers were matched with their support sleeves to ensure free running.

2) All fibre guides were made free running and were polished to minimize chafe.

3) The tensioning springs were removed.

4) All surfaces on which braided or braided and taped wire would run were overlaid with plastic cushioning.

5) New guide wheels were installed to ensure that wire changing direction in the machine would do so over the largest convenient radius to minimize tape damage.

Braiding 12 AWG wire with the mixed yarn load established optimum coverage to be at a pick count of 16-25/inch; on 20 AWG, 100% coverage was achieved at 14-18 picks/inch. As noted in Section B-1 , the minimum diameter of overbraided conductor considered acceptable for successful taping was 10 AWG (∿0.102 inches). As may be seen in Table 29, this diameter was maintained in the taping trials. In was obvious, however, that the 14 through 22 AWG conductors would require overlaying with multiple braids in order to bulk the O.D. to a nominal 10 AWG.

A section of 20 AWG copper was overbraided with three layers of the mixed yarn load, evaluating several pick counts on each layer. The optimum count was found to be 16.1/inch on the inner braid, 18.3/inch on the intermediate layer, and 32.1/inch on the outer. The resultant braid sleeve was very tight and showed no twist around the conductor. The resultant outside diameters were:

Conductor	0.031 inches
Plus 1 braid	0.054 "
Plus 2 braids	0.090 "
Plus 3 braids	0.120 "

3. Braiding Trials with the Preferred Yarn

Short lengths of 12 AWG Inconel clad copper (Kulgrid 28®, Sylvania Wire), were overbraided with AB312 390 $^{1}/_{2}$ yarn. The optimum pick count was found to be 16 to 20/inch, yielding an overbraid O.D. of ∿0.11 inches. The material braided well but showed some tendency to "fuzz". Conversations with the Bentley Harris Company of Lionville, PA, who have expertise in braiding AB312, established that this was (a) a function of damage during braider bobbin winding, and (b) the radius of the fibre guides which cause filamentary snapping in the high modulus yarn. Braider loading damage could not be overcome as the yarn was already in house; adjustments were made to the braider to minimize guide damage by increasing guide diameters.

AB312 390 $^{1}/_{0}$, a yarn of one half the diameter of 390 $^{1}/_{2}$, was recommended by Bentley Harris for overbraiding >16 AWG wire. Upon receipt of an order of this yarn from the 3M Company, a short length of 20 AWG OFHC copper was overbraided, using no spring tension. The optimum pick count for the inner braid was 20/inch, yielding an O.D. of ∿0.051 inches.

Some difficulties were encountered with fraying, which was overcome with adjustments; more serious, however, was the tendency of the yarn to jump off the guides and snarl. No improvement accrued from applying spring tension; the problem was corrected by reducing the braiding speed. The difficulty was ultimately traced to the fact that $^{1}/_{0}$ yarn is twisted "wild", i.e. all yarns handed in the same direction, whereas $^{1}/_{2}$ is twisted "balanced", i.e. base yarns in the same direction, composite yarn in the opposite direction to the base components.

Upon completion of these trials, overbraiding of the 12 AWG through 22 AWG experimental Inconel clad silver conductor with AB312 was completed. Combinations of $^{1}/_{2}$ and $^{1}/_{0}$ yarn were employed to obtain the preferred O.D. of 0.100 - 0.000 + 0.005 inches, as shown in Table 29. All outside diameter values were measured by micrometer, and represent the diameter of conductor and compressed braids to the slip ratchet loading of the micrometer, which parallels the compressive effects of commercial horizontal taping equipment. The nominal diameter of the braided wires is ∿0.010 inches greater than the values reported in Table 29. Although it would be desirable to achieve the desired O.D. without this discrepancy, due to the minimal tension necessary to braid AB312 and the effects of yarn twist, this could not be accomplished.

Approximately 350 feet of 12 AWG and 100 feet of each of 14 through 22 AWG conductor were overbraided. All experimental conductor had approximately 20 feet of Inconel 600 "leader" of the same gauge and 10 feet of Inconel 600 "tail" attached by soldering. This extra length of wire was to facilitate feeding into the taping equipment. The leads and tails were attached by overlapping the Inconel wire and the conductor for ∿3 inches and soldering the resultant parallel pair using Handy and Harman silver/copper solder 541 with borax flux.

All braids were started on the leader, between 6 and 12 inches before the joint, and braided straight over the joint. The same procedure was used when finishing off the braid on the tail wire. The braid ends were anchored using contact cement and twisted wire ties. To minimize breakage from the "wild" twist yarn, the average braider speed was ∿1 feet/minute. Considerable effort was expended throughout the operation to minimize machine jamming by the loose filamentary "fuzz" which collected on the bobbin guides. Because all spring tension was removed from the bobbin feeds, the wear on the guides was less severe than anticipated.

It is intended that all braided wire will be fired before taping to remove the Kraton/glycerine lubricant. The recommended firing schedule is either ambient to 600°C, holding there for 15 minutes, or ambient to 800°C over 1 hour, cooling immediately.

TABLE 29

COMPOSITION OF EXPERIMENTAL BRAIDS
OVER PROTOTYPE CONDUCTOR

Wire Gauge (AWG)	Sequence of Yarn Layers (picks/inch)			Final Outside Diameter (inches)
12		1/0 (24.6)		0.101
14	1/0 (21.8)	1/0 (36.4)		0.102
16	1/0 (21.8)	1/2 (28.4)		0.102
18	1/0 (18.3)	1/0 (28.4)	1/0 (36.4)	0.104
20	1/0 (16.4)	1/0 (22.8)	1/0 (32.1)	0.101
22	1/0 (16.4)	1/0 (21.8)	1/2 (28/4)	0.102

C Armour

1. Wire

The preferred subcontractor for armouring in Phase I was the Radcon Corporation of Roselle, New Jersey([1]). Although this company did a generally satisfactory job, due to their small size they did not have a large selection of knitting equipment and thus were only able to apply an approximation to the outside diameter of the wire submitted. This armour tended to have many "misses" in the knit and tended to be slack. As Radcon had indicated that considerable time and expense would be required to overcome these problems, an alternate source, the Metex Corporation, of Edison, New Jersey, was identified.

Sample lengths of 12 and 22 AWG OFHC copper wire were overbraided with cotton to bulk them to a nominal 8-6 AWG. The finished 12 AWG had a diameter of ∿0.160 inch, the 22 AWG a diameter of 0.142 inch. These were forwarded to Metex for overknitting.

The overarmoured samples were promptly returned and examined, showing that on the wire based on the 22 AWG conductor, a satisfactory overknit could be achieved. On the 12 AWG containing wire, however, difficulties with centering the wire in the knitters resulted in gaps and poor quality fit. Knitting a tube and applying it over the wire resulted in quality equivalent to that for the smaller wire. Metex have indicated that in-situ knitting on the larger gauge could well be possible, depending on finished outside diameter, using a modified centering device costing between $500-$1,000. Metex is recommended as the primary armour subcontractor for Phase II.

2. Cable

As the ultimate goal of the harness section of the program is to develop four conductor cables mated to the connector, experiments were undertaken to make short lengths of cable, using the armoured wire returned from Metex.

Discussions with Canada Wire and Cable cable experts indicated that, using plastic jacketed wire, the four wire bundle known as a star quad configuration would be twisted to form a cable whose lay was 40 times the diameter of the individual wires. As there is some concern that the armouring on the individual wires would detrimentally interfere with the abrasion resistance and flexibility of the cable, three samples of cable were hand layed up by clamping lengths of wire in the head and tailstock of a lathe and twisting the head stock to achieve the lay. The resultant cable was then clamped at both ends and forwarded to Metex for the application of a constrictive knitted overarmour of Inconel 600.

Three samples were fabricated, a star quad using 12 AWG Cu core, cotton overbraided and Inconel 600 armoured, which had an average wire O.D. of 0.160 in. and a lay of 6.40 in., a star quad of 22 AWG Cu core, cotton overbraided and Inconel 600 armoured, which had an average wire O.D. of 0.142 in. and a lay of 5.68 in., and a star quad of 12 AWG Inconel 600 sheathed silver, Sil Temp® overbraided and containing three mica tape layers with Inconel 600 overarmour, having an average wire O.D. of 0.170 in. and a lay of 6.80 in. It was noted during the lay-up of the 22 AWG copper cored cable that there was potentially severe relaxation or springback on releasing the twisting tension. It is anticipated that, due to the fact that the experimental wire has even more resilience, springback on cabled small gauges of the prototype wires may be severe.

Upon receipt of the overarmoured cable from Metex, it was found that all three samples were satisfactory for knit quality and continuity; cable flexibility was excellent. However, as anticipated, the 22 AWG copper conductor based cable showed appreciable springback.

3. Overarmour Wire

A 1000-hour exposure study was initiated to evaluate the thermal stability of the 36 AWG Inconel wire employed in the overarmour configuration.

Weight change, tensile strength deterioration, and elongation were determined at 250-hour increments. Tensile strengths were determined at a crosshead speed of 8 inches/minute. The results are shown in Table 30.

TABLE 30

THERMAL EXPOSURE OF 36 AWG INCONEL WIRE

(1000 hours at 1250°F, in air)

Exposure Time (hours)	Weight Change (%)	Tensile Strength (psi)	Elongation (%)
0	0	112,000	14.0
250	+0.16	85,800	12.5
500	+0.22	87,700	14.0
1000	+0.24	90,000	13.5

Although there was an initial deterioration of tensile strength, the material stabilized as exposure continued. Oxidative deterioration was evidenced by a low increase in weight. Elongation retention remained relatively constant. This evidence, coupled with subjective flexure tests which show no evidence of embrittlement, indicates that Inconel 600 in very thin section is considered satisfactory for use as the primary wire armour and cable armour.

4. Alternative Fastenings

As mentioned in Section 2, some difficulty was encountered when attempts were made to cable a star quad configuration of wires containing a 22 AWG copper conductor due to the resiliency of the conductor. As 22 AWG Inconel sheathed silver conductor is even more resilient than copper, considerable difficulty might be anticipated in cabling the experimental wire. An alternative approach, if cabling were proven to be non-feasible, would be to bundle the four individual wires, restraining them either through an Inconel 600 overknitted armour sheath, or alternatively a form of "strapping" applied at finite points along the cable bundle. One form of strapping potentially useful for this approach is the Velcro® configuration, from the Velcro Corporation([11]). This concept is a pair of fabricated tapes, one of which has loops attached which interlock with hooks attached to the other tape. A sample of stainless steel Velcro tape, with loops and hooks made from Inconel 600 series wire was obtained, and was evaluated for

long term stability at 1250°F. During conversations with the supplier it was noted that this tape could be obtained in an all Inconel configuration and that the present tapes are already in use for restraining wire and cable and attaching harnessing to airframes and bulkheads.

As with Inconel wire, the Velcro was monitored for weight change, and was also evaluated for peel strength. This latter was performed by locking the two tapes together over ∿ 3 inches by passing the tapes between the bench top and a roller, peeling back about 1/4", locking the two ends in the tensile tester jaws, and then monitoring resistance to release at a 12"/minute crosshead rate. The test was repeated four times, with the values averaged. The results are shown in Table 31.

After 1000 hours there was appreciable oxidative darkening, subjective stiffening and some buckling. Oxidative degradation, as monitored by weight change, was not excessive. There was some loss of peel strength which can be ascribed to the loss of heat set built into the Velcro hooks during fabrication which in turn reduces the resistance of the hook to disengage from the Inconel loop in the other tape. In summary, however, the performance of the Velcro configuration is encouraging and can be recommended in its present form, or an optional all Inconel form, as an alternative wire bundle fastening means, or as a bulkhead attachment means for the cable.

TABLE 31

EVALUATION OF THERMAL STABILITY OF
STAINLESS STEEL/INCONEL VELCRO CONFIGURATION FASTENING

Exposure Time (hours)	Weight Change (%)	Peel Resistance (lb)
0	0	0.35-0.45
250	+0.86	-
500	+1.34	-
1000	+1.81	0.25-0.45

SECTION IV

CONNECTOR DEVELOPMENT

A Introduction

This section covers Phase II development of the family of connectors designed to terminate the family of wires under concurrent development.

The overall objectives of the Phase II connector development program are:

1. To optimize the materials, processes and finishes that were created or established in Phase I([1]), and to identify methods for their commercial fabrication.

2. To achieve a satisfactory integration of the various connector/conductor components which would meet the requirements of the Phase II Statement of Work.

3. To fabricate seven mated pairs of prototype connectors attached to lengths of prototype star quad experimental cable, as follows: two pairs of connectors attached to lengths of experimental cable based on 12 AWG conductor and one pair of connectors attached to a corresponding length of experimental cable based on 14 AWG through 22 AWG conductor inclusive.

The performance requirements of the connector are laid out in the Statement of Work, as follows:

1. General Requirements

(i) Operating temperature range - -65°F to 1250°F.

(ii) Pressures corresponding to altitudes from sea level to 110,000 feet.

(iii) Humidity - 0 to 100%.

(iv) Vibration - 20 g, 10 to 2000 cps.

(v) Physical shock - 50 g, 11 ± 1 millisecond.

(vi) Design life at maximum temperature - 1000 hours.

(vii) Flight profile test - connector/wire assembly.

2. Specific Connector Performance Requirements

(i) Contact spacing and configuration to permit a contact-to-contact and contact-to-shell steady state working voltage of 250 volts rms, 400 Hz.

(ii) The insulation resistance between any two contacts and between the shell and any contact of mated connectors at high temperature shall be 500 megohms when tested in accordance with MIL-STD 202, Method 302, Test Condition B.

(iii) Definition of contact design and arrangement.

(iv) Mated contact resistance at temperature and aging shall be not greater than 120 millivolts.

(v) Definition of contact retention.

(vi) Definition of shell design including coupling method and cable support.

(vii) Resistance to ozone, hydraulic fluids, salt spray and kerosene (JP4 or JP5).

(viii) Corona resistance.

(ix) Compatibility with wire and insulation system developed under this program.

(x) Interfacial sealing of mated and individual connector halves to provide a splashproof seal.

3. Status of Connector Development at the End of Phase I

a. Materials

The following recommendations were made for the materials to be used in the high temperature connector component parts.

i) Shells and End Bells

a) Stainless steel types 316L or 304L (magnetic).
b) Titanium alloy ATL (5% Al-2.5% Sn) (non-magnetic).

Surface passivation recommended.

ii) Inserts

High purity alumina ceramic (99.5% Al_2O_3)

iii) Contacts

a) Socket - Pd/Ag 50:50.
b) Pin - Mo TZM alloy (0.5% Ti-0.08% Zr) or Pd/Ad 50:50.
c) Pin and socket - Inconel alloys 601, X750 or 617, and stainless steel alloys 304L, 316L or 317L.

All contacts should be plated.

iv) Contact Retention Springs

Inconel alloys 601, 617, 706 or 718.

v) Seals

a) Contacts-to-dielectric and dielectric-to-shell - Mo/Mn metallization plus gold braze.
b) Interfacial - metal-to-metal as presently employed in ITT Cannon Electric "HR" Series connectors.

vi) Contact Joining

Induction brazing or laser beam welding.

b. Connector Design

The decision was made to utilize an ITT Cannon Electric "HR" Series 14S shell configuration with four #16 contacts. This would allow production of prototypes which could be fabricated on standard industrial equipment, thus effecting cost savings and establishing general purpose standardization. Full advantage was to be taken of standard tooling, dielectric moulds and production performance results available within the ITT organization. An initial design configuration was established, as shown in Figure 17.

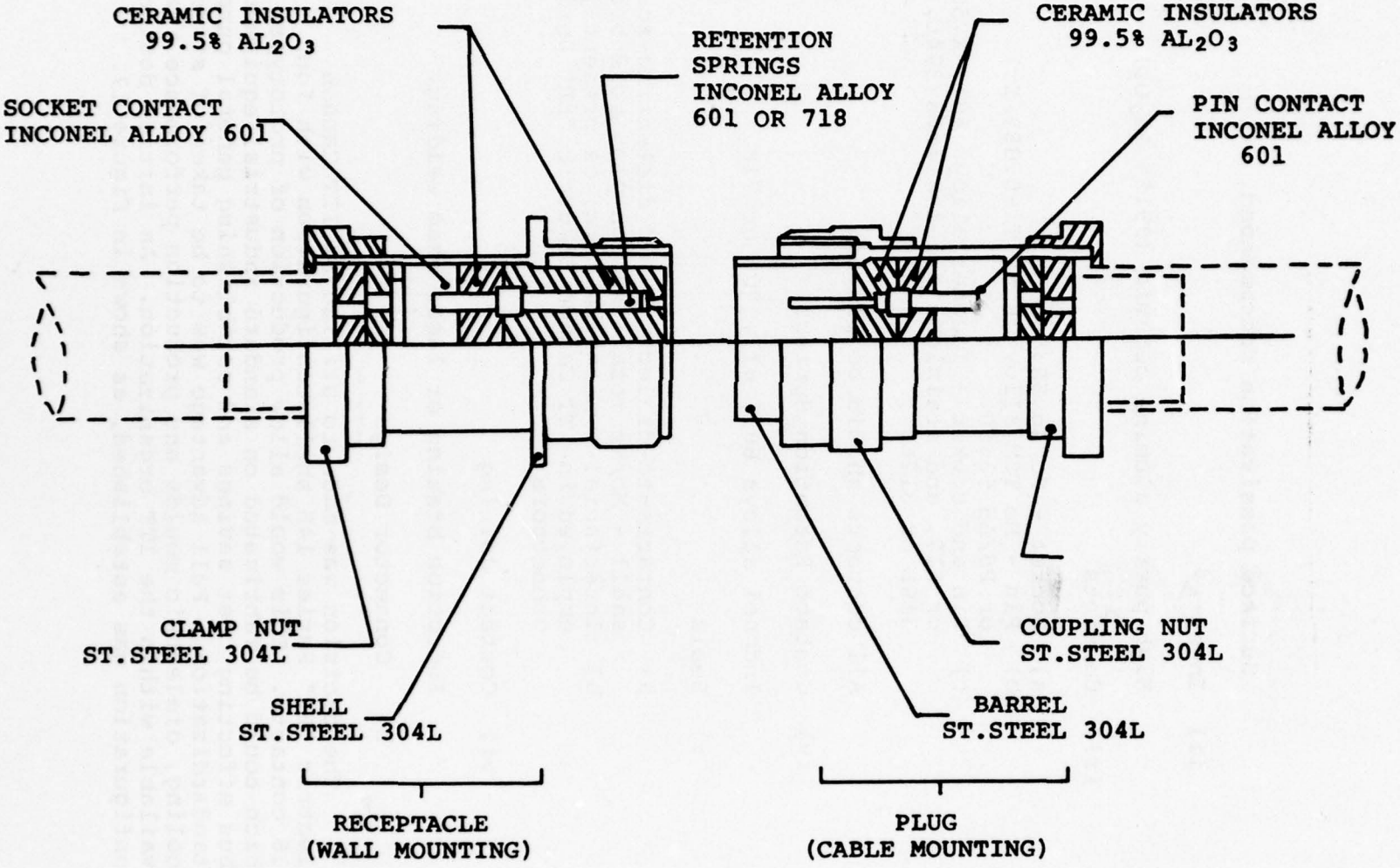


Figure 17. Proposed connector design for 1250°F operation.

B Connector Development, Phase II

Phase II development to date has necessitated modifications to some of the recommendations for materials outlined above as well as to the connector design approach. It is most convenient to present the work accomplished by dealing separately with each element of the proposed connector design and assembly.

1. Connector Design Optimization

Early in Phase II a proposed modified connector and cable interface design was established, as shown in Figures 18 to 20. which it was felt would optimize manufacture and assembly as well as meet the connector specification requirements. This design was intended to provide for contacts and conductors to be joined after the contacts were plated and the entire insert assembly fired into the shell.

Joining of contacts and conductors would be accomplished by utilizing an induction coil applied around the outside of the protruding contact brazing pots. The brazing process was facilitated by backing the end bell along the cable and removing the split spacer sleeve while the conductors remained positioned in the brazing pots.

After conductor/connector brazing, the cable would be clamped by threading the end bell on to the shell as shown in Figure 18. If required, additional individual conductor clamping could be accomplished by the use of the clamping discs also shown in Figure 18.

Once assembled, a splashproof metal-to-metal seal would be achieved between the end bell and the receptacle or barrel. Final sealing and locking would be accomplished by continuous fillet welding the end bell to the cable termination ferrule. Both pin and socket connectors would utilize the same end bell design.

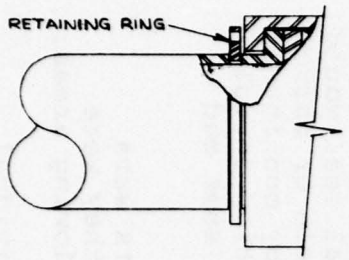
The first mated pair of prototype connectors were based on this design; however, after evaluation they were found to require further modification in the following areas:

a) A redesigned cable clamp to secure individual conductors. Incorporation of the original alternate clamping disc approach was predicated by the cable design; however, modifications were necessary to avoid damage by crushing of the relatively fragile insulation.

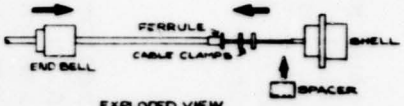
SYM	DCR/DCN	DESCRIPTION	DATE	APPROVAL

Figure 18

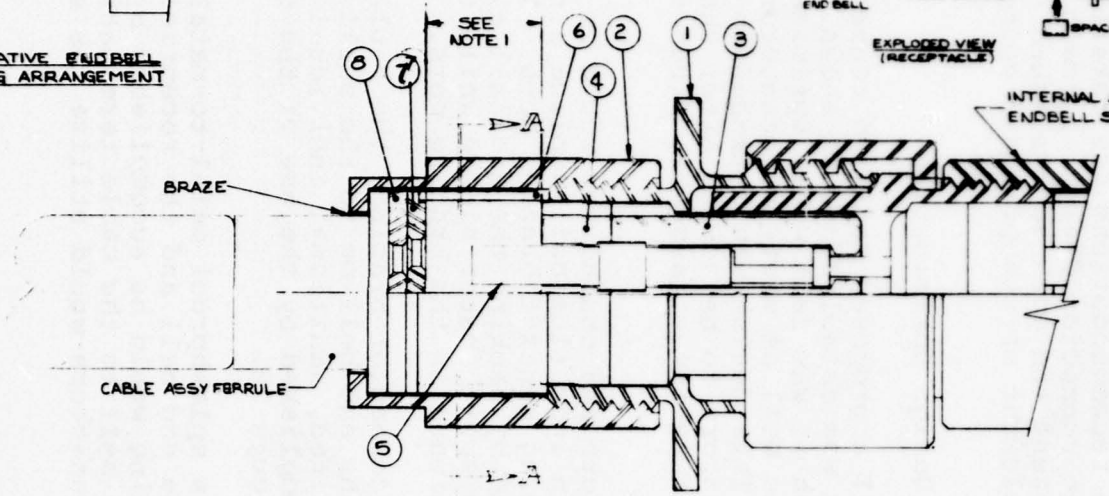
NOTE:
1. THIS DISTANCE IS DEPENDENT UPON SPACE REQUIREMENTS FOR THE BRAZING OF CONTACTS.
2. ASSY PROCEDURE
   a)-SLIDE END BELL OVER CABLE.
   b)-FEED CONDUCTORS THRU REAR CABLE CLAMP(IT.8) AND THRU FRONT CABLE CLAMP(IT.7) DRAW CLAMPS UP FIRMLY AGAINST CABLE FERRULE SHOULDER.
   c)-BRAZE CONDUCTORS TO CONTACTS.
   d)-INSERT SPACER (IT.6), AND TURN END BELL ON TO REAR OF SHELL (IT.1), TIGHTEN FIRMLY AND BRAZE REAR OF END BELL TO CABLE ASSY FERRULE. (OR SEE ALTERNATIVE.

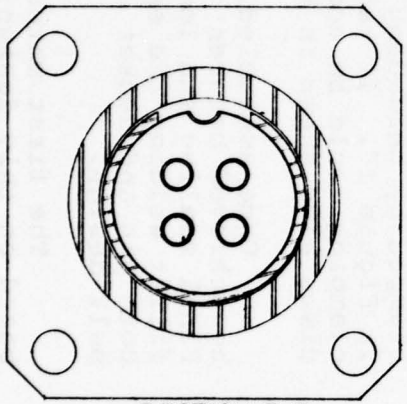


ALTERNATIVE ENDBELL LOCKING ARRANGEMENT



EXPLODED VIEW (RECEPTACLE)





ITEM	DESCRIPTION	QTY
8	CABLE CLAMP - REAR	1
7	CABLE CLAMP - FRONT	1
6	SPACER CABLE CLAMP	1
5	SOCKET CONTACT - NO.16	4
4	INSULATOR-SOCKET-REAR	1
3	INSULATOR-SOCKET FRONT	1
2	END BELL	1
1	SHELL RECEPTACLE - ASSY	1

EXCEPT WHERE NOTED:
1. REMOVE ALL BURRS & SHARP EDGES.
2. ✓ ALL OVER (PER MIL-STD-10).

DIMENSIONS ARE IN INCHES
TOLERANCES ON
DECIMALS: .X ± .020; .XX ± .010; .XXX ± .005
ANGLES: ± 1°-0'; .XX ± 30'

MATERIAL

FINISH

TREATMENT

DRAWN: E. Crouse DATE: 25 APR. 75
CHECKED
APPROVED: 25 APR. 75
APPROVED
APPROVED

ITT CANNON CONNECTORS (CANADA)
160 Bartley Drive, Toronto, Canada
A Division of ITT Canada Limited

APP
INTENDED USE: PROPOSAL

TITLE
PROPOSED HI-TEMPERATURE CONNECTOR MECHANICAL ARRGT.

CODE IDENT NO.
SIZE: C
DRAWING NUMBER: X830-1-000 FIGURE-18
SCALE
SHEET

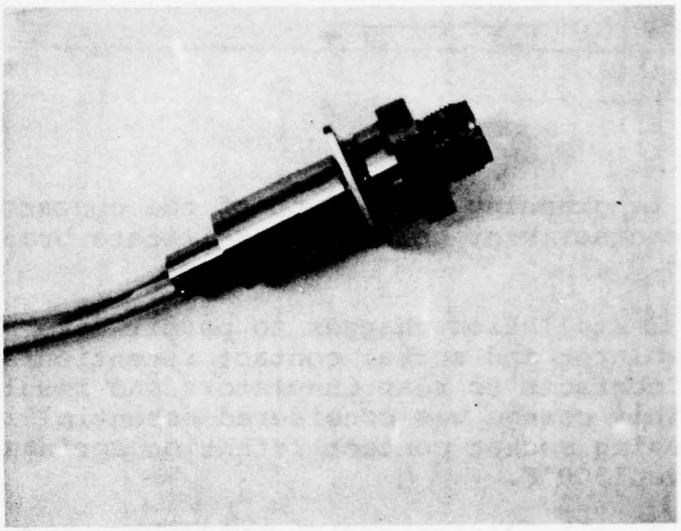


Figure 19

Prototype high temperature connector assembly *including simulated cable.*

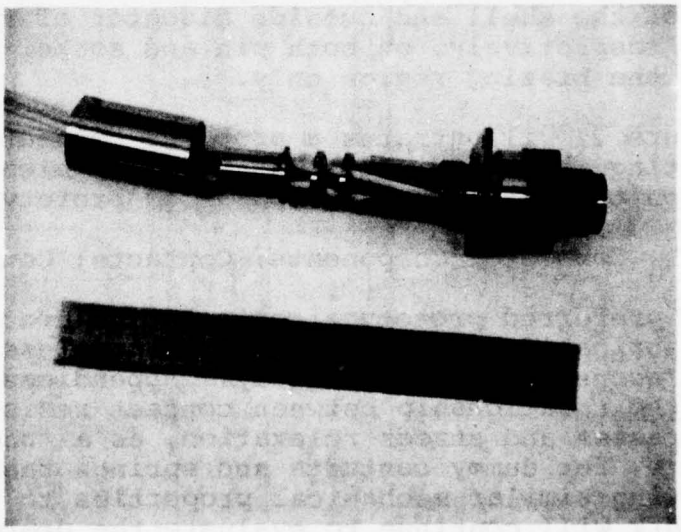


Figure 20

Exploded view of first prototype high temperature connector and cable.

b) Lengthening of the end of the contact braze pot to clear contact vent holes and facilitate brazing to conductors.

c) Installation changes to permit assembly of front socket insulator and socket contact retention springs after firing of contacts to rear insulators and insulators to shells. This change was considered essential in order to avoid exposing socket contact retention springs to temperatures above 1300°F.

d) Ferrule design modifications to conform to the characteristics of the Inconel outer armour on the prototype cable.

e) Incorporation of a receptacle on the cable clamp front component to contain ceramic potting compound. This is a potential approach to preventing ingress of moisture into the connector back shell by wicking of the insulation sheath.

f) Deletion of keys and keyways from the inside diameter of the shell and outside diameter of the rear insulator respectively, on both pin and socket connector halves in the brazing region only.

Figure 21 illustrates a proposed connector pair design incorporating these modifications, all of which were included on the next two mated pairs of prototype connectors.

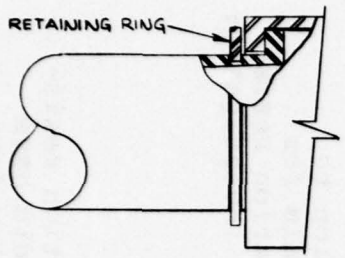
2. Connector Components; Contacts; Contact Springs

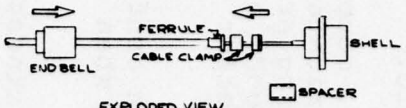
The preferred prototype contacts and contact springs will be fabricated from nickel plated stainless steel type 308L and Inconel X750 respectively. Appendices B, C and D describe the relationship between contact resistance, contact spring stresses and stress relaxation, as a function of temperature, for dummy contacts and springs fabricated from metals having similar mechanical properties to the prototypes. Work will continue to evaluate the prototypes, as received.

SYM	DCR/DCN	DESCRIPTION	DATE	APPROVAL
A		REVISED TO SHOW UP-DATED CLAMPING ARRGT. (RE-DRAWN)	28 JAN 76	
B		CONFIGURATION REVISED; FERRULE & CLAMP	16 FEB 76	

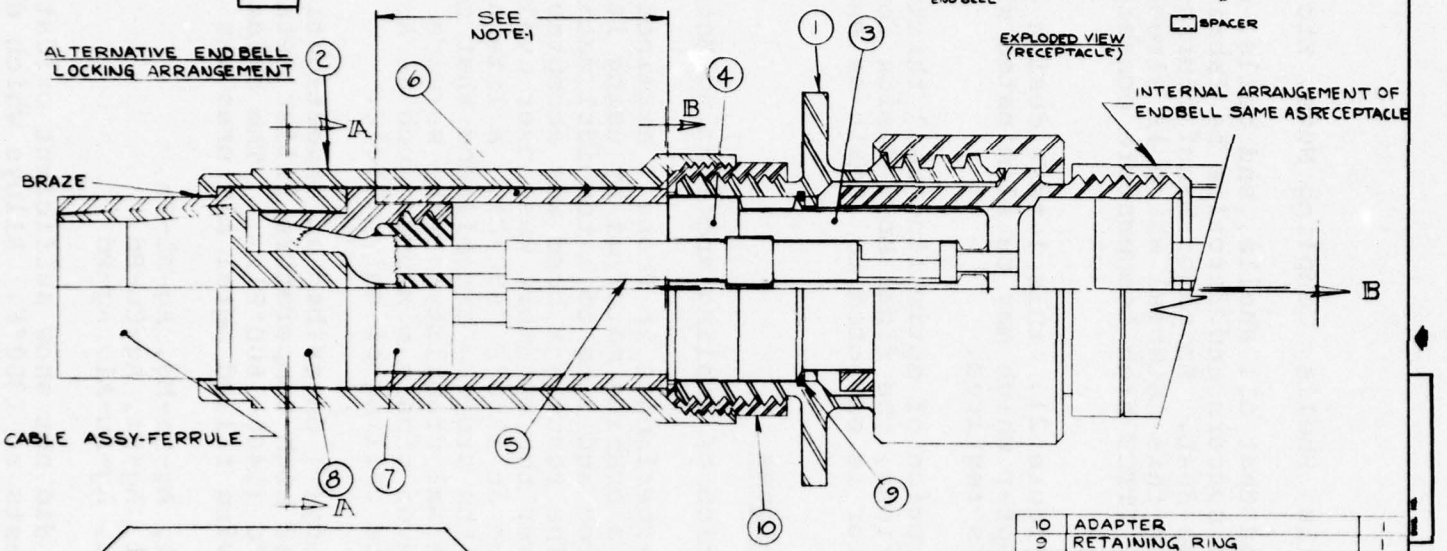
Figure 21

NOTE:
1. THIS DISTANCE IS DEPENDENT UPON SPACE REQUIREMENTS FOR THE BRAZING OF CONTACTS.
2. ASSY PROCEDURE
   a)-SLIDE END BELL OVER CABLE.
   b)-FEED CONDUCTORS THRU REAR CABLE CLAMP(IT.8) AND THRU FRONT CABLE CLAMP(IT.7) DRAW CLAMPS UP FIRMLY AGAINST CABLE FERRULE SHOULDER.
   C)-BRAZE CONDUCTORS TO CONTACTS.
   d)-INSERT SPACER (IT.6), AND TURN END BELL ON TO REAR OF SHELL (IT.1), TIGHTEN FIRMLY AND BRAZE REAR OF END BELL TO CABLE ASSY FERRULE. (OR SEE ALTERNATIVE.)

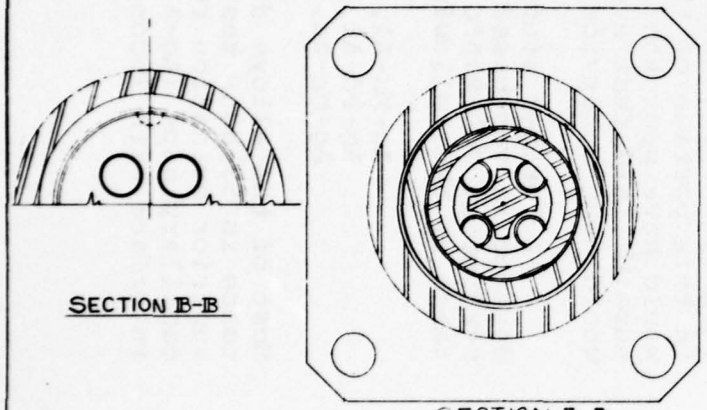




EXPLODED VIEW (RECEPTACLE)



ITEM	DESCRIPTION	QTY
10	ADAPTER	1
9	RETAINING RING	1
8	CABLE CLAMP - REAR	1
7	CABLE CLAMP - FRONT	1
6	SPACER CABLE CLAMP	1
5	SOCKET CONTACT - NO16	4
4	INSULATOR-SOCKET-REAR	1
3	INSULATOR-SOCKET FRONT	1
2	END BELL	1
1	SHELL RECEPTACLE - ASSY	1



EXCEPT WHERE NOTED:
1. REMOVE ALL BURRS & SHARP EDGES.
2. ✓ ALL OVER (PER MIL-STD-10).

DIMENSIONS ARE IN INCHES
TOLERANCES ON DECIMALS: X ± .030, XX ± .010, XXX ± .005
TOLERANCES ON ANGLES: X° ± 1°-0', X'X' ± 30'

MATERIAL
FINISH
TREATMENT

DRAWN E. CROUSE DATE 28 JAN 76
CHECKED
APPROVED 29 JAN 76
APPROVED
APPROVED

ITT GREMAR CONNECTORS (CANADA)
100 Bentley Drive, Toronto, Canada
A Division of ITT Canada Limited

REF
INTENDED USE PROPOSAL

TITLE
PROPOSED HI-TEMPERATURE CONNECTOR MECHANICAL ARRGT.

CODE IDENT NO.
SIZE C
DRAWING NUMBER X830-1-000 - REV. B
FIGURE - 21
SCALE
SHEET

3. End Bells, Shells, Coupling Nuts, Etc.

It is proposed that all shells, end bells, coupling nuts, cable clamps, spacers and ferrules be fabricated from stainless steel type 304L. No major manufacturing problems are anticipated with this material which is already extensively used on many ITTCE high temperature connector products.

As shown in Figure 21, this latest design configuration employs an adapter which may be eliminated when a larger shell size is required.

With the exception of optimizing wall thicknesses and cable clamp proportions, the final body design for the high temperature connector is expected to remain as shown in Figure 21.

C Assembly Techniques

1. Evaluation of Sealing and Brazing Materials

Preliminary experiments in Phase I examined the efficacy of using copper as a brazing material by using it to fasten a stainless steel rod and ring to a toroidal metallized Al_2O_3 insulator. The resultant bond was acceptably strong and complete, however the assembly was never evaluated for oxidation resistance at 1250°F (680°C). A literature review in this portion of the program established that copper alone would have poor thermal stability at the service temperature[12], and that even binary mixtures such as Ag-28% Cu would have a service ceiling of only ∿750°C.

A further study[13] described an investigation to develop silver-based high temperature filler metals for service at 760-870°C (1400-1600°F). The oxidation resistance of the following filled metal mixtures was investigated:

Ag-Mn-Al, Ag-Mn-Mg, Ag-Al-Zn,
Ag-Pd-Al, Ag-Si, Ag-Cu-Pd,
Ag-Mn-Zn, Ag-Au-Al, Ag-Pd

Most of the alloys did not show sufficient oxidation resistance in 55-hour tests at 1300°F. Alloys which did have superior oxidation resistance either would not flow into capillary gaps (Ag-Al) or formed brittle compounds at the interface with Inconel (Ag-Si).

Alloys based on the Ag-Cu eutectic, with Pd alloy additions to increase the melting point and other alloy additions to decrease oxidation (i.e. Al, Cr, Si, Mg and Be), showed significant failure by scaling after only 16 hours at 1400°F.

Sistare and McDonald[13] also investigated Cu-Ni-Mn and Cu-Al-Sn alloys. The former were not sufficiently oxidation resistant, and the latter showed poor strength (1000 psi at 1200°F), as well as requiring a flux to enhance flow. The use of a flux is highly undesirable for the proposed connectors because of the difficulty of removing it after brazing.

As indicated above, no silver-based brazing alloy has been reported which retains its full strength and oxidation resistance up to 1250°F, the operating temperature for the present system. However, it should be realized that for this application the high temperature strength of the brazed joint is of lesser importance than its oxidation resistance and its retention of plasticity at high temperature so it can accommodate expansion differences between the metallic and ceramic parts of the connector. Any silver-based brazing alloy containing a less noble metal such as Mn would certainly disqualify itself for this purpose as internal oxidation of the less noble metal would soon destroy the plasticity of the brazed joint[14]. Another approach would be to braze with pure silver, however, if, during brazing, the silver dissolved any less noble element, i.e. Mo from the metallization, local internal oxidation would again result. Pure silver would yield relatively weak joints at 1250°F, as this is close to its melting point of 1760°F. This could be overcome by alloying with Pd or Au, to raise the melting point, however, this too would have little effect on the inherent high oxygen solubility of silver which causes internal oxidation. A further adverse property of silver is its high vapour pressure at elevated temperatures. The vapour pressure of brazing metals is significant as high vaporization rates may lead to deposition of metallic films on the ceramic insulator which would reduce the resistivity. A second disadvantage is the relatively severe loss of metal during brazing.

As may be seen in Table 32, the vapour pressure of silver is 56 times greater than that of gold, while the vapour pressure of nickel is significantly lower than that of any of the other metals listed. Thus it was concluded that, in descending order of acceptability, the major metal component of potentially useful brazing alloy would be nickel, gold and silver.

TABLE 32

COMPARATIVE VAPOUR PRESSURES OF SELECTED METALS

(Test Temperature 1100°C)

Metal	Vapour Pressure (mm Hg)
Ag	7.23×10^{-3}
Cu	2.50×10^{-4}
Au	1.29×10^{-4}
Ni	9.32×10^{-7}

To substantiate this reasoning at the program service temperature of 680°C, a sample of a silver/copper/tin alloy was exposed in air at 1100°F for 65 hours, cooled, visually examined, mounted, and microscopically examined. As may be seen in Figure 22, the sample was covered in grey-blue oxide, suggestive of copper oxide. As well, the copper phase had oxidized internally to an average depth of ∿200 μm (0.008 inch). Under these circumstances brazes containing even a low proportion of copper were disqualified for use in the present application.

2. Oxidation Resistance of Nickel-Based Brazing Alloys

To evaluate the merits of nickel-based brazing alloys, specimens of 304L stainless steel were brazed together under vacuum using three commercial nickel-based alloys:

BNi - 1 (14% Cr, 3% B, 4.5% Si, 4.5% Fe, 0.7% C, 73.3% Ni)

BNi - 4 (2% B, 3.5% Si, 0.06% C, 94.44% Ni)

BNi - 5 (19% Cr, 10.2% Si, 70.8% Ni)

The brazed joints were then exposed in air at 1255°F for 960 hours. Sample cross-sections of the brazes after this test are shown in Figure 23, which also illustrates that all the materials displayed very good oxidation resistance.

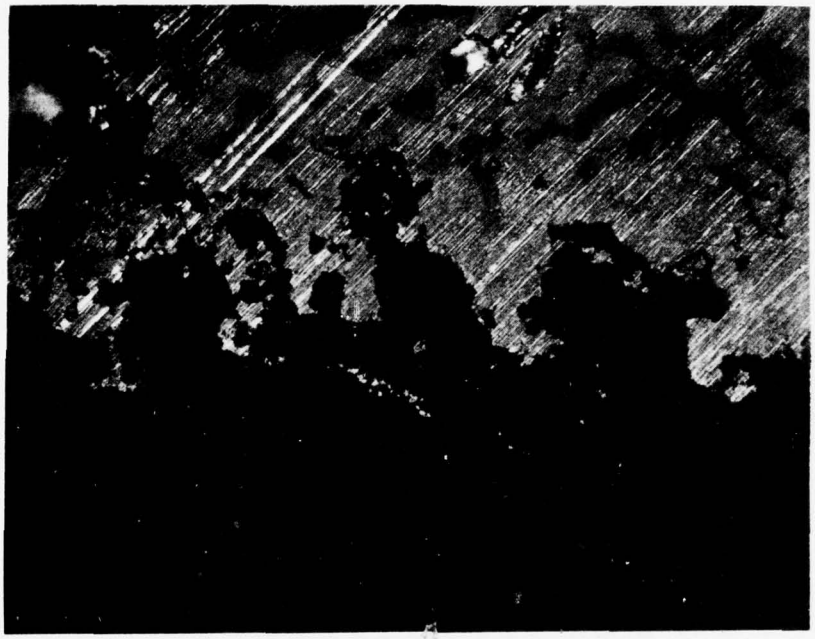
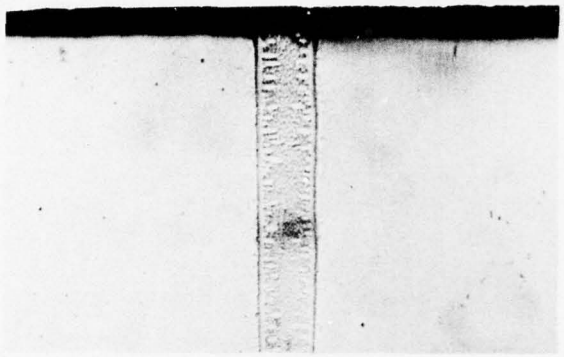
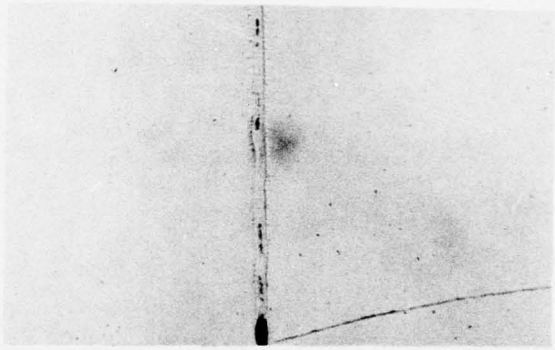


Figure 22

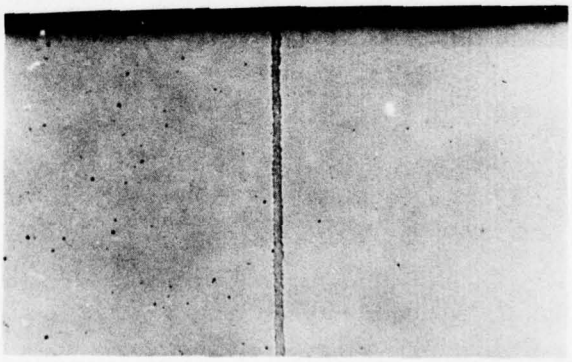
Metallographic section of the surface of a Ag-Cu-Sn alloy after exposure. (200X)



(a) BNi-1 Braze



(b) BNi-4 Braze



(c) BNi-5 Braze

Figure 23

Sections through braze and 304 stainless steel. (At or very close to specimen surfaces, after 960 hrs at 680°C (1255°F) in air). 37X

The above Ni-based alloys have higher melting points than Ag or Cu-based brazing alloys. Lower melting temperature Ni-based alloys are commercially available, but were not available during this study. Work will continue to evaluate these to determine their thermal resistance and compatibility with the other components during service.

a. Au-Based Alloys

Au-based brazing alloys appear to be ideal from many aspects, but are very expensive. Au-Ni alloys have been reported[13] to have excellent oxidation resistance, good wetting ability, and ductile deformation behaviour. In Phase I an Au-Ni-In alloy was employed to braze Pd-coated and Inconel-coated silver wire to Mo and noble metal contacts. Small quantities of both Au-18 Ni-5 In were also prepared. As the two alloys have different melting temperatures, one could be used subsequent to brazing with the other. The In is completely dissolved and should not significantly affect the properties of the basic Au-Ni alloy.

Details of brazing tests using Au-Ni alloys are outlined in Appendix E.

3. Metallization of Alumina Inserts

Two processes are known to deposit molybdenum coatings on alumina, but little has been reported on the relative adhesion of the coatings produced. The present commercial method uses molybdenum-manganese pastes, fired in wet hydrogen or wet dissociated ammonia at 1400-1620°C[15]. Apparently the water oxidizes the manganese, which then forms a glass with the alumina, surrounding metallic molybdenum particles[16]. This requires close control of the H_2O content in the gas. Another method[16] involves making a glass of MnO-SiO_2-Al_2O_3 by melting the three components together in a nitrogen atmosphere. A mixture of this glass and molybdenum particles can then be fired onto an alumina substrate in a dry hydrogen atmosphere, making the firing procedure simpler.

A few simple high purity Al_2O_3 shapes, (insulating beads), were metallized using a commercial MnO/Mo mixture[15]. The metallizing treatment was carried out at 1100°C in a 96/4 N_2/H_2 atmosphere. This treatment yielded a well-adhering layer which accepted a nickel plate. Although the initial results were encouraging, further experiments, outlined in Appendix E, were required to determine both the quality of the metallized layers and the subsequently applied nickel plate.

Prototype rear socket insulators and rear pin insulators were metallized as described in Appendix F. An Inconel 600 tube was employed in the closed system dictating a maximum metallizing temperature of ∿1250°C, which in turn required a relatively long treatment time of 4.5 hours. Although the manufacturer([17]) recommends a firing temperature between 1425°C and 1600°C, it was found that satisfactory metallization could be achieved at 1250°C. The typical layer, about 0.001 inch thick, was very adherent and accepted nickel plating; the morphology was the same as that in a recent publication([18]) and the metallized, plated alumina beads were readily brazed to stainless steel, yielding a metallurgically sound joint.

Considering the morphology, Figure 24 illustrates the surface of one such metallized layer. The structure shown is in all respects the same as that obtained by firing at 1600°C([19]). The transverse microsection of the metallized layer, Figure 25, has the same thickness and phase distribution as those shown for a layer produced at higher temperatures([20]).

It was still felt, however, that the preferred method of metallization should employ temperatures between 1450°C and 1600°C.

Further experiments were undertaken to metallize insulators at higher temperatures. Alumina metallization tubes were substituted for the previously used Inconel tube to contain the mixed N_2/H_2 atmosphere. The tube was fitted with a water cooled end fitting which would allow manipulation of a fused alumina specimen boat in the furnace.

The first attempts at metallization at 1450°C were unsuccessful. The furnace was raised to temperature with flowing N_2/4% H_2, the boat carrying the prepainted insulators inserted into the hot zone, left for a minimum of 30 minutes and then withdrawn into a relatively cooler zone outside the furnace. In some cases the boat or the manipulator wire broke, making it impossible to remove the specimens. On one occasion the exit of the boat from the furnace fractured the alumina tube due to thermal shock. Most importantly, however, no evidence of metallization was evident on the alumina. It was finally found that acceptable metallization occurred when pieces of chromium were inserted into the hot zone. It is postulated that the chromium acted to scavenge excess O_2 in the atmosphere.

The gas train was then modified to reduce O_2 by passing the gas mixture over copper turnings at 550°C, followed by passage through calcium sulfate before entering the metallization furnace. Despite this approach, chromium coupons inserted into the furnace still showed evidence of heavy oxidation.

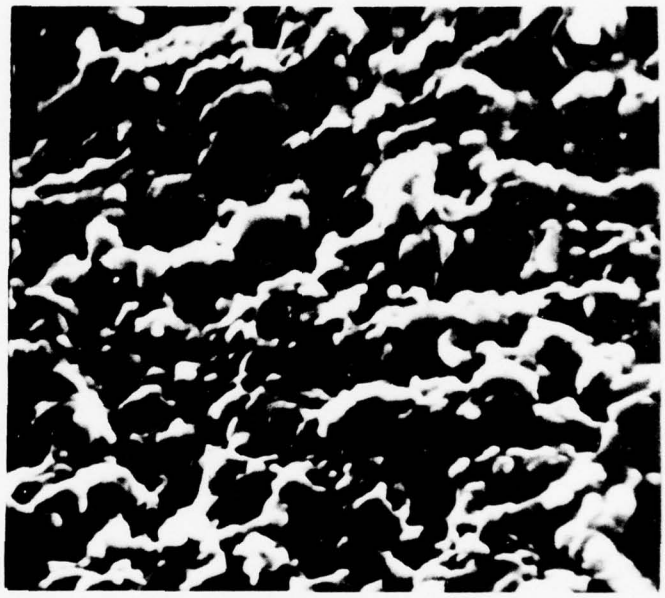


Figure 24

Morphology of metallized Mo layer on 99.5% Al_2O_3

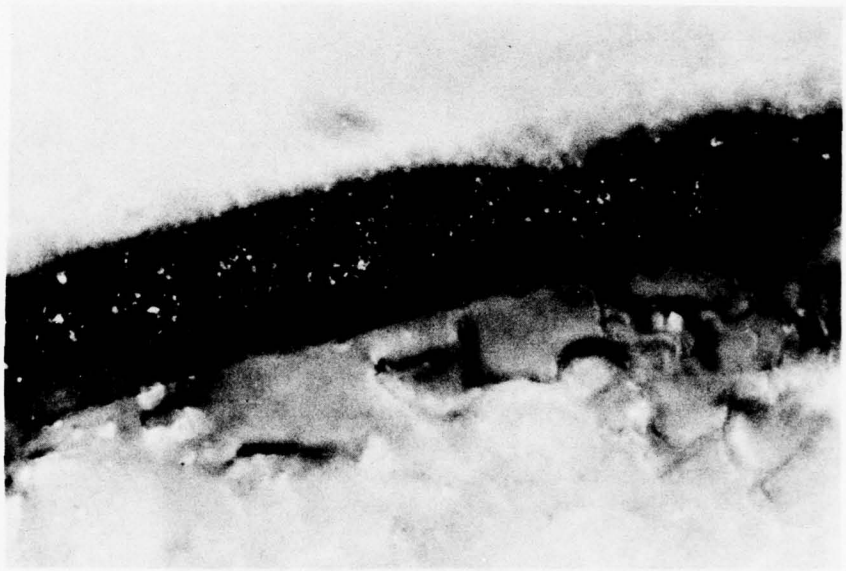


Figure 25

Cross-section of metallized Mo layer on 99,5% purity Al_2O_3. (The dark phase in the metallized layer is molybdenum polarized light). 600X

The preferred sequence of metallization was established as follows:

1) Prepainted insulators are loaded in an alumina boat, inserted into the furnace cool zone, and the furnace end plate inserted.

2) The furnace is heated to 1450°C. During heating, the tube is evacuated and purged with the N_2/H_2 gas mixture.

3) At 1450°C, the boat with specimens is inserted into the hot zone and held for 30 minutes.

4) The furnace temperature is then slowly reduced, allowing the specimens and boat to cool in situ.

The process successfully and reproducibly yields strong metallized layers with good conductivity for metal plating. Full details of technique are given in Appendix F.

4. Brazing of Metallized Dielectrics to Stainless Steel Sleeves/Contacts

According to basic considerations[20], the oxygen partial pressure developed in the apparatus used for gold alloy brazing of conductors to contacts is not low enough to allow for fluxless nickel brazing of metallized Al_2O_3 to stainless steel or Inconel sleeves. Despite this, however, as considerable savings could be effected by using such a simple apparatus, experiments were performed in this equipment to braze metallized and nickel plated Al_2O_3 insulating beads to nickel plated sleeves of 304 stainless steel. Figure 26 illustrates a cross-section of the insulator/sleeve arrangement, which used a commercial Ni-Cr-Si brazing powder (Nicrobraz N-30) as a filler.

In several of the initial brazing experiments, using different argon purging times, brazing material did not flow into the brazing gap, but simply changed colour, indicating surface oxidation of the powder particles. These experiments established that the brazing of the metallized dielectric to connector shells and contacts with nickel-based braze materials would have to be performed either in vacuum or in a reducing gas atmosphere.

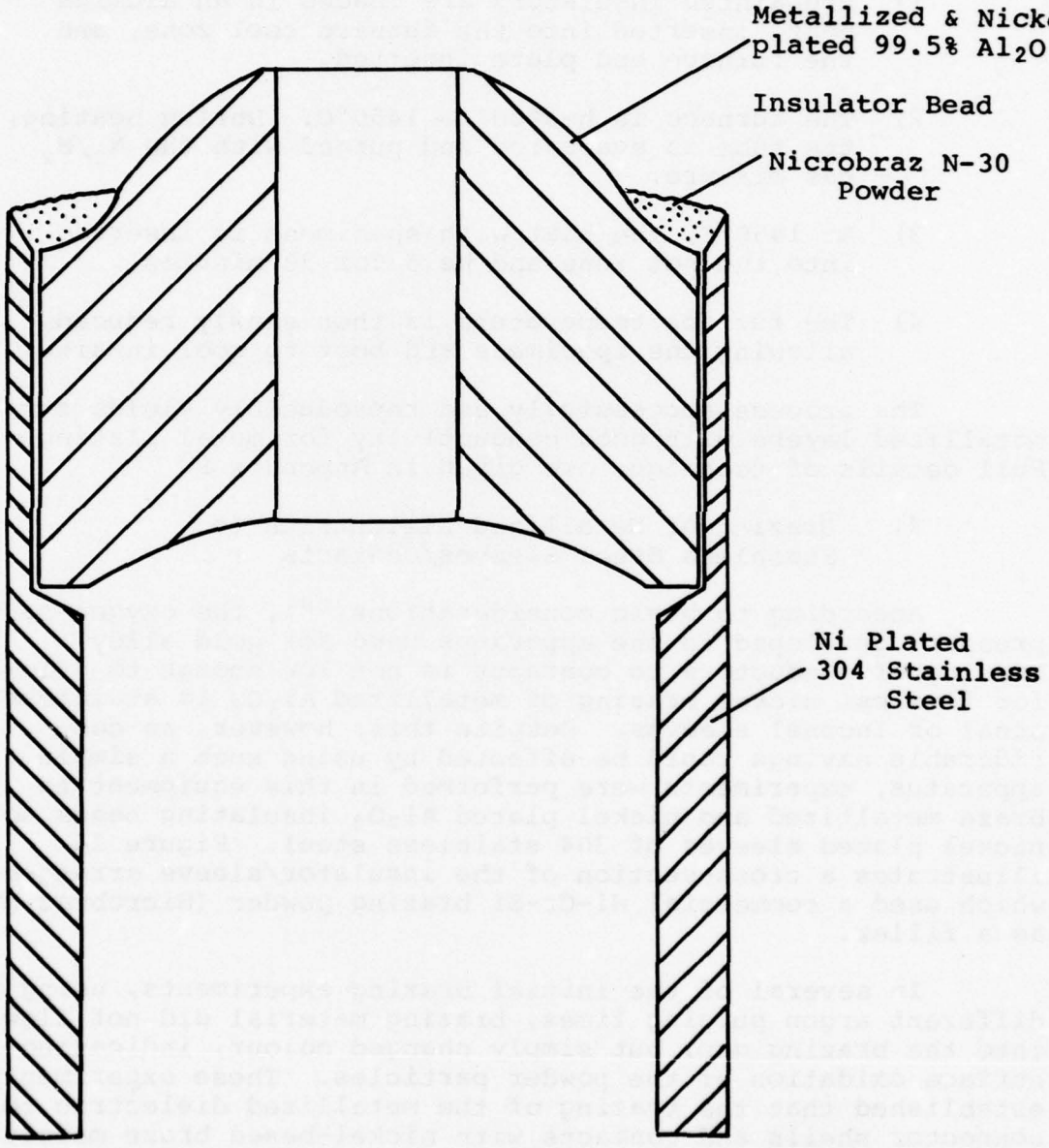


Figure 26

Cross-section of Insulator/Sleeve Arrangement.

Further brazing experiments were performed to test this hypothesis, using a closed system held under a vacuum of $<5 \times 10^{-5}$ mm Hg. The assembly illustrated in Figure 26 was placed in a ceramic coated graphite susceptor and inserted into an induction coil. The sample was raised slowly ($\sim$5 minutes) to brazing temperature where visual observation of the flow of the braze material indicated the end of the process. The samples were then cooled for examination.

Nicrobraz N-30 showed excellent flow characteristics and wetting. It was also evident from these experiments that a very small clearance between the insulator and metal is desirable, with a gap of between 0.001-0.002 inch estimated to be optimal. It was also found that liquid braze alloy which comes in contact with non-metallized alumina does not wet or react with alumina and thus is easily removed.

Figure 27 illustrates a cross-section of a brazed insulator/sleeve assembly, which shows that the brazed joint is tight and smooth. A micrograph of the brazed joint is shown in Figure 28. It can be seen that excellent bonding of the braze metal both to the stainless steel and metallized alumina was achieved.

Figure 29 shows this same Al_2O_3/N-30 braze metal interface at higher magnification. The excellent visual adhesion of the glass phase is evidence that the metallizing treatment is effective.

Further studies attempted to simultaneously braze a 0.417 inch diameter metallized Al_2O_3 disc to two penetrating Inconel 601 contacts and to a 304L stainless steel sleeve. This configuration, which simulates a connector, is diagrammed in Figure 30. The Al_2O_3 disc, which had previously been nickel plated using $\sim$2V for 20 minutes, had a layer of Nicrobraz N-30 powder spread around the pins and the circumference of the disc, and the configuration was then brazed in vacuum. After brazing, the various parts were joined, but penetration of the braze metal was not uniform. This may be partly due to the contacts and sleeve being at the same height as the alumina disc (Figure 30). allowing the Nicrobraz N-30 to flow over their free surfaces, rather than into the gap. Further experiments will use stop-off materials to limit the flow paths of braze material.

The brazed unit was then sectioned with a diamond saw for examination. Because of the hardness of the alumina disc, this sectioning required two days, and during the operation one of the Inconel 601 contacts broke out of the

Figure 27

Polished cross-section of an alumina insulating bead brazed into a 304 Stainless Steel sleeve. 24X

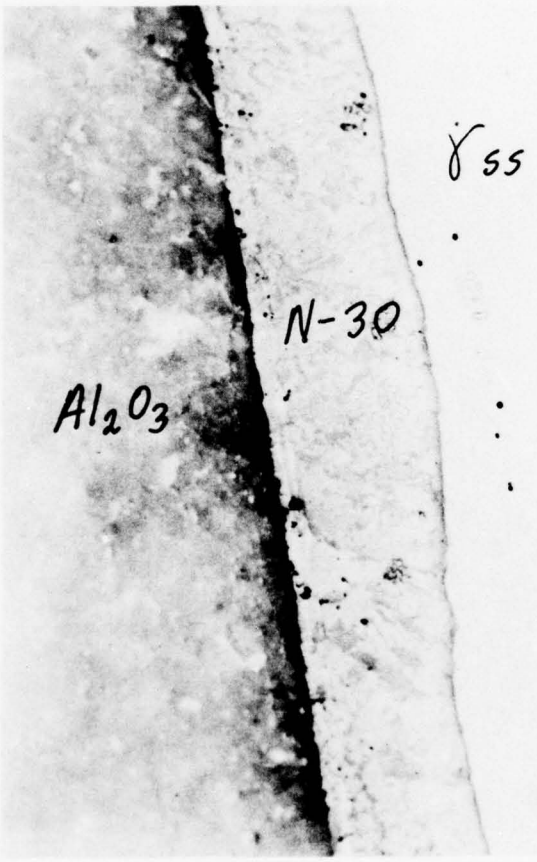


Figure 28

Brazed joint between metallized Al_2O_3 and stainless steel. 120X

Figure 29

Al_2O_3/Braze metal interface. (Note the excellent bonding to the glass phase layer). 300X

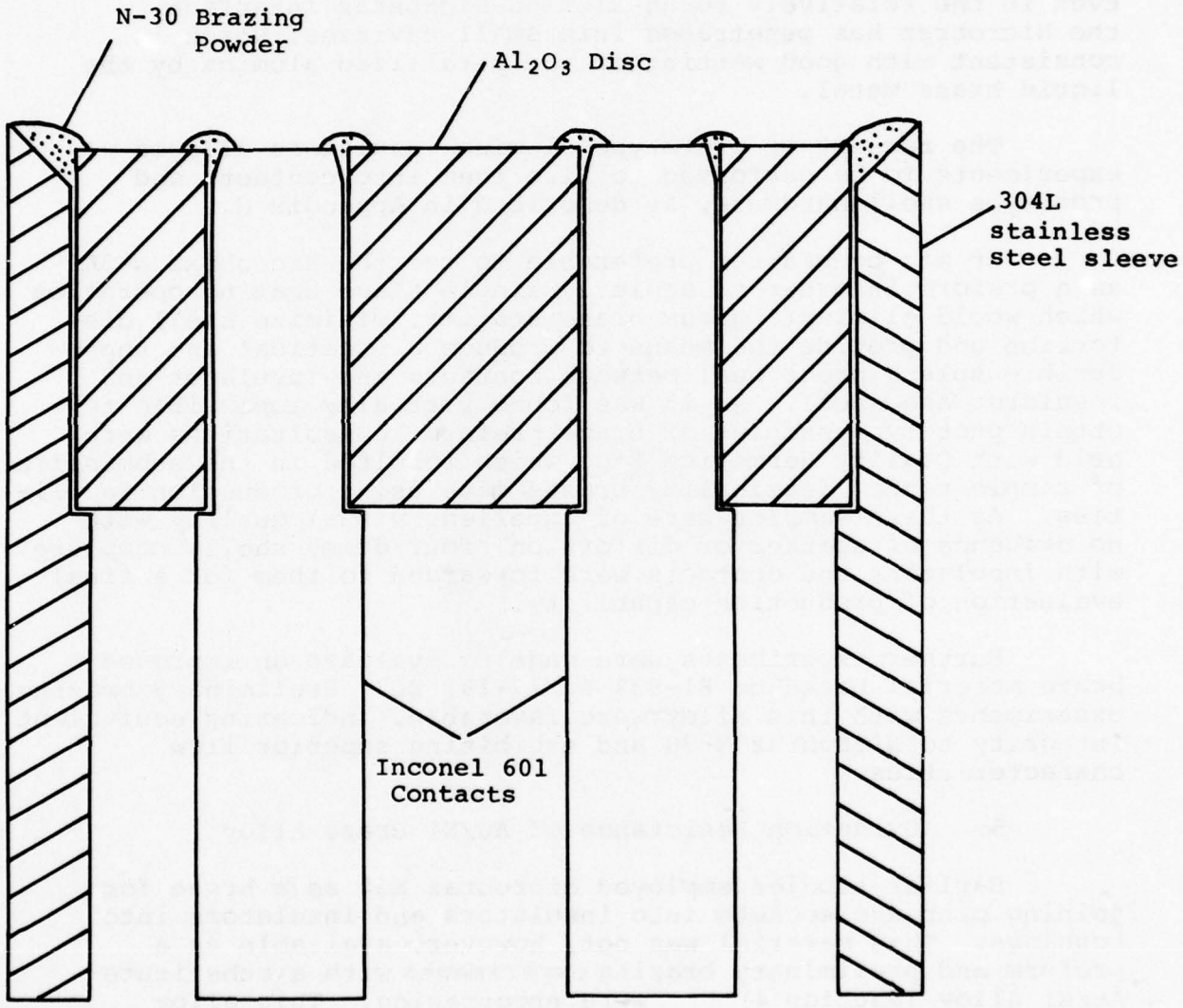


Figure 30

Cross-section of sleeve-contact-disc specimen
before brazing

unit, but the sleeve-disc braze remained intact. Considering that the metallizing process produces a glassy phase, the constant vibration and applied stresses during the sectioning process are a severe test of the brazed joints.

Sections of the two types of brazed joint are shown in Figures 31 and 32. In both cases it can be seen that the Nicrobraz N-30 braze material has flowed uniformly into the gap, and has wetted perfectly the metal-base side of the joint. Even in the relatively rough alumina-Nicrobraz interface, the Nicrobraz has penetrated into small cavities, which is consistent with good wetting of the metallized alumina by the liquid braze metal.

The receipt of prototype alumina insulators allowed experiments to be performed to fire them into contacts and prototype shell hardware, as described in Appendix G.

It was considered preferable to use the Nicrobraz N-30 as a preform in order to achieve a single stage brazing operation which would eliminate gross braze spatter, minimize shell distortion and provide the means to produce a practical and reproducible splash proof seal between contacts and insulator and insulator and shell. As it was found virtually impossible to obtain prototype samples of braze preform, consultations were held with Quality Hermetics Ltd. which resulted in the submission of sample random feasibility brazes made using production facilities. As these samples were of excellent visual quality with no evidence of spatter or distortion, four dummy shells complete with insulators and contacts were forwarded to them for a final evaluation of production capability.

Further experiments were made to evaluate an improved braze material based on 81-83% Au/17-19% Ni. Preliminary brazing experiments with this alloy were favorable, indicating equivalent integrity to Nicrobraz N-30 and exhibiting superior flow characteristics.

5. Oxidation Resistance of Au/Ni Braze Alloy

Earlier studies employed Nicrobraz N30 as a braze for joining pins and sockets into insulators and insulators into housings. This material was not, however, available as a preform and preliminary brazing experiments with a substitute Au/Ni alloy (Section 4) were encouraging. This alloy is available as wire which can be drawn and shaped into brazing preforms having good oxidation resistance and tensile properties. Metallized and nickel plated alumina insulators were successfully joined to a stainless steel 304L shell and stainless steel 308L contacts. This assembly was then held in air at 710°C for 335 hours. It was sectioned using a diamond saw, polished and examined for degradation and oxidation.

Figure 31

Section of metallized alumina disc-304L stainless steel sleeve brazed joint, as brazed. 213X

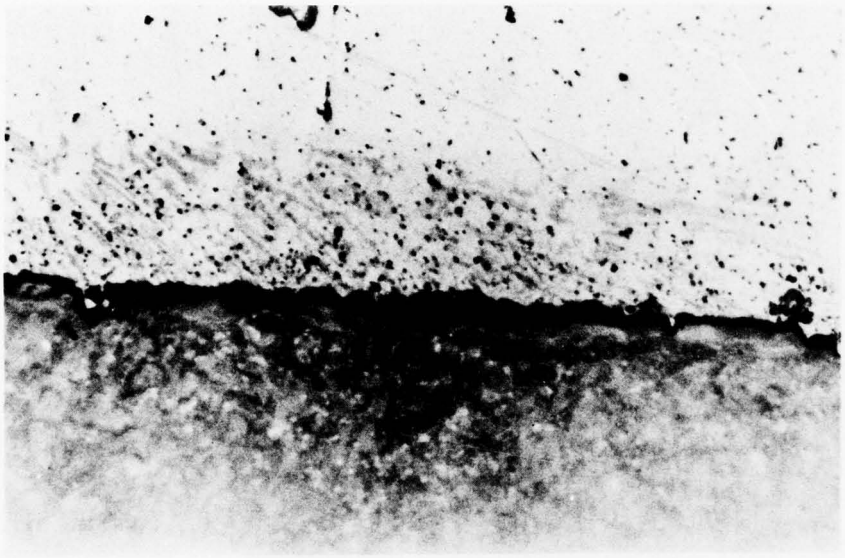


Figure 32

Section of metallized alumina disc-601 Inconel contact brazed joint, as brazed. 213X

A typical section through the brazed joint is shown in Figure 33. No evidence of oxidation can be seen, nor is there evidence of grain boundary diffusion. Since both oxidation rates and diffusion are generally exponential functions of temperature, the use of an exposure temperature 30°C above the maximum recommended operating temperature of 680°C is deemed adequate to ensure the usefulness of this alloy for 1000 hours at the program service temperature of 680°C.

6. High Temperature Oxidation Studies of Plating and Brazing

Plated and unplated Inconel 601 and 308L stainless steel pin contacts were placed in a furnace at 680°C to examine their relative rates of oxidation, and the integrity of the Ni plating. After approximately 100 hours at 680°C, the furnace overheated due to a controller malfunction, reaching approximately 900°C. The experiment was then terminated. Examination of the specimens showed that the nickel plating was flaking off both types of pin contact, but uniform and approximately equal oxidation was evident on the unplated pins.

Further experiments were then performed to examine the performance of the following pin combinations at high temperature: unplated Inconel 601, nickel plated Inconel 601, unplated stainless steel 307, and nickel plated stainless steel 308. Samples in triplicate were held for 1010 hours at 680°C in air. After exposure all samples were dark grey. The most uniform coloration was on the nickel plated specimens (Figure 34), with the unplated Inconel specimens showing some patches of lighter colour. None of the latter samples showed deep oxidation or flaking oxide scale. In contrast to the results of the first oxidation run, no flaking of the nickel plating was observed. However, the nickel did not prevent the contacts from acquiring a thin oxide layer as evidenced by discoloration. Because the nickel coated contacts underwent a rather uniform colour change,it could be presumed that they also formed a uniform oxide layer. This presumption is reasonable as for thin oxide coatings colour

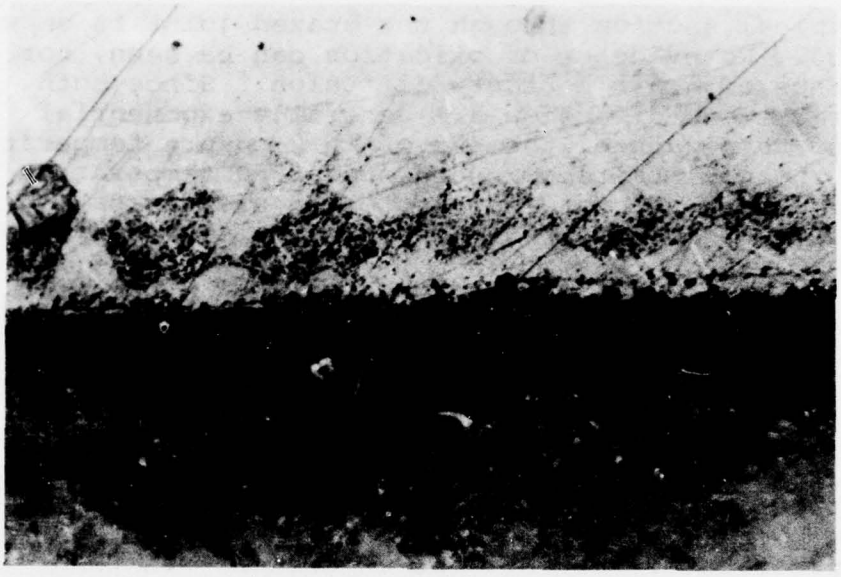


Figure 33

Dummy Au-Ni Braze Aged at 710° C for 335 hours.
200x

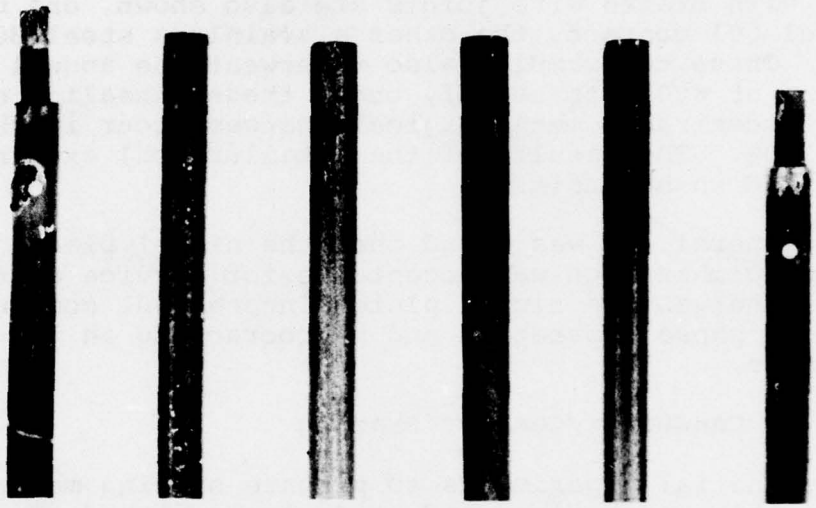


Figure 34

Appearance of contacts exposed to air for 1010 hrs
680°C

From left to right:

Ni plated 308 Stainless Steel
Ni plated 308 Stainless Steel
unplated 308 Stainless Steel
unplated Inconel 601
Ni plated Inconel 601
Ni plated Inconel 601

uniformity is generally a good indication of oxide uniformity. It is therefore advisable that nickel plating of contacts become part of the production of connectors.

At the extreme left and right sides of Figure 34, contacts with brazed wire joints are also shown, one being an Inconel 601 contact, the other a stainless steel 308L contact. These two samples also underwent the anneal of 1010 hours at 680°C to see if, under these annealing conditions, undesirable metallurgical changes occur in the braze joints. The results of the metallurgical examination are reported in Appendix H.

In general, it was found that the nickel plated stainless steel combination was acceptable for service at 680°C (1250°F), whereas the nickel plated Inconel 601 combination showed some phase separation and microcracking in the area of the braze.

7. Conductor/Contact Brazing

The initial experiments to prepare brazing materials, develop plating techniques and to devise a method of induction brazing of Inconel clad silver conductors into contact pins have been detailed in Appendix E. However, the conductor/contact pin brazing studies have been continued to optimize this process.

As shown in Figure 35, brazing methods now yield properly filled brazed joints with a smooth fillet between conductor and contact pin. This contact pin was a commercially Ni-plated Inconel contact with a 0.085" ID bore. The brazing material is the ternary Au-Ni-In alloy described previously. The induction brazing was performed in a high purity argon atmosphere in the apparatus described in Appendix E. The quartz tube was purged with argon for five minutes before brazing.

Examination of brazed joints at high magnifications revealed some regions of unsound interface between the brazing material and Inconel. Such a region is shown in Figure 36. It appears that these flaws are areas of separation between the braze metal and the Inconel, i.e. microcracks. It was found that these microcracks developed only in the contact-pin braze metal interface and never in the conductor-braze metal interface. It is suspected that this microcracking is due to a poorly adherent nickel plating in the inside bore of the contact pin. This reasoning is reinforced by the observation that the cracking

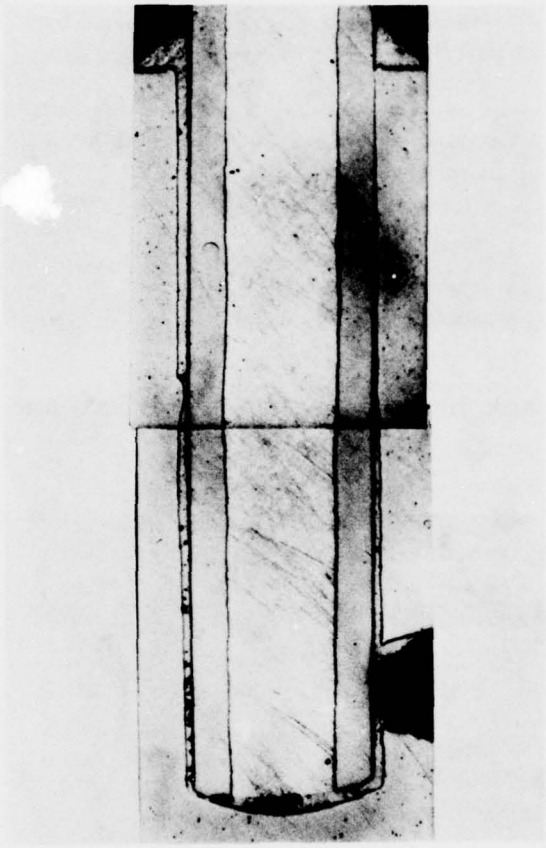
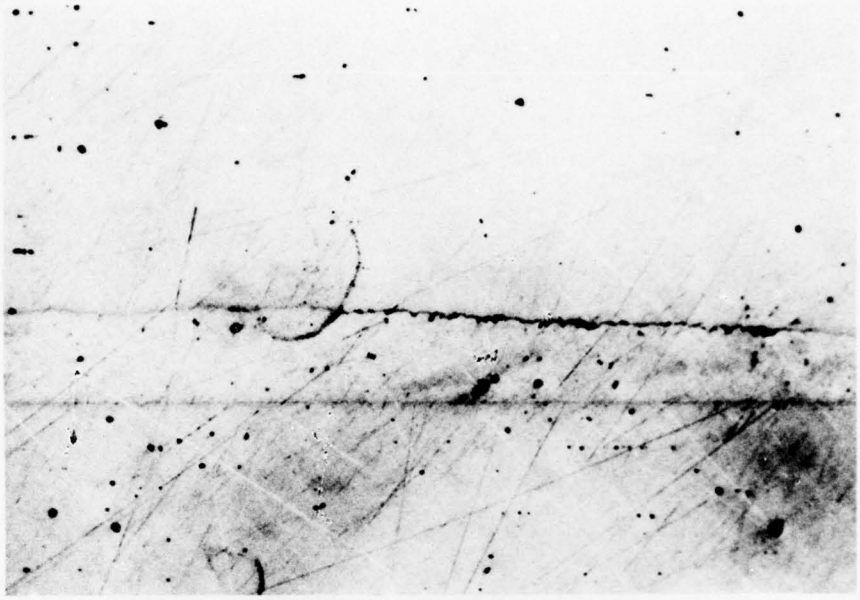


Figure 35

Inconel clad silver conductor brazed into Inconel contact pin; Ag-Ni-In brazing alloy. 23X



Microcrack between braze material and contact pin.
100X

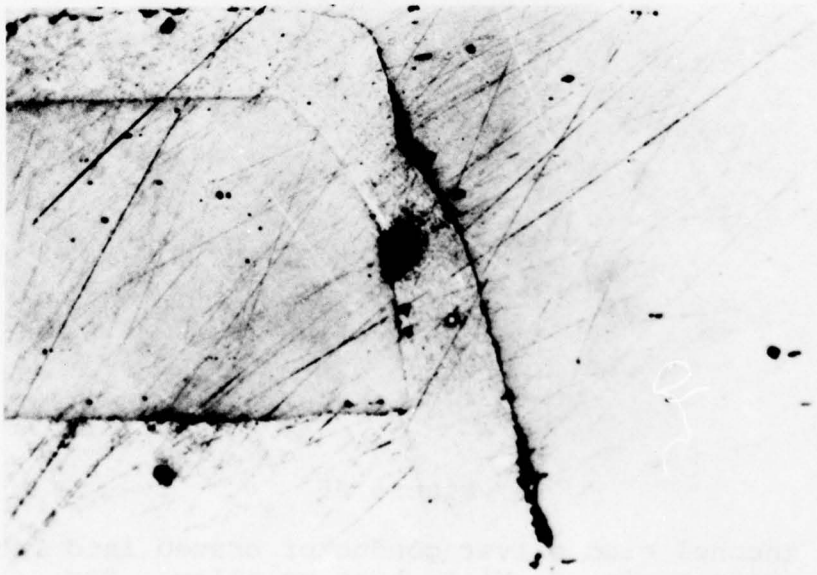


Figure 36

Microcrack between braze material and contact pin.
(Semi-continuous crack, located at bottom of bore).
100X

is most severe at the bottom of the bore (Figure 36), an area which is the most difficult to reach in plating operations. These fine cracks are not expected, however, to adversely influence the strength and electrical properties of the joint between the contact and the wire. Considering too that the microcracks are in the relatively malleable gold base brazing alloy, there is little danger that the microcracks would propagate into large and continuous cracks between the wire and contact.

8. The Effect of Braze Microcracks on Tensile Strength of Conductor/Contact Joints

Although it was considered that microcracking would have little effect on the strength of conductor contact joints, further experiments were performed to thoroughly investigate the phenomenon.

Tensile tests of brazed conductor/contact assemblies were performed at room temperature and at 1250°F, as described in Appendix A. A typical load elongation curve for room temperature is reproduced in Figure 37 showing, as expected, that the Inconel sheath dictates the shape of the deformation curve. After failure of the sheath has occurred, the silver core carrying the load work hardens rapidly to its ultimate tensile strength and then fails in a gradual manner characterized by chisel point fracture (Figures 38 and 39). The developed yield and ultimate stress are characteristic of Inconel and are in good accordance with the theoretical values (Appendix J). The tensile test curves were similar to Figure 37 at 1250°F, although, as expected, the strength values were lower. Failure at high temperature occurred in the conductor close to the "shoulders" of the specimens (Figure 40) as would be expected since these areas are regions of stress concentration. Careful examination of the braze fillets showed no evidence of damage. As it is likely that the brazed joints of these test samples contained some of the microcracks previously discussed, these tests indicated that, using the Au-Ni-In braze, joints between the conductor and contact are made which are stronger than the conductor itself, both at room temperature and 1250°F.

To further quantify the effect of microcracking on bond strength, an improved method of nickel plating the inside walls of the braze pots was developed (Appendix K). When wires were brazed into contacts plated in this fashion, only a few very short flaws were observed in the contact braze metal interface. Tensile strength evaluation yielded comparable results to those previously described.

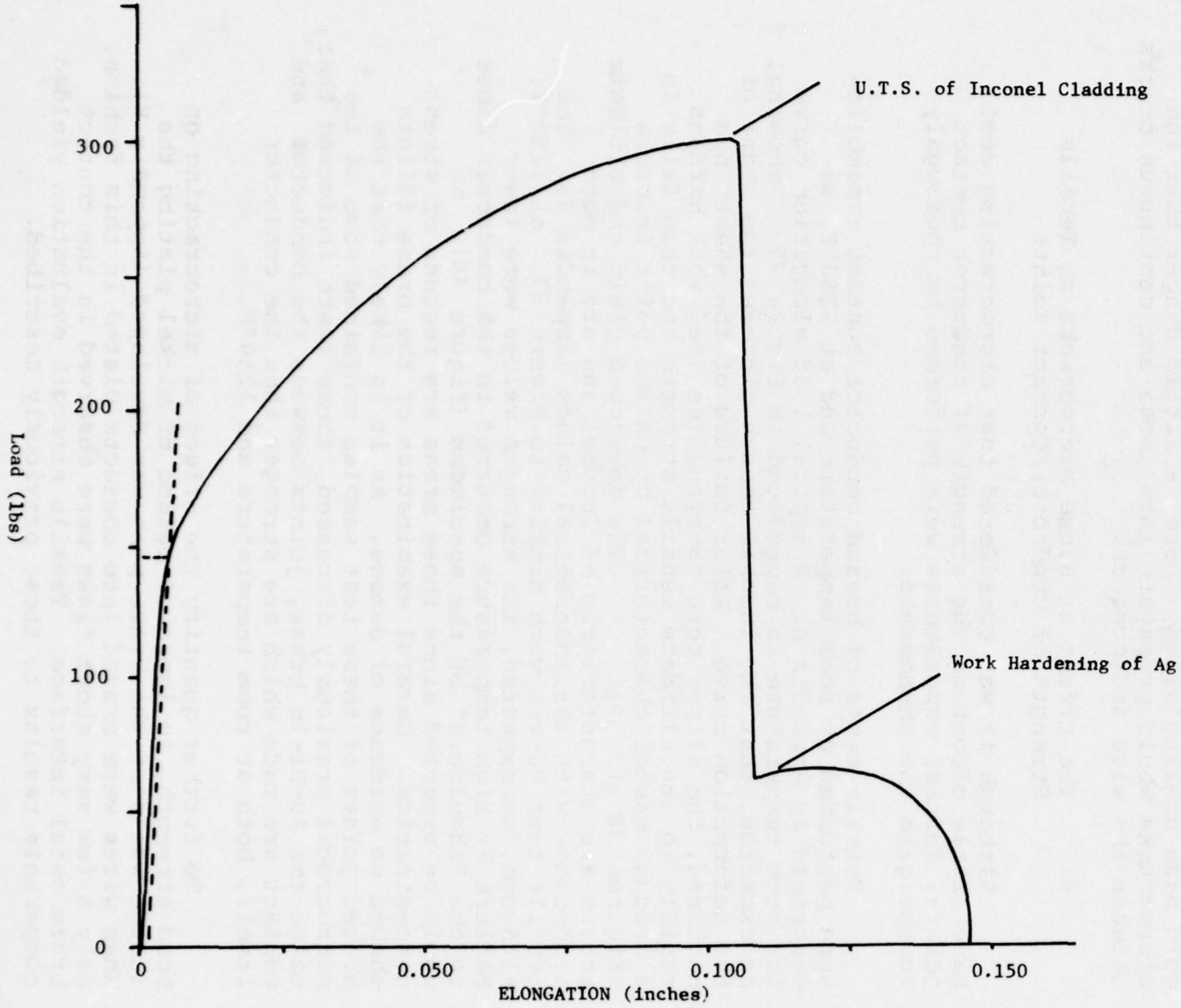


Figure 37. Load vs. Elongation Curve for Conductor/Contact Pin Assembly. (Test performed at room temperature)

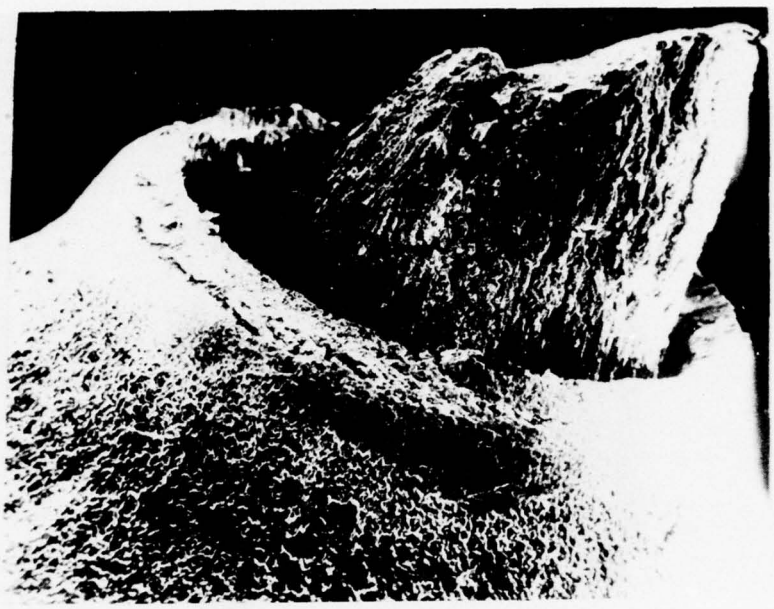


Figure 38

Composite fracture of Inconel-clad silver conductor, fractured at room temperature.

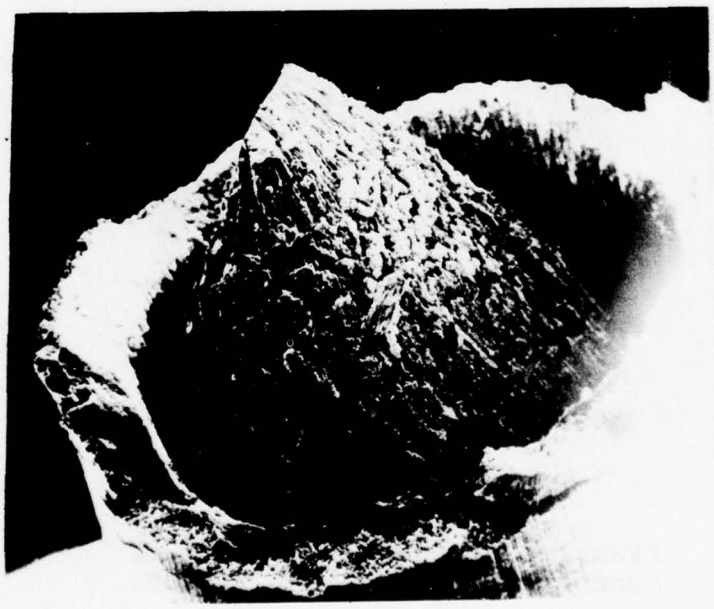


Figure 39

Composite fracture of Inconel-clad silver conductor, fractured at 1250°F.

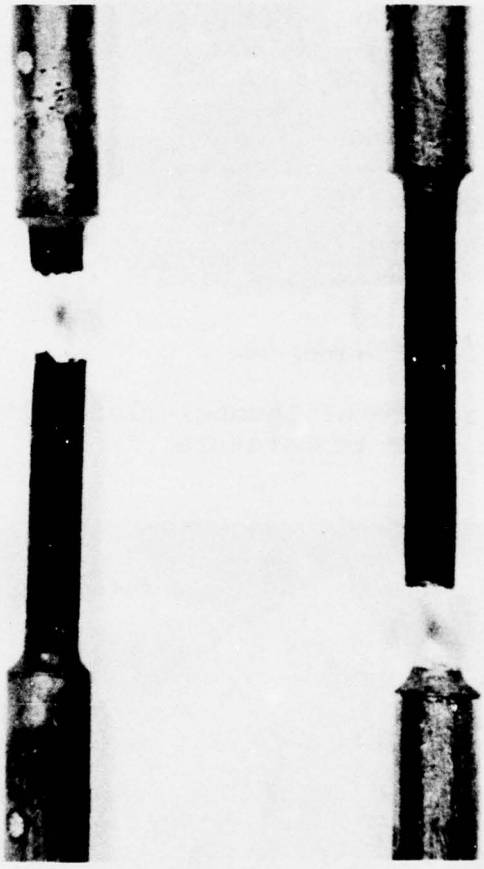


Figure 40

Examples of tensile failure of conductor/contacts, at 1250°F.

left - room temperature
right - 1250°F

Contact-wire brazes were also produced with no nickel plating on either the contact or the wire. Under these conditions the flow characteristics of the Au-Ni-In braze were much poorer than for nickel plated samples, so that no smooth fillets between the contact and wire were produced. However, the brazed joint between wire and contact was excellent with no faults in the braze metal-contact interface as shown in Figure 41.

From these experiments it was concluded that:

a) The contacts should be nickel plated, with special emphasis on control plating conditions to obtain adequate throwing power in the braze pot bores;

b) The bore should be provided with a side vent to encourage circulation of the electrolyte.

c) The plating bath should be agitated to improve electrolyte circulation in and around the braze pot.

Although acceptable wire/contact braze joints could be produced without nickel plating of the surfaces, this would require brazing in vacuum or in a reducing atmosphere, negating the advantages of the present kinetic high purity argon technique.

Selected specimens of brazed contact/conductors were heat aged at 1250°F for ∿1000 hours. The results of tensile strength studies and metallographic examination for oxidative deterioration are given in Appendices I and L.

A brief discussion of the Stop-off materials employed in these studies may be found in Appendix M.

Figure 41

Wire/contact brazed joint (1-Silver core of wire, 2-Inconel wire sheath, 3-Braze metal, 4-Inconel contact). 100X

D Prototype Connector Design Development

1. First Prototype Connector

Figures 42-46 show the first mated pair of prototype connectors harnessed to a short length of 12 AWG quad cable. These connectors were fabricated according to the Phase II first design (Figure 18). It should be noted that the disc cable clamps initially considered for use in this connector have been left off to minimize compressive strength to the wire insulation.

All contact/conductor joints were satisfactory, however, continuity testing revealed random short circuits which were finally traced to loose strands of knitted armour touching the conductor. Considerable difficulty was experienced in minimizing fraying of the armour and insulation during assembly. As a consequence no voltage drop experiments were performed. Mating and unmating of the connector pair were found to require more force than anticipated, which is possibly due to excessive thermal distortion resulting from repeated sealing operations.

2. Second Prototype Connector

As a result of damage to the wire insulation resulting from the use of shear prone cable clamp discs in the first prototype (Figure 18), a modified conical cable clamp was designed and fabricated which was found effective in both clamping the individual conductors, and minimizing mechanical damage to the wire insulation and sheath. Four views of this clamp are shown in Figures 47-50. The modified connector design is shown in Figure 21.

Further modifications were made to the design of the connector ferrule as a means of improving the clamping and securing of the outer knitted armour. A solid, conical version of the Kellems design approach was tentatively chosen (Figure 21), and a prototype fabricated for evaluation. The clamping mechanism over the knitted Inconel 600 armour was found to be satisfactory as the inner ferrule slid easily onto the wire bundle and the outer ferrule captured the flared overarmour satisfactorily. The design was adopted for the second prototype connector. It is intended that once the armour has been clamped solidly, the inner clamp gap will be brazed closed.

3. Preparation of Dummy Connectors

Following the successful development of a technique to metallize insulation at 1450°C and the encouraging oxidation resistance tests of the Au/Ni brazing alloy, several dummy connector specimens were made to optimize the fabrication of the housing/insulator/contact combination.

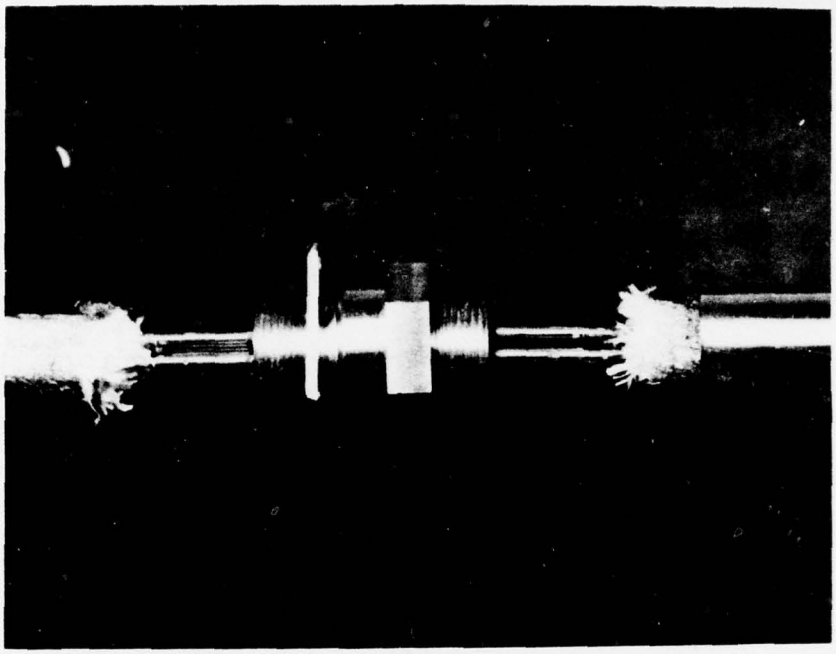


Figure 42. First prototype mated pair of high temperature connectors brazed to prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

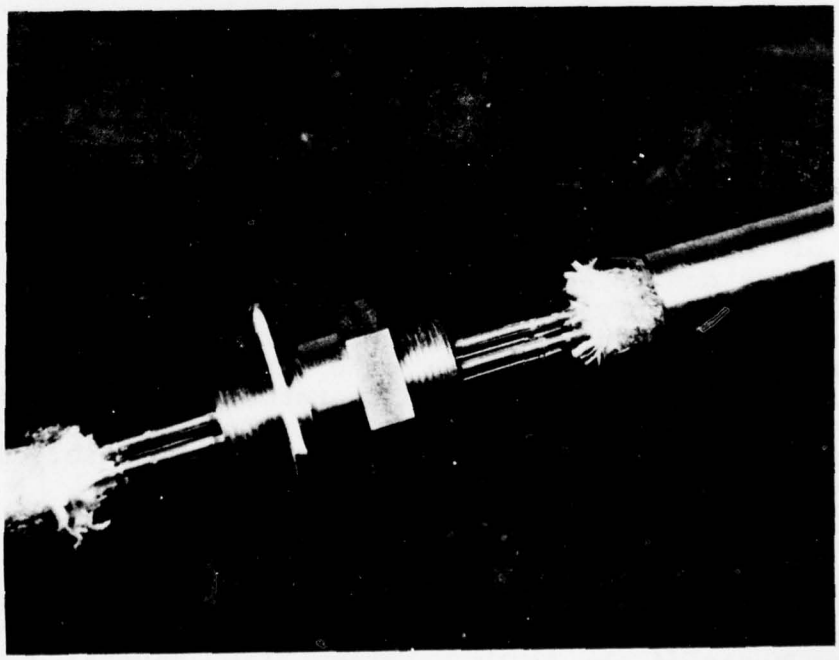


Figure 43. First prototype mated pair of high temperature connectors brazed to prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

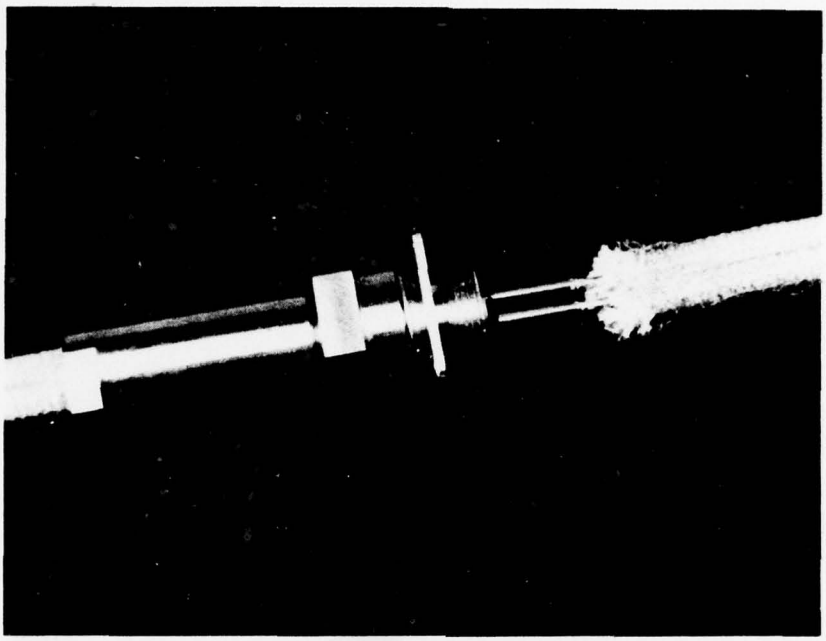


Figure 44. First prototype mated pair of high temperature connectors brazed to prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

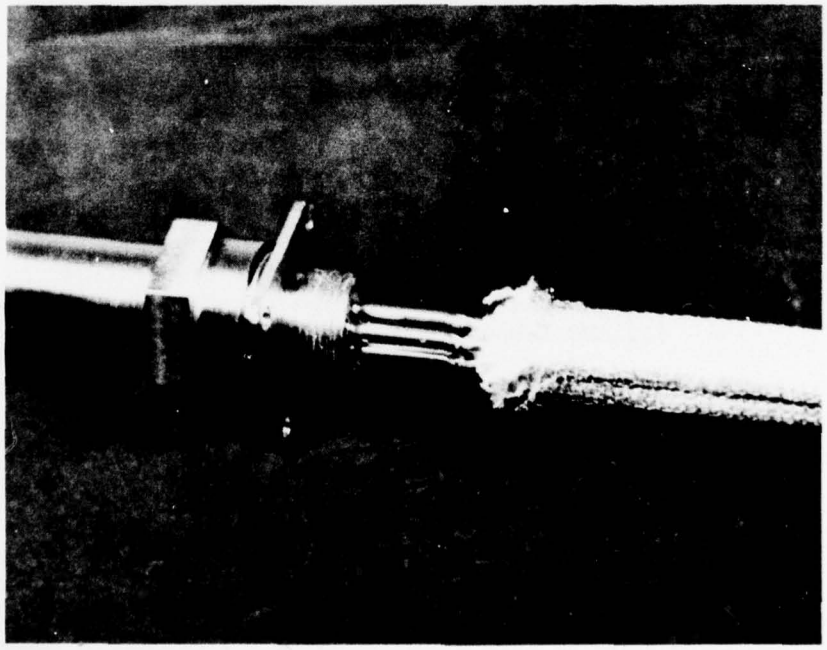


Figure 45. First prototype mated pair of high temperature connectors brazed to prototype cable containing 4 - #12 AWG Inconel clad silver conductors.

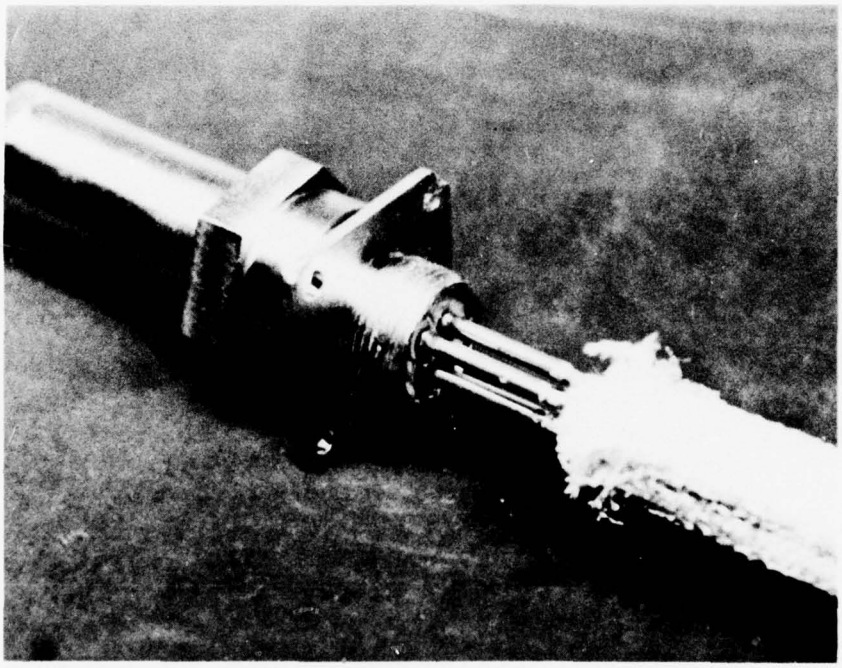


Figure 46. First prototype mated pair of high temperature connectors brazed to prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

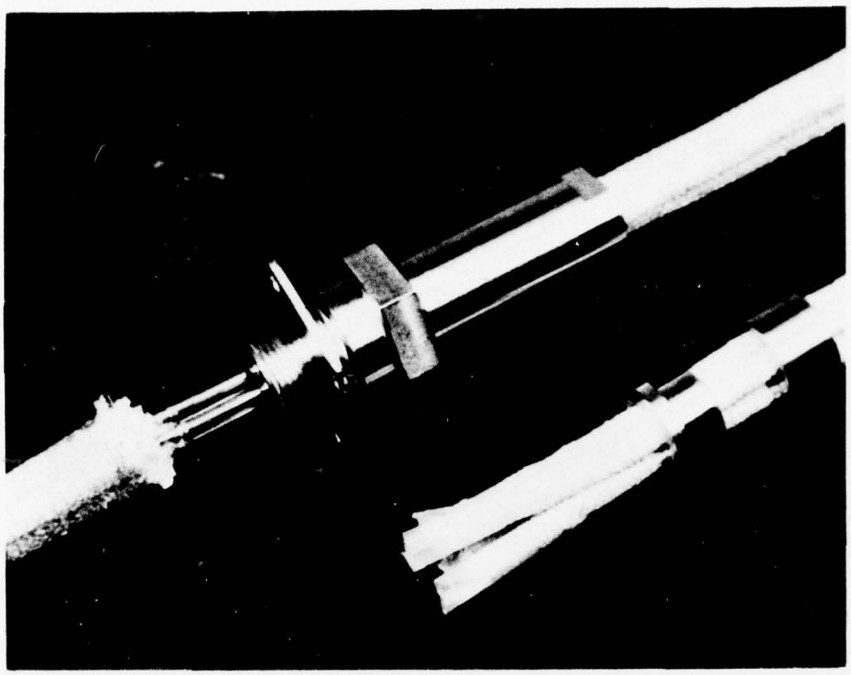


Figure 47.Modified cable clamp and prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

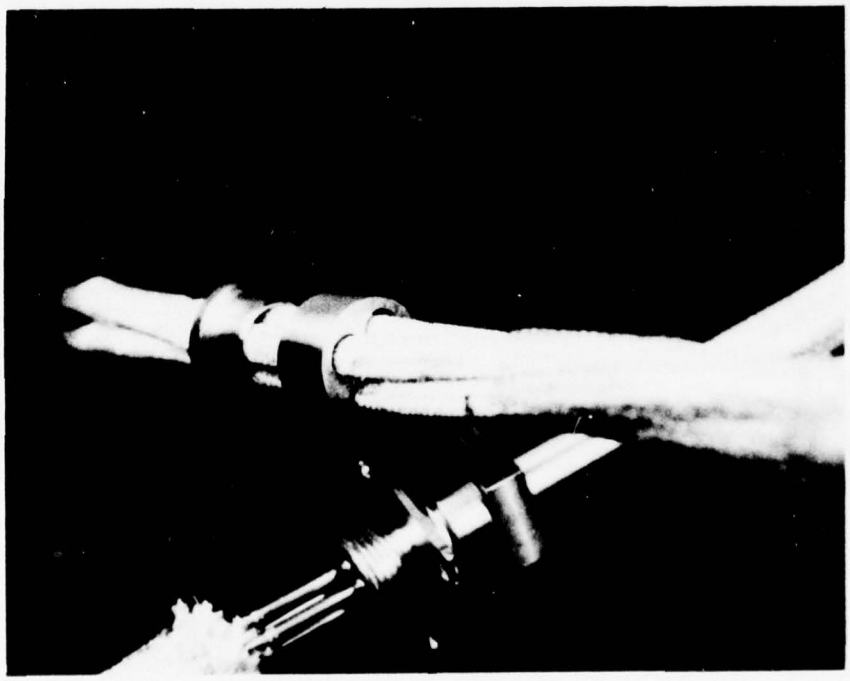


Figure 48.Modified cable clamp and prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

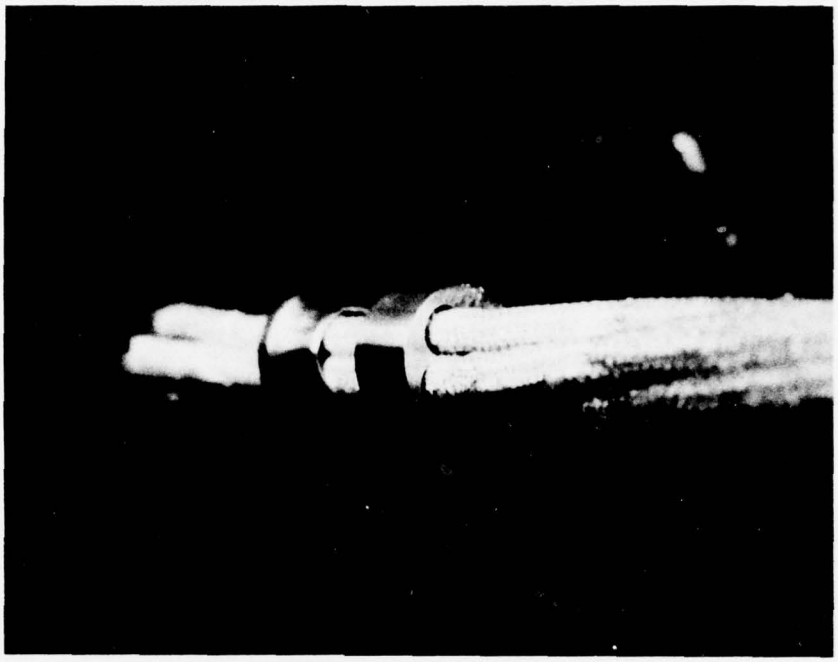


Figure 49. Modified cable clamp and prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

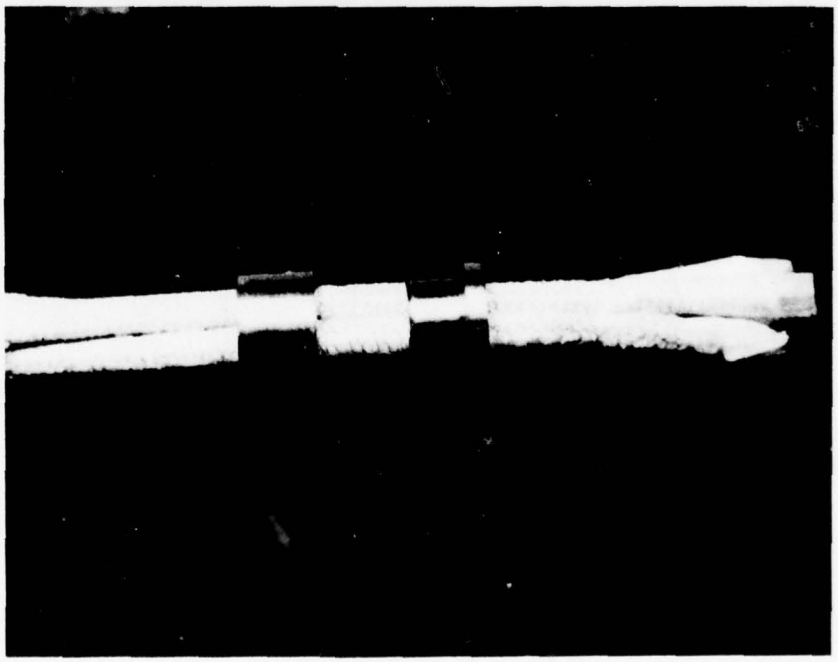


Figure 50. Modified cable clamp and prototype cable containing 4 - #12 AWG Inconel 600 clad silver conductors.

The first attempts used a powdered version of the alloy. Using insulators without keyways, it was found the joint between the insulator and housing could be made leak tight.

Upon receipt of alloy wire, relaxation tests were performed to determine if the wire would hold its shape on heating. The wire was first reduced by drawing to 0.028 inches, annealed and wound around a shaft of 0.103 inch diameter. The elastic springback allowed wire shaped in this way to fit snugly around a 0.123 inch diameter contact. Heating the wire to its melting point resulted in no observable shape change up to the onset of flow.

Further experiments demonstrated the superior capillary action of the braze material as excellent flow with no discernible brazement "shoulder" found in the samples.

The results of leak testing experiments on the dummy connector assemblies may be found in Appendix N.

4. Sealing of Cable Clamps

It can be seen from Figure 21 that a potential risk of moisture ingress exists by wicking along the wire insulation into the cable clamp. Furthermore, it had been found that, during attachment of prototype wire to the connectors, there was an undesirable fraying of the ceramic braid. To eliminate these problems a modification to the inner end of the cable clamp was proposed, to form a cup into which a suitable ceramic potting compound could be poured to lock the insulation.

Dummy cups were made up of stainless steel 304L alloy, to the design of the prototype. Discussions with Aremco Industries of Ossinning, New York established that their most suitable casting ceramic would be Ceramacast 528, an alumina ceramic having a service ceiling of 3200°F. This is a two-component material, being a powder with liquid activator normally used in the ratio of 5 parts powder to 1 part of activator, with a pot life of ∿10 minutes. Used at this ratio, however, it was found to be too stiff to work into the interstices, and, when set, was very grainy with minute occlusions. Increasing the activator to 1.25 parts effected a significant improvement in pourability but at the expense of pot life which was reduced to ∿5 minutes.

The initial experiments were performed in dummy plastic cups. Upon achieving a satisfactory pot, samples were prepared in stainless steel. A proportion of the samples were fired at the recommended curing temperature of 1860°F, and, as a means of reducing the potential thermal damage to the primary dielectric, samples were also fired at 1250°F. Both sets of samples were tested for electrical performance. The results may be found in Appendix O.

This approach was found to be unsatisfactory, as the preferred electrical resistivity characteristics could not be attained. Further experiments to examine alternate approaches were cancelled, due to the cost overrun.

5. Prototype Connector Delivery

Two mated pairs of connectors for 12 AWG based cable and 1 mated pair of connectors for each of 14 through 22 AWG based cable have been delivered. The final bores in ferrules and cable clamps have not been cut pending determination of the actual size of the prototype cable.

SECTION V

TEST METHODS

A Introduction

Table 33 summarizes the performance properties obtained from the various fabricated prototype assemblies developed in Phase I([1]), compares these values with the specification requirements listed in the original Statement of Work and provides recommendations for realistic performance values which are felt to be achievable in a commercial product. The justification for the changes, based on performance experience, may be found in Phase I final report([1]).

A revised Statement of Work was drafted at the beginning of Phase II, and is as follows:

1. Performance Requirements

a. Conductor

Demonstration of the following characteristics is required of the 12, 14, 16, 18, 20 and 22 AWG 40% Inconel 600 clad silver conductors.

(1) Conductor Resistivity

- less than 6×10^{-6} ohm-cm at ambient temperature.
- less than 12×10^{-6} ohm-cm at 1250°F

(2) Tensile Strength

- greater than 35,000 psi at ambient temperature
- greater than 25,000 psi at 1250°F

b. Insulated Wire

For wire evaluation purposes, only sizes #12 and #22 AWG need be tested.

(1) The design life at 1250°F shall be greater than 1000 hours.

(2) The maximum continuous potential rating at temperature (1250°F) shall be 600 Volts a.c.

(3) The insulation shall be compatible with the conductor chosen and shall have an insulation resistance greater than 1 megohm/ft under all conditions.

(4) Blocking shall be negligible at 1250°F.

TABLE 33

COMPARISON OF PERFORMANCE PROPERTIES OF PROTOTYPE WIRE WITH REQUIREMENTS DETAILED IN THE STATEMENT OF WORK

Test	Test method	Requirement[1]	Wire size (AWG)	Performance properties				Recommended performance standards	
				Room temp.		1250°F			
				Unaged	Aged[2]	Unaged	Aged[2]	Room temp.	1250°F
Conductor resistivity (microhm-cm)	Fed.Std.No.228 method 6021	<6	12	2.72	2.71	9.61	9.64	<4	<12
			18	2.84	2.78			"	"
			22	2.76	2.89			"	"
Tensile strength (psi)	Fed.Std.No.228 method 3211	35,000	12	58,900	56,000	38,800	30,300	>45,000	>25,000
			18	59,500	53,300			"	"
			22	54,000	51,200			"	"
Insulation resistance (megohms/ft.)	ASTM D257	>1 at 1250°F	12	1.3×10^7		8.0	3.5[a]	>1	>1
Dielectric strength (kilovolts/ac)	ASTM D149	1.20 under all environmental conditions	12	>5.0	2.0[b]	2.4		2.2	>1.2
Mandrel	Statement of Work	½20X wire diam.	12	Passed	Passed[c] /D.W.[3]			½20X wire diam.	½20X wire diam.
Thermal shock resistance	MIL-STD-202D method 107C (modified)		12	"	"			5 cycles of -65°F & 1250°F	

TABLE 33 (CONTINUED)

Test	Test method	Requirement[1]	Wire size (AWG)	Performance properties				Recommended performance standards	
				Room temp.		1250°F			
				Unaged	Aged[2]	Unaged	Aged[2]	Room temp.	1250°F
Vibration resistance	MIL-STD-202D method 204B	10-2000 cps, 20 g's	12	Passed[d] /D.W.[3]	Passed[d] /D.W.[3]			20 g's/10-2000 cps	
Mechanical shock resistance	MIL-STD 202D method 213A	50 g's/11 ± 1 msec.	12	"	"			50 g's/11 ± 1 msec.	
Abrasion resistance (in. of garnet tape)	Fed.Std.No.228 method 2211		12	30				25 minimum	
Flame resistance	Fed.Std.No.228 method 5211		12	Passed /D.W.[3]					
Life cycle' '	Statement of Work	1) 1000 hr. at 1250°F 2) 600 volts at 1250°F for 1000 hr.		Planned for Phase II					
Flexibility & flex fatigue	AFAPL-TR-66-97	20X wire diam.	-	Planned for Phase II				20X	20X
Blocking	MIL-C-27500A		-	Planned for Phase II					
Shrinkage (in.)	MIL-C-13777F	0.06	-	"					
Sublimation	No test	At normal & elevated temperatures & altitudes	12	Minimal				-	Minimal

(1) As per the Statement of Work.
(2) Aged for 1000 hours at 1250°F (unless stated otherwise).
(3) After testing, passed dielectric withstand of 2.2 Kv/5 sec.

(a) After 180 hours at 1250°F
(b) After 800 hours at 1250°F
(c) After 5 hours at 1250°F
(d) After 25 hours at 1250°F

(5) The dielectric strength shall be sufficient to pass 1200 Volts to ground under all environmental conditions.

(6) Shrinkage - shall be less than 0.06 inches.

(7) The wire shall be capable of being wound on a mandrel of a suitable diameter without physical or electrical detriment.

(8) Thermal Shock Resistance. The wires must be capable of passing a dielectric withstand of 2200 Volts for 5 seconds after five thermal shock cycles.

(9) Abrasion Resistance. The wires must be capable of withstanding greater than 25 inches of garnet tape before and after thermal exposure.

(10) Sublimation at normal and elevated temperatures and altitudes shall be negligible.

(11) Degree of flexibility including flex-fatigue life. The wire must be capable of withstanding a voltage of 2200 V for 5 seconds after flexing around 20X and 10X diameter mandrels after 0, 250, 500, and 1000 hours exposure at 1250°F.

c. Connector

(1) Contact spacing and configuration shall permit a contact to contact and contact to shell steady state working voltage of 250 volts rms 400 hz.

(2) The insulation resistance between any two contacts and between the shell and any contact of mated connectors at high temperature shall be 500 megohms when tested in accordance with Mil-Std-202, method 302, test condition B.

(3) Design life at maximum temperature, 1000 hours.

(4) Contact design and arrangement.

(5) Mated contact resistance at temperature and aging, 125 millivolts.

(6) Contact retention.

(7) Shell design including coupling method and cable support.

(8) Resistance to ozone, hydraulic fluids, salt spray, and kerosene (JP-4 or JP-5).

(9) Corona

(10) Compatability with wire and insulation system developed under this program.

(11) Interfacial sealing of connector halves.

d. Connector/Wire Assembly

(1) The wire and connector assembly shall be capable of passing a 1.2 kV dielectric withstand after a modified Flight Cycle Test.

(2) The mated star quad cable shall be capable of withstanding 1.2 kV for 5 seconds after vibration testing at 20 g's from 10 to 2000 hertz frequency.

(3) The mated star quad cable shall be capable of withstanding physical shocks of 50 g's peak acceleration for a duration of 10±1 millisecond.

(4) The mated wire/connector assembly shall be capable of operating in surroundings of 0 to 100% relative humidity. It must also be capable of operating at altitudes up to 110,000 feet, that is, under pressures of from 760 to 7 mm Hg.

A series of test methods and specifications have been drafted to allow thorough evaluation of the components developed during the program. Established military and U.S. federal test methods have been used as guidelines, where applicable.

2. Categories of Test Methods

The methods are divided into four main categories:

A. Conductor

Method	TMA-1	Conductor resistivity
	TMA-2	Conductor tensile strength

B. Insulated Wire and Cable

TMB-1	Insulation resistance
TMB-2	Maximum continuous potential
TMB-3	Dielectric withstand
TMB-4	Blocking
TMB-5	Shrinkage
TMB-6	Mandrel
TMB-7	Thermal shock resistance
TMB-8	Abrasion resistance
TMB-9	Vibration resistance
TMB-10	Physical shock resistance
TMB-11	Sublimation
TMB-12	Degree of flexibility, including flex fatigue life

C. Connector

TMC-1	Steady state voltage withstand
TMC-2	Insulation resistance
TMC-3	Mated contact resistance
TMC-4	Contact retention
TMC-5	Resistance to ozone
TMC-6	Resistance to hydraulic fluids and kerosene (JP-4 or JP-5)
TMC-7	Resistance to salt spray
TMC-8	Corona resistance
TMC-9	Interfacial sealing

D. Mated Connector/Wire Harnessing

TMD-1	Modified life cycle
TMD-2	Vibration resistance
TMD-3	Physical shock resistance
TMD-4	Flexibility

B Method TMA-1

Conductor Resistivity

1. Scope

This method is intended for use in determining the electrical resistivity of Inconel clad fine silver core conductor, both at normal room ambient and elevated temperatures.

2. Specimen

A 4-foot sample of a conductor will be tested for electrical resistivity at 72°F and 1250°F, both before and after aging the conductor at 1250°F for 1000 hours.

3. Apparatus

The apparatus shall consist of:

(a) A direct current source, electronically regulated to 1 ampere.

(b) A suitable four terminal resistance bridge with an accuracy greater than 0.1 percent. Figure 51.

(c) A test fixture having both current and potential contacts with a distance accuracy of ± 1 percent for determining the room temperature resistivity.

(d) A Vycor tube furnace that will maintain 1250°F ± 10°F over a distance of at least six inches of the conductor. Figure 52.

4. Procedure

a. The d.c. resistance of the conductor shall be measured in accordance with the applicable sections of Method 6021 of U.S. Federal Std. No. 228 at room temperature.

b. High temperature resistance measurements shall be made with nickel wire potential connections silver soldered to the wire, over the distance that the tube furnace can maintain 1250°F ± 10°F.

c. The length of the specimen shall be measured to an accuracy of 0.2% and the value recorded as L.

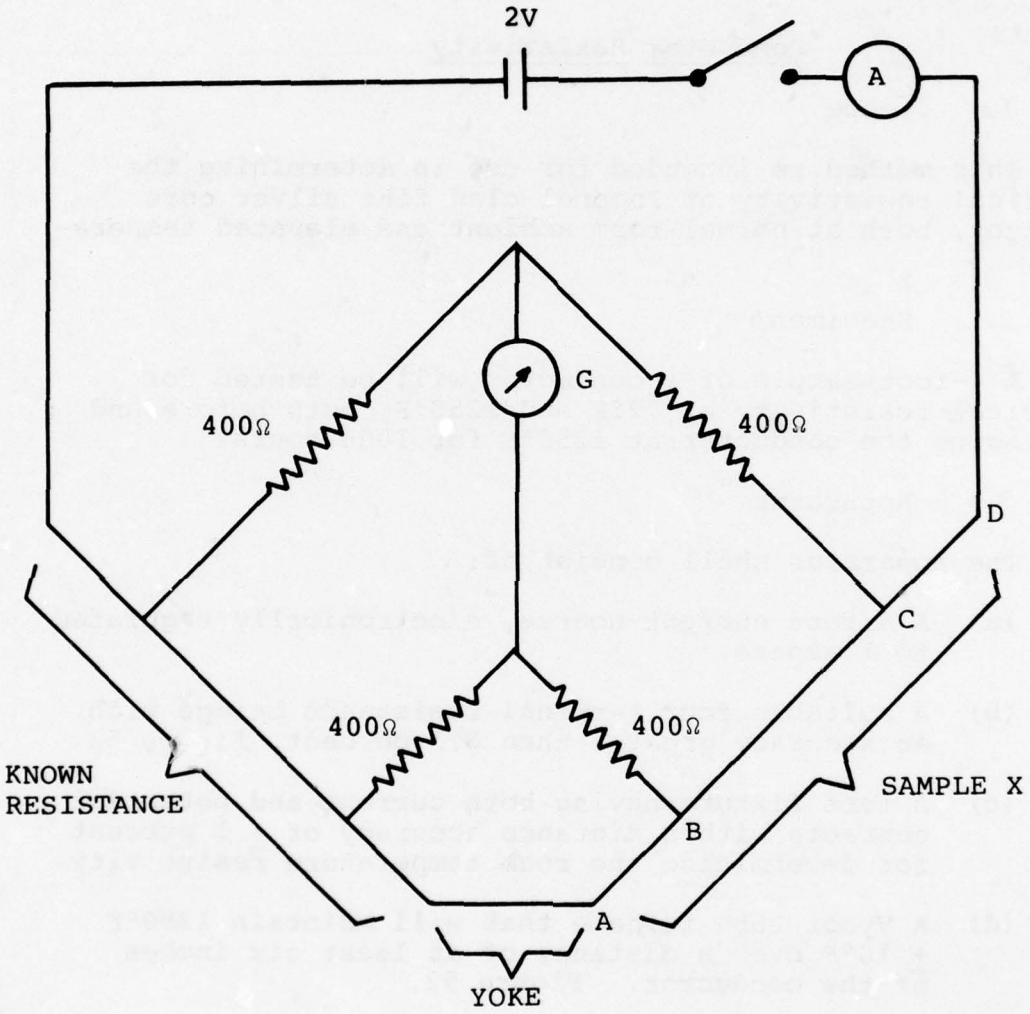


Figure 51

Bridge circuit for resistance measurements on conductors.

(The yoke resistance is close to zero. There are four leads to the sample. Leads at B and C are used in the bridge circuit for potential and virtually no current passes through them. Leads at nodes A and D supply the current to produce the voltage drop between B and C.)

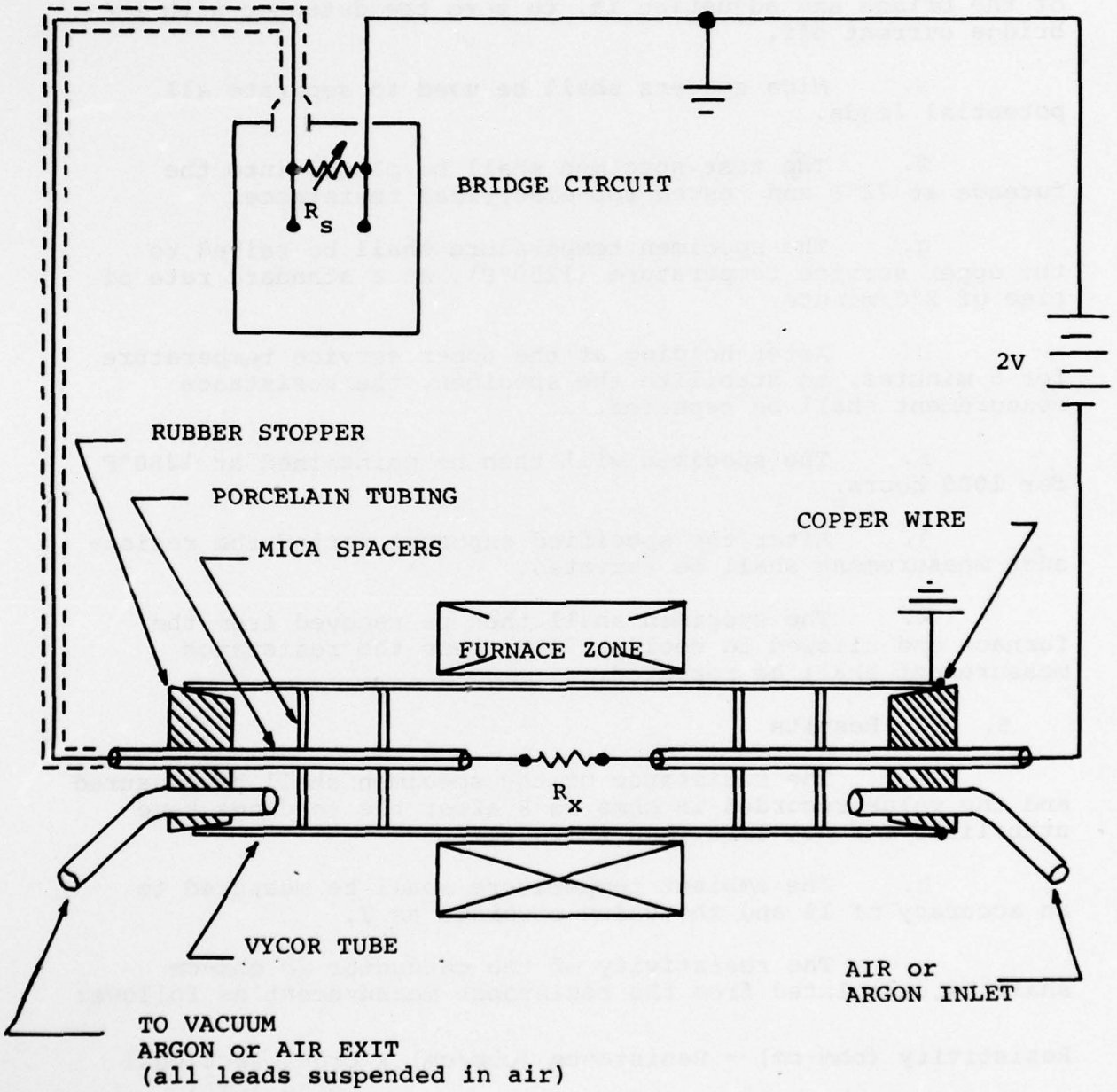


Figure 52

Conductor resistance measuring apparatus.

d. Stray thermal cmf's shall be neutralized by connecting a microvolt source to one of the potential leads of the bridge and adjusting it, to zero the detector with the bridge current off.

e. Mica spacers shall be used to separate all potential leads.

f. The test specimen shall be placed into the furnace at 72°F and tested for electrical resistance.

g. The specimen temperature shall be raised to the upper service temperature (1250°F), at a standard rate of rise of 2°C/minute.

h. After holding at the upper service temperature for 5 minutes, to stabilize the specimen, the resistance measurement shall be repeated.

i. The specimen will then be maintained at 1250°F *for 1000 hours.*

j. After the specified exposure period the resistance measurement shall be repeated.

k. The specimen shall then be removed from the furnace and allowed to cool to 72°F where the resistance measurement shall be repeated.

5. Results

a. The resistance of the specimen shall be measured and the value recorded in ohms as R after the readings have stabilized for not less than 1 minute.

b. The ambient temperature shall be measured to an accuracy of 1% and the value recorded as T.

c. The resistivity of the conductor in ohm-cm shall be calculated from the resistance measurement as follows:

Resistivity (ohm-cm) = Resistance (ohm/cm) x cross sectional area (cm^2)

$$\rho = \frac{R}{L} \times \frac{\pi d^2}{4}$$

where d = diameter of wire specimen, cm
and L = length of specimen between potential connections, cm.

6. Specification

The resistivity at room temperature both before and after aging shall be less than 6.0×10^{-6} ohm-cm. The resistivity at 1250°F shall be less than 12.0×10^{-6} ohm-cm.

(This test is based on Method 6021 of Fed-Std-228).

C Method TMA-2

Tensile Strength of Conductors

1. Scope

This method is intended for use in determining the tensile strength of solid Inconel 600 clad fine silver conductors (or other suitable high temperature resistant conductors), and wires of stranded conductors.

2. Specimen

The specimens for each test shall be four samples of the bare conductor, or a wire from a stranded conductor, of at least 16 inches in length each.

3. Apparatus

a. The tensile testing machine shall be of such capacity that the maximum load required to break the specimen is not greater than 85 percent nor less than 15 percent of rated capacity.

b. Grips of either the spool-type or those designed specially for wire shall be used, to produce as nearly as possible pure axial tension in the specimen.

c. The tension shall be measured by a dial, scale or automatic recorder to within $\pm$ 1 percent when properly calibrated.

d. The crosshead speed shall be a uniform 2.0 $\pm$ 1 inches per minute under load.

e. The sample must break at least 1 inch away from the grips, or the result is discarded.

f. The percent elongation, if required or if feasible, shall be determined by measuring the increase in distance between two parallel bench marks placed 4.0 $\pm$ .05 inches apart on the test specimen.

g. The apparatus shall be fitted with a suitable oven arranged to surround the specimen once gripped in the holding means, and maintaining the upper service temperature with an accuracy of ± 10°F.

4. Results

a. The results shall include the average wire diameter, the ultimate tensile force (pounds), tensile strength (psi) and percent elongation, if feasible.

b. The tensile strength and elongation of the conductor shall be the average of the results obtained from all the specimens tested.

c. The tensile strength shall be reported to the nearest 10 pounds per square inch.

d. Results shall be reported for:

(1) room temperature (no aging)

(2) room temperature (after 1000 hours of heat aging at 1250°F)

(3) 1250°F (after heat aging for 1000 hours at 1250°F).

5. Specification

All conductors shall be required to have tensile strengths of:

(a) greater than 35,000 psi at room temperature, and,

(b) greater than 25,000 psi at 1250°F, both before and after aging.

(This test is based on Method 3211 of Fed-Std-228)

D Method TMB-1

Insulation Resistance

1. Scope

This method is intended for use in determining the insulation resistance of insulated wire and cable. The test measures the resistance offered by the insulating members of the wire to an impressed direct voltage tending to produce a leakage of current through or on the surface of these members. The test is especially helpful in determining the extent to which insulating properties are affected by deteriorative influences, such as heat, moisture, oxidation or loss of volatile materials.

2. Specimen

a. The insulated wire specimens shall consist of at least four 10 inch lengths of wire with at least 6 inches of the outer Inconel braid removed for testing.

b. This test shall be carried out on wire specimens at ambient room temperature and at 1250°F, both in the as-fabricated condition and after aging at 1250°F for 1000 hours.

3. Apparatus

a. The insulation resistance measurements shall be carried out using similar apparatus used in the conductor resistivity, test method TMA-1. A megohm bridge, megohm meter, insulation resistance test set or other suitable apparatus that will give an accuracy of $\pm$ 10 percent shall be used.

b. A d.c. voltage source shall be used that can supply 500 V $\pm$ 10%.

c. The Vycor tube furnace shall have an operational temperature of 1250°F.

4. Procedure

a. After carefully removing the Inconel 600 knitted mesh from the wire, a five-inch strip of 0.001 inch thick Inconel 600 or other suitable foil shall be carefully wrapped around the wire to guarantee smooth coverage of the insulation.

b. The foil shall be secured at both ends with nickel wire. Extension leads shall be brazed to one of the nickel wires and to the conductor.

c. Guard electrodes consisting of nickel wire shall be wound around the insulation a short distance on either side of the foil wrap. Mica spacers shall be used to keep the various leads separated.

d. The specimen shall then be placed into a Vycor tube furnace along with a thermocouple.

e. The insulation resistance shall be measured to an accuracy of $\pm$ 5 percent using a suitable megohm bridge and meter, at a potential of 500 Volts d.c. after an electrification time of one minute. By connecting the bridge circuit so that the guard terminals are grounded, all leakage to ground shall effectively be eliminated from the measurement.

f. The insulation resistance shall be measured at room ambient temperature (72°F).

g. The tube furnace shall then be raised to 1250°F $\pm$ 10°F. After stabilizing for 5 minutes the insulation resistance shall be determined.

h. The sample shall be aged in a furnace at 1250°F for 1000 hours. After this heat aging, the insulation resistance shall be determined at 1250°F.

i. The wire sample shall be slowly cooled to room temperature and the insulation resistance determined again at 72°F.

j. Care shall be taken in handling so as not to damage the ceramic insulation.

5. Results

The insulation resistance shall be determined and reported for both the unaged and aged wire, at 72°F and 1250°F.

6. Specification

The insulation resistance is required to be greater than 1.0 megohm/ft under all experimental conditions.

(This test is based on Method 6031 of Fed-Std-228, and Method 302 of Mil-Std-202D).

E Method TMB-2

Maximum Continuous Potential

1. Scope

This method is intended for use in determining whether the wire will pass a continuous potential rating of 600 Volts a.c. while operating at 1250°F for the 1000 hour design life of the wire.

2. Specimen

The specimen shall consist of at least an 8 inch sample of finished insulated wire.

3. Apparatus

a. The apparatus shall consist of a variable a.c. voltage power source, a voltmeter of ± 5% accuracy.

b. The high temperature chamber shall consist of a Vycor tube furnace and controller capable of maintaining 1250°F over the wire length.

c. An ammeter shall be used to measure the current through the wire.

d. A dielectric tester shall be required to measure the insulation dielectric withstand voltage, according to test TMB-3, after this present test.

4. Procedure

a. Suitable silver potential leads shall be soldered to the wire ends and the sample placed in the Vycor tube furnace.

b. The temperature shall be raised to 1250°F.

c. The voltage shall be raised to 600 Volts rms, while in series with a variable resistor so that the current as measured by the ammeter remains less than 1 amp during the test.

d. The voltage shall be maintained at 600 Volts ± 5% for 1000 hours.

e. The current shall be monitored periodically and any fluctuations or stoppages recorded.

f. While the sample is still in the apparatus at 1250°F, the dielectric withstand test TMB-3 shall be carried out.

5. Results

a. Any fluctuations in the current reading during testing shall be recorded.

b. The results of the dielectric withstand test after the continuous potential rating test is performed shall be reported.

6. Specification

The wire shall be capable of passing a 1.2kV dielectric withstand for 5 seconds at 1250°F after the continuous potential rating test.

F Method TMB-3

Dielectric Withstand

1. Scope

This method is intended for use in determining whether the insulation over a metallic conductor will withstand a specified voltage without rupture. The dielectric strength test consists of the application of a voltage higher than the rated voltage for a specific time between mutually insulated portions of the finished wire, or between insulated portions and ground. It is not intended that this test cause dielectric breakdown or that it be used for detecting corona, rather it serves to determine whether the insulating materials and spacings in the wire and connector parts are adequate.

2. Specimen

The wire sample to be tested shall be at least 8 inches in length and the Inconel 600 mesh shall be pulled back or unravelled from the conductor ends. This will eliminate the possibility of corona occurring between the conductor and metallic mesh.

3. Apparatus

a. The dielectric tester shall be a suitable high voltage breakdown tester capable of supplying up to 500 VA of power, with a maximum output voltage of 10kV.

b. The current cutout sensitivity shall be adjustable for each high voltage range.

c. The tester shall be capable of raising the voltage automatically or manually, and the rate of rise of the voltage shall be fully adjustable.

d. The high voltage source shall be used to deliver a voltage of 60 hertz in frequency and shall approximate a true sine wave in form.

e. A voltmeter shall be used to measure the applied voltage to an accuracy of at least 5 percent.

4. Procedure

a. A one inch wide by 0.001 inch thick Inconel 600 foil shall be wrapped tightly around the outer metallic sheath to be used as the high voltage electrode. The conductor shall be the ground electrode.

b. The Inconel 600 mesh shall be pulled back or unravelled at least 1/2 inch from each end of the conductor.

c. A voltage of 1.2kV a.c. shall be applied across the insulation for five seconds using a current trip sensitivity of 8 ma. A suitable ammeter method with fault indicator shall be provided to indicate the occurrence of disruptive discharge and leakage current in case it is not visually evident in the specimen.

d. Any dielectric breakdown values shall be obtained using a voltage rate-of-rise of 350 volts/sec at 60 cycles a.c. and the same current sensitivity as used for the dielectric strength test.

e. Each specimen shall be subjected to all the test environments for at least 30 minutes before testing in the progressive order listed in Table 34. The dielectric strength measurements shall be made while the sample is still in the testing environment at the completion of each stage of conditioning.

TABLE 34

SAMPLE ENVIRONMENTAL CONDITIONS

TEST CONDITION	TEMPERATURE	% RELATIVE HUMIDITY	PRESSURE (mmHg)
1	70°F	50	760
2	70°F	100	760
3	1250°F	0	760
4	1250°F	0	7
5	-65°F	0	766

5. Results

a. Each wire shall be tested at each environmental condition and a pass or fail rating will be given for the dielectric withstand test.

b. If breakdown occurs during the test, the dielectric breakdown voltage shall be recorded and a visual examination of breakdown area shall be carried out.

6. Specification

All wires shall be required to pass 2.2kV a.c. for 5 seconds dielectric withstand at room temperature for all samples, and shall be required to pass 1.2kV a.c. for 5 seconds at 1250^{o}F.

(This test is based on Method 301 of Mil-Std-202D, and Method 6111 of Fed-Std-228).

G Method TMB-4

Blocking

1. Scope

This method is intended for use in measuring the amount of sticking or adhesion between wires after a high temperature exposure.

2. Specimen

The wire or cable to be tested shall be of sufficient length to be wrapped around a 20X diameter mandrel nine times. This will require approximately a twelve foot length for the 12 AWG finished wire.

3. Apparatus

a. A mandrel made of high temperature resistant metal of not greater than 20 times the diameter of the wire shall be used for winding the wire.

b. A high temperature furnace capable of containing the mandrel and maintaining 1250°F is required.

4. Procedure

a. One end of the sufficient length of wire shall be fixed to the mandrel.

b. The wire shall then be spirally wound around the mandrel so that four complete turns are in close contact with one another. A tension weight of 3 pounds shall be attached to the end of the wire during winding to effect an even winding tension of each layer.

c. The windings shall be continued so that the second and third layers of wires contain three and two complete turns respectively, with each layer in close contact with one another.

d. The free end of the wire shall then be fixed to the mandrel so as to prevent unwinding or loosening of the turns and/or layers.

e. The mandrels and wrapped wires shall then be placed in the furnace at a temperature of 1250^{o}F for 24 hours.

f. After removal from the furnace the mandrel and wire shall be cooled to room temperature and the wire unwound slowly from the mandrel using the 3 pound tensioning weight.

5. Results

a. Observations shall be recorded of any fume or exudate on the insulation surface, or any changes in the physical appearance of the Inconel mesh.

b. Any signs of sticking are to be recorded.

6. Specification

The 12 AWG finished insulated wire shall be tested and there shall be no adhesion or sticking of adjacent turns or layers during the unwinding process. Because the Inconel 600 mesh and refractory outer braid do not undergo oxidation or softening, it is anticipated that there will be no blocking of the insulated wire.

(This test is based on test method Mil-C-27500A).

H Method TMB-5

Shrinkage

1. Scope

The purpose of this test is to determine the amount of shrinkage of the insulation layers that is occurring during high temperature exposure. Due to the ease with which inorganic refractory braided materials unravel, combined with the inherent stretch and flexibility of the insulation package, the shrinkage of the experimental high temperature wire is difficult to measure accurately. Previous high temperature testing of these wires has indicated no noticeable amount of shrinkage is occurring.

2. Specimen

This test shall be carried out on 6.0 inch length of wire. Sizes #12 and #22 AWG shall be tested for evaluation purposes.

3. Apparatus

a. The equipment shall consist of a Vycor tube or muffle furnace capable of maintaining 1250°F $\pm$ 10°F.

b. A suitable caliper micrometer shall be used to measure the shrinkage of any of the insulation layers.

4. Procedure

a, A six inch specimen of the finished wire shall be cut so that the conductor and insulation are flush at both ends. The metallic knitted mesh shall also be cut flush with the conductor to aid in holding the ceramic braids in place.

b. The wire shall be heated to 1250°F in a muffle furnace or Vycor tube furnace, and maintained at 1250°F for at least 24 hours.

c. The sample shall then be carefully removed from the furnace and cooled to room temperature in a dessicator.

d. The shrinkage shall then be measured as the greatest distance which any insulation layer has receded from either end of the conductor.

5. Results

a. The greatest shrinkage distance shall be recorded for the 6 inch length of wire.

b. Observation of the end of the wire shall be carried out to observe signs of any charring, fuming or fraying.

6. Specification

The shrinkage of any layer must not exceed 0.06 inches from either end of the test sample.

(This procedure was based on test method Mil-C-13777F).

I Method TMB-6

Mandrel Test

1. Scope

This test is intended to determine the ability of the finished insulated wire to be wrapped on a mandrel of diameter not greater than 20X the diameter of the wire.

2. Specimen

The 12 and 22 gauge wires are to be tested, using a length of wire sufficient to allow at least 3 wraps on a 20X mandrel.

3. Apparatus

a. A series of mandrels or an adjustable mandrel shall be required to have diameters equivalent to 20 times the diameter of the wires to be tested.

b. A two pound tensioning weight is required.

c. A suitable dielectric tester with variable voltage at a frequency of 60 Hertz will be required for electrical testing.

4. Procedure

a. This test shall be conducted on finished wire at room temperature.

b. One end of the specimen shall be secured to the 20X mandrel and the other end weighted with a 2 lb. tension.

c. The mandrel shall be rotated until the full length of the specimen is wrapped around the mandrel.

d. The process shall be repeated in reverse until the full length of the specimen is again wrapped around the mandrel with the surface of the specimen previously outside during the first wrapping now next to the mandrel.

e. Procedure 5.9.3.3 and 5.9.3.4 shall be repeated until five bends in each direction have been formed in the same section of the specimen.

5. Results

a. At the completion of the test, the insulation of the specimen shall be examined for fraying or other physcal damage.

b. The test specimen will be tested for dielectric withstand of 2.2kV/5 seconds, as per test procedure TMB-3.

c. This sample may subsequently be used for evaluation in the thermal shock test.

6. Specification

a. All wires must pass a dielectric withstand of 2.2kV/5 seconds after the mandrel test.

J Method TMB-7

Thermal Shock Resistance

1. Scope

This method is intended for use in determining the stability of the finished wire to exposure at extremes of high and low temperature, and to the shock of cyclical exposures to these extremes. Effects of thermal shock are mainly exhibited by changes in electrical characteristics due to mechanical displacement or rupture of conductors or of insulating materials.

2. Specimen

A sample of wire (12 and 22 gauges) of at least 6 inches in length. This wire may be from the mandrel test.

3. Apparatus

a. Two temperature chambers shall be maintained at -65°F and 1250°F, and shall be placed in close proximity to each other.

b. These ovens shall be of such design that a wire sample may be inserted quickly without altering the temperature by more than $\pm$ 2 percent.

c. A suitable dielectric withstand tester shall be required for evaluating the wire after the thermal shocking.

4. Procedure

a. Separate chambers shall be used for the extreme temperature conditions. Direct heat conduction to the specimen should be minimized.

b. The time of transfer from one chamber to the other should be less than two minutes.

c. The specimen shall be subjected to 1250°F for 20 minutes, then -65°F for 20 minutes. This cycle will be repeated five times in a continuous manner.

d. After 5 cycles, the wire will be checked for physical damage and shall be required to pass a dielectric withstand of 2.2kV for 5 seconds. The result will include a pass or fail result, and the breakdown voltage if the specimen failed.

5. Results

a. A description of any damage to the sheath or insulation shall be given.

b. The results of the dielectric withstand test shall be included.

6. Specification

The sample shall be required to pass a dielectric withstand of 2.2kV for 5 seconds after 5 cycles of 1250°F/-65°F.

(This test is based on Method 107C of Mil-Std-202D)

K Method TMB-8

Abrasion Resistance

1. Scope

This method is intended for use in determining the resistance to abrasion of the insulation package while on the finished insulated wire or cable.

2. Specimen

a. A 12 and 22 AWG finished wire of approximately 18 inches in length shall be tested.

b. A portion of the 12 and 22 AWG star quad cables shall also be tested for abrasion resistance.

3. Apparatus

A Jenco Abrader shall be used with a garnet abrasive tape, using the test apparatus specified in test method Mil-T-5438.

4. Procedure

a. The apparatus will consist of an abrasion tester and garnet abrasive tape as specified in Mil-T-5438.

b. An inch of the insulation and outer covering shall be removed from one end of an 18 inch specimen of the finished wire. The stripped end of the specimen shall be fastened to the clamp over the tape-feed reel of the tester and the other end shall be attached to a 1 lb tension load for the 18 through 22 gauge and a 2 lb. tension load for the 12 through 16 gauge wires.

c. At the start-up for each test measurement, the centre of a conducting strip on the abrasive tape shall be at the point of contact with the wire.

d. The reading of each measurement shall be the length of abrasive tape, in inches, to come in contact with the wire to the point where the machine stops.

e. After each reading the specimen shall be moved forward 2 inches and rotated clockwise 90 degrees.

f. Eight readings shall be obtained for each specimen. An average shall be obtained by calculating the arithmetic mean of all the readings which are individually less than the arithmetic mean of all the eight readings per specimen. This average shall define the abrasion resistance of the wire under test.

5. Results

a. Each of the eight readings shall be recorded and an average shall be obtained by calculating the arithmetic mean of all the readings which are individually less than the arithmetic mean of all the eight readings per specimen.

b. The weight, tension load, and type of abrasive tape used shall be recorded.

6. Specification

All insulated wire must be able to pass greater than 25 inches of garnet abrasive tape, both before and after thermal exposure. Only 12 and 22 gauge wire are required to pass this specification, as these will represent the best and worst condition respectively.

(This test method is based on Method 2211 of Fed-Std-228).

L Method TMB-9

Vibration Resistance

1. Scope

This method is intended for use in determining the effect on wire and connector parts of vibration in the frequency range of 10 to 2000 Hertz (Hz) as may be encountered in aircraft and missiles.

2. Specimen

a. This test shall be carried out on finished insulated wires of approximately 8 inches in length, both before and after exposure to 1250°F for 1000 hours.

b. The star quad connected cables shall be tested under high frequency vibration after the life cycle testing is complete.

3. Apparatus

a. A suitable jig shall be used to support the specimen at both ends while leaving the centre portion free to vibrate.

b. An Unholtz-Dickie 89B Vibration System or other suitable apparatus shall be calibrated and used.

c. A suitable d.c. power supply shall be available in series with a resistor to indicate any current shorts during vibration testing.

d. A suitable dielectric withstand test machine shall be used to evaluate electrical deterioration due to vibration.

4. Procedure

a. The wire specimens shall be mounted in a suitable clamping jig consisting of 2 metal plates having matched 90° V wedges cut in each plate to hold the sample at both ends. The formed square clamping area between the two plates shall be 0.10"-2.20" less than the diameter of the wire. Suitable jigs are shown in Figures 53 and 54.

b. For the wire and connector systems the receptacle shall be held in the mounting jig, and the plugs shall be engaged with the receptacles and held by normal locking means only. A minimum of 8 inches of wire or cable shall be unsupported behind the rear of each connector, at which point the cable shall be clamped to the jig.

c. All contacts shall be wired in series with 1.0 ± 0.1 amperes allowed to flow. A suitable instrument shall be employed to monitor current flow and to indicate discontinuity of contact or interruption of current flow.

d. The vibration input shall be monitored on the mounting fixture in the proximity of the support points of the specimen.

e. The clamp plates with the wire shall be mounted on the side and the top of a suitable vibration system (e.g. Unholtz-Dickie 89B Vibration System), for parallel and perpendicular testing.

f. A suitable d.c. power supply shall be used to pass a small current (e.g. 1.5 amperes) through the wire in series with a resistor to detect any short of the wire to the metallic sheath.

g. The sample wire shall be vibrated for 4 hours in the direction of each axis, and the vibration frequency shall be varied logarithmically between the limits of 10 to 2000 to 10 Hz, each complete sweep being of 20 minutes duration.

g. (continued)
The wire shall be vibrated 0.06" double amplitude from 10 to 81 Hz and at 20g peak vibration from 81 to 2000 Hz.

h. A critical resonant frequency is that frequency at which any point on the specimen is observed to have a maximum amplitude more than twice that of the support points. Resonant frequencies shall be determined either by monitoring parameters such as contact opening, or by use of resonance-detecting instrumentation.

i. Vibration shall be carried out on the wire as fabricated and on a wire after aging at 1250°F for 1000 hours.

j. Samples shall be tested for a dielectric withstand of 1.2kV for 5 seconds after vibration testing, according to method TMB-3.

5. Results

a. A description of the mounting jig shall be given.

b. The method of determining resonance shall be specified.

c. The results of the dielectric withstand test shall be given.

6. Specification

Samples from this test will be tested for shock resistance in the same test jig immediately subsequent to this test, at which time it must pass a dielectric withstand of 1.2kV/5 seconds on the aged samples, according to test TMB-3.

(This test method is based on Method 204B of Mil-Std-202D).

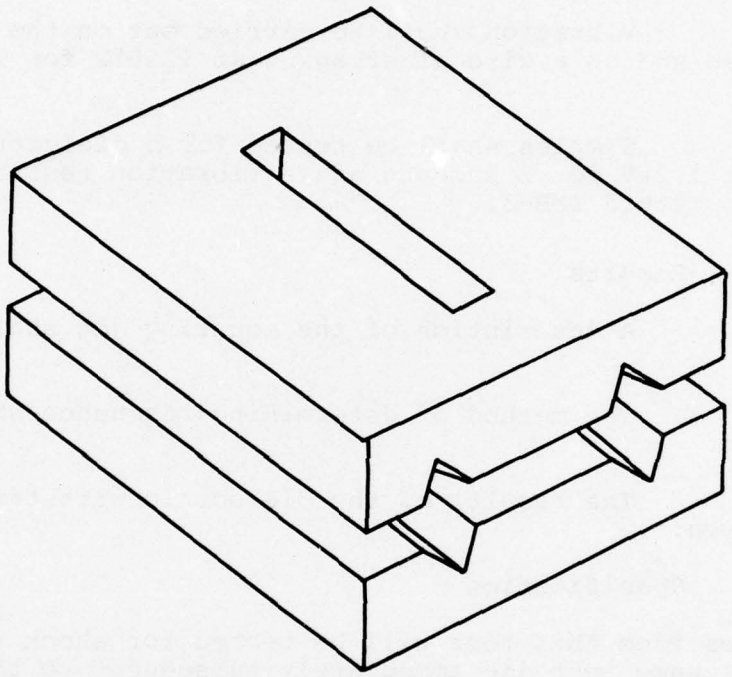


Figure 53

Mounting jig for vibration and shock testing of insulated wire.

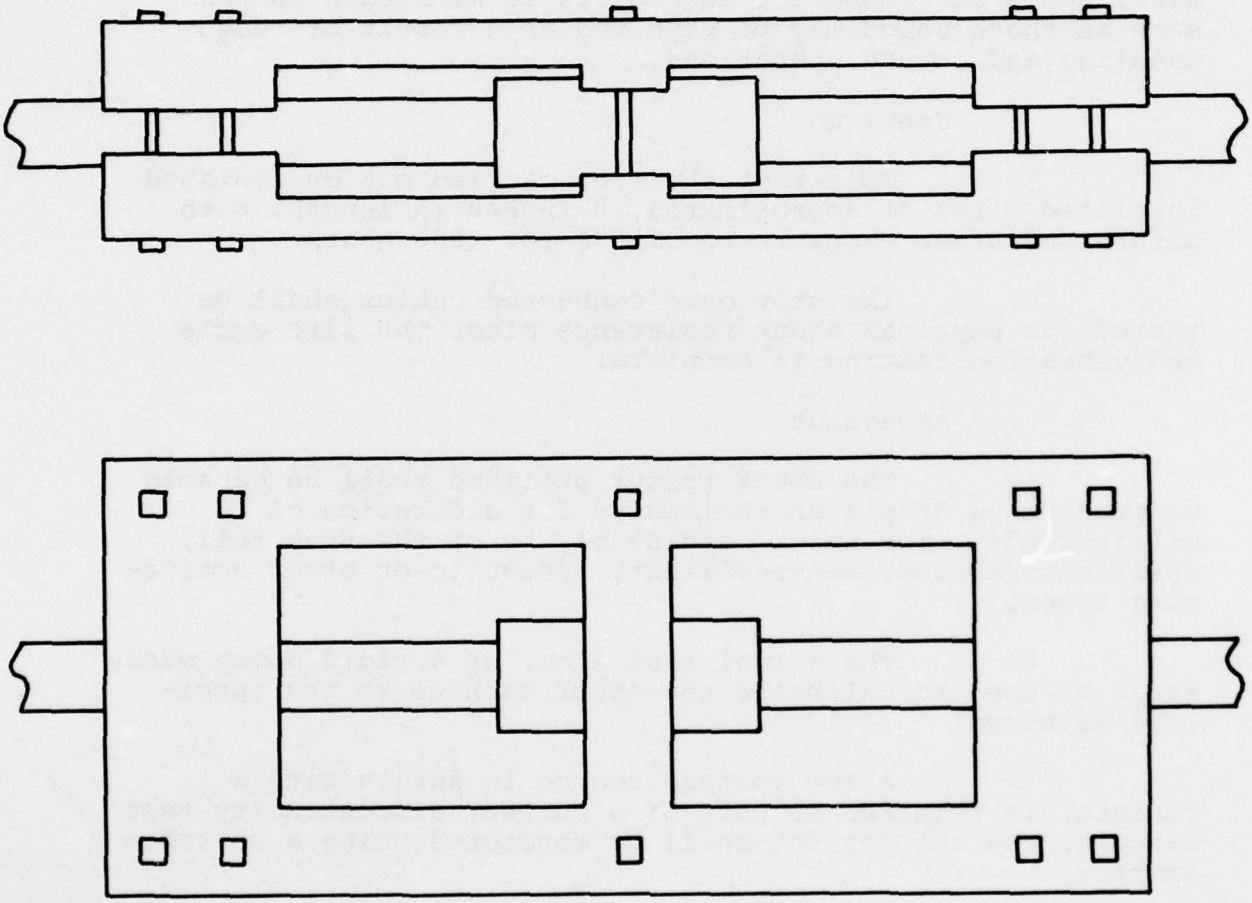


Figure 54

Mounting jig for vibration and shock testing of star quad connected cable.

M Method TMB-10

Physical Shock Resistance

1. Scope

This method is intended to determine the ability of wire, cable and connector assemblies to withstand shocks such as those which may be expected as a result of rough handling and in-use operations.

2. Specimen

a. This test shall be carried out on finished insulated wires of approximately 8 inches in length, both before and after exposure to 1250°F for 1000 hours.

b. The star quad connected cables shall be tested for physical shock resistance after the life cycle and vibration testing is complete.

3. Apparatus

a. The shock tester utilized shall be capable of producting 50 g's shock impulse for a duration of 11 $\pm$ milliseconds. The shock machine may be of the free fall, resilient rebound, non-resilient, hydraulic or other activating types.

b. The actual test item, or a rigid dummy mass, shall be used to calibrate the shock machine to the specified waveform.

c. A low voltage source in series with a resistor is required as part of a current discontinuity test circuit, and the current shall be monitored using a suitable ammeter.

4. Procedure

a. The wire specimens shall remain mounted in the suitable clamping jig used for vibration testing. This jig shall consist of two metal plates having matched 90° V wedges cut in each plate to hold the samples at both ends. The formed square clamping area between the two plates shall be approximately 0.10"-0.20" less than the diameter of the wire.

b. For the wire connector systems the receptacle shall be held in the mounting jig, and the plugs shall be engaged with the receptacles and held by normal locking means only. A minimum of 8 inches of wire or cable shall be supported behind the rear of each connector, at which point the cable shall be clamped to the jig according to section 5.13.3.1.

c. All contacts shall be wired in series with 1.0 ± 0.1 amperes allowed to flow. A suitable instrument shall be employed to monitor current flow and to indicate discontinuity of contact or interruption of current flow.

d. The specimen and fixture shall be mounted on the Avco Shock machine (or other suitable equipment). The test load shall be distributed uniformly on the test platform in order to minimize the effects of unbalanced loads.

e. The specimen shall then be given six half sine shock pulses of 50g peak value for a duration of 11 milliseconds in both the horizontal and vertical axes of the wire specimen.

f. The specimen shall then be tested for a dielectric withstand of 1.2kV for 5 seconds according to test method TMB-3.

5. Results

a. There shall be no indication of a short in the monitoring circuit during or after the shock test.

b. The wire shall be removed from the clamping jig and examined under a microscope to identify any signs of deterioration to the knitted sheath or braided ceramic insulation.

c. A detailed description of the clamping jig shall be given.

6. Specification

Wire specimens, after being tested under vibration and physical shock shall be required to pass a dielectric withstand of 1.2kV/5 seconds. The connected star quad cable shall be required to pass a dielectric withstand test of 1.2kV/5 seconds after shock testing is complete.

(This test is based on test Method 213A of Mil-Std-202D).

N Method TMB-11

Sublimation

1. Scope

This method is intended for use in determining the visual presence of fuming evolving from the finished wire as compared to other commercially available high temperature wires.

2. Specimen

The specimen shall consist of at least a 6 inch length of finished insulated wire, of both a 12 and 22 AWG size.

3. Apparatus

The apparatus shall consist of a tube furnace and controller capable of maintaining 1250°F, where the sample may be suspended in the constant temperature zone. A low flow rate of air will be maintained throughout the test and will impinge on a metallic "cold finger" which will be placed downstream, just outside the high temperature zone.

4. Procedure

a. The sample shall be suspended in the constant temperature zone of the Vycor tube furnace to minimize direct heat conduction.

b. The temperature will be raised slowly to 1250°F ± 20°F.

c. A continuous observation will be recorded including presence of sublimate on cold finger, colour and description of sublimate, and a qualitative estimate of amount of sublimate.

d. This test will be carried out on the 12 and 22 gauge wires.

e. The samples will be held at 1250°F for a total of 24 hours.

f. At least one commercial wire shall be tested as a comparison.

5. Results

A description of the type and amount of sublimation shall be recorded for both the commercial and experimental wires.

6. Specification

The wire shall exhibit little or no sublimation.

O Method TMB-12

Degree of Flexibility including Flex-Fatigue Life

1. Scope

This method is intended for use in determining any physical or electrical deterioration occurring during flexing and high temperature exposure.

2. Specimen

The specimen shall be of at least 24 inches in length and testing shall be carried out on both 12 and 22 AWG finished wires for evaluation purposes.

3. Apparatus

a. The flexing apparatus shall consist of two mandrel wheels positioned closely to each other so that the wire can be wound around in a tight S-shape configuration. These mandrel wheels shall require little or no tension to turn (Figure 55).

b. The mandrel wheels shall consist of several grooves of varying size so that an approximate 20X and 10X wire diameter size is available for each wire tested.

4. Procedure

a. Before thermal exposure, each wire shall be flexed 10 times around two shaped 20X diameter pulleys and tested for a dielectric withstand of 2.2kV/5 seconds. This flexing will be repeated using 10X diameter pulleys, and the dielectric withstand test will be repeated.

b. The wire will then be placed in a muffle furnace or a Vycor tube furnace at 1250°F and removed for testing at 250, 500 and 1000 hours. After each exposure, the flexing will be performed using the 20X and 10X diameter pulleys. The dielectric withstand test of 2.2kV/5 sec. will be determined after each flexing.

c. After 1000 hours exposure, the insulation resistance, dielectric breakdown voltage and abrasion resistance of the wire shall be determined.

5. Results

a. Any breaking or fraying of the braided yarns shall be recorded.

b. Any breaking of the knitted Inconel outer mesh shall be recorded.

c. If dielectric breakdown occurs during the testing, the thermal exposure and the size of mandrel used just prior to testing shall be recorded.

6. Specification

The wires shall be required to pass a dielectric withstand test of 2.2kV for 5 seconds after every test condition.

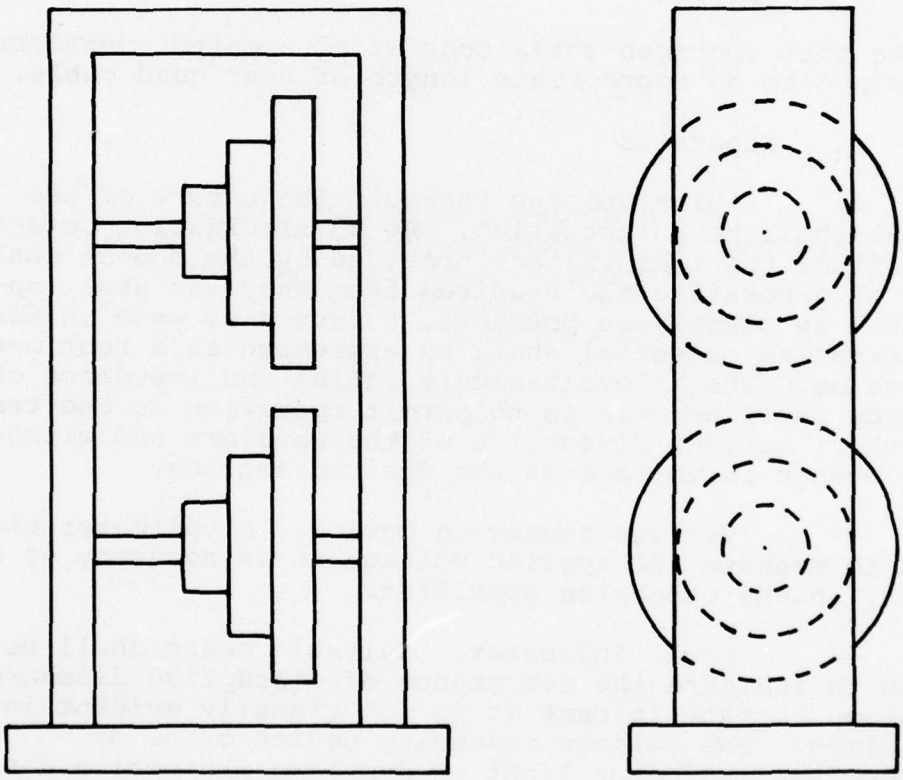


Figure 55

Mandrel apparatus used in flexibility testing.

P Method TMC-1

STEADY STATE VOLTAGE WITHSTAND

1. Scope

This method is intended for use in determining that the component part can operate safely at its rated voltage. It is not intended that this test cause dielectric breakdown or that it be used for detecting corona.

2. Specimen

The test specimen shall consist of a mated connector unit, joined to an appropriate length of star quad cable.

3. Apparatus

a. High Voltage Source. The nature of the potential shall be as specified. As an alternating potential is specified, the test voltage provided by the source shall be capable of generating the required frequency and shall approximate, as closely as possible, a true sine wave in form. The alternating potential shall be expressed as a root mean square value. The Kilovolt-ampere rating and impedance of the source shall be such as to permit operation at the test load without serious distortion of the waveform and without serious change in voltage at the desired setting.

b. Voltage Measuring Device. A voltmeter shall be used to measure the applied voltage to an accuracy of at least 5%, unless otherwise specified.

c. Fault Indicator. Suitable means shall be provided to indicate the occurrence of disruptive discharge and leakage current in case it is not visually evident in the specimen. The voltage measuring device of b. or an appropriate indicator light or overload protective device may be used for this purpose.

4. Procedure

a. Specimens shall be subjected to the test voltage of the magnitude and nature (a.c. or d.c.) specified, which shall be applied between mutually insulated portions of the specimen or between insulated portions and ground.

AD-A046 406 CANADA WIRE AND CABLE TECHNOLOGY DEVELOPMENT DIV POI--ETC F/G 9/1
DEVELOPMENT OF HIGH TEMPERATURE (1250 F) WIRE AND CONNECTORS.(U)
JUL 77 M A DUDLEY, F D BAYLES, P O SCADDING F33615-75-C-2040

UNCLASSIFIED AFAPL-TR-77-25 NL

3 OF 4
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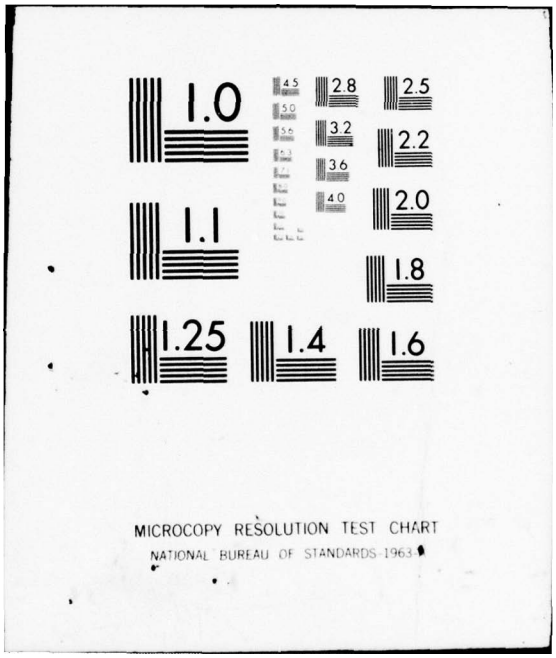
1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
5.6
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

b. The test voltage shall be raised from zero to the specified value as uniformly as possible, at a rate of approximately 100 volts (rms or d.c.) per second.

c. The test voltage shall be maintained at the specified value for a period of 60 seconds. Upon completion of the test, the test voltage shall be removed instantaneously.

d. During the dielectric withstand test, the fault indicator shall be monitored for evidence of disruptive discharge and leakage current.

5. Results

The following details are to be specified:

1) Magnitude of test voltage
2) Nature of potential (a.c. or d.c.)
3) Duration of application of test voltage
4) Points of application of test voltage
5) Failure phenomena, if noted.

6. Specification

Contact spacing and configuration shall permit a contact to contact and contact to shell steady state working voltage of 250 volts rms at 400 Hertz.

(This method is written in accordance with Mil-Std-202E, Method 301).

Q Method TMC-2

Insulation Resistance

1. Scope

This method is intended for use in measuring the resistance offered by the insulating members of a component part to an impressed direct voltage tending to produce a leakage of current through or on the surface of these members.

2. Specimen

The test specimen shall consist of a mated connector unit joined to an appropriate length of star quad cable.

3. Apparatus

a. Insulation resistance measurements shall be made on an apparatus suitable for the characteristics of the component to be measured such as a megohm bridge, megohm meter, insulation resistance test set or other suitable apparatus. The direct potential applied to the specimen shall be 500 Volts ± 10%.

b. The test procedure shall be performed on the unit both at room temperature and in a suitable oven capable of maintaining the service temperature of 1250°F (677°C).

4. Procedure

a. A direct potential of 500 Volts ± 10% shall be applied to the specimen through appropriate contacts to the cable leads of the specimen.

b. The insulation resistance measurements shall be made between the mutually insulated points or between insulated points and ground.

c. The measurement shall be made within 1 minute of application of uninterrupted test voltage.

d. After performance of a. to c. the specimen shall be inserted into a furnace and brought to an ambient temperature of 1250°F ± 10°F at an average rate of rise of 2°C/second.

e. After holding for 5 minutes at 1250°F, procedures a. to c. shall be repeated.

5. Results

The following details are to be specified:

1) Test potential
2) Points of measurement
3) Electrification time
4) Measurement error at the insulation resistance value required, if other than 10%.

6. Specification

The insulation resistance between any two contacts and between the shell and any contact of mated connectors shall be 500 megohms, under all conditions.

(This method is written in accordance with Mil-Std-202E, Method 302, Condition B).

R Method TMC-3

Mated Contact Resistance

1. Scope

This method is intended to determine the resistance offered to a flow of current during its passage between the electrical contacting surfaces of mated contracts. It is important to keep the specimen free from vibration, and to prevent changes in normal contact pressure which might result from improper application of test jigs or clamps.

2. Specimen

The test specimen shall consist of a mated connector unit joined to an appropriate length of star quad cable.

3. Apparatus

a. A Kelvin bridge, voltmeter/ammeter, ammeter/potentiometer or other suitable means shall be employed to measure contact resistance.

b. An oven shall be provided capable of maintaining 1250°F $\pm$ 10°F.

c. A representative test circuit is shown in Figure 56, which includes an optional series resistor to ensure that the open circuit test voltage is not exceeded.

4. Procedure

a. The test specimen shall be appropriately mated, and fitted with joined leads so as to ensure electrical continuity during testing.

b. A test voltage shall be passed through the circuit such that a direct current of 2.0 Amps is developed.

c. Upon achieving the current value of 2.0 Amps, the specimen shall be held for 1 minute before measuring contact resistance.

d. The test specimen with leads shall then be inserted in the test furnace and, under electrical load, raised to the service temperature of 1250°F $\pm$ 10°F standard rate of rise of $\sim$4°F/second.

e. Upon attaining the service temperature of

d, the specimen shall be held for 1 minute and then tested for contact resistance.

f. The specimen shall then be held, without electrical load, for 1000 hours at 1250°F ± 10°F.

g. After the thermal exposure of f., the specimen shall be removed from the furnace and allowed to cool to room temperature where the contact resistance shall again be measured under electrical load.

h. All measurements under all conditions shall be made in duplicate.

5. Results

The following details are to be specified:

1) Method of connection
2) Test current
3) Specimen thermal history
4) Resistance measurements

6. Specification

The mated contact resistance at temperature, and after aging, shall not exceed 125 millivolts.

(This method is written in accordance with Mil-Std-202E, Method 307 and Mil-C-23216C (Navy), Section 4.6.5).

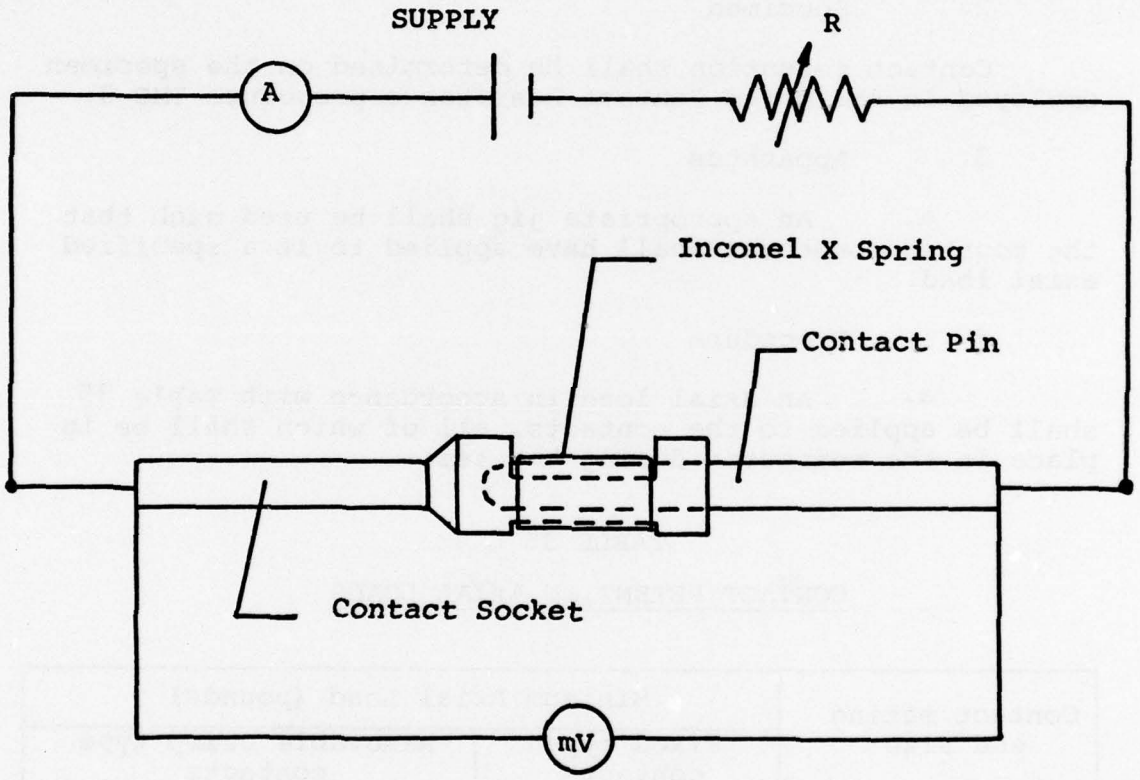

Figure 56

Test circuit for determination of contact resistance.

S Method TMC-4

Contact Retention

1. Scope

This method is intended for use in determining the retention of contact strength after aging.

2. Specimen

Contact retention shall be determined on the specimen employed in the Mated Contact Resistance procedure TMC-3.

3. Apparatus

a. An appropriate jig shall be used such that the mounted connector shall have applied to it a specified axial load.

4. Procedure

a. An axial load in accordance with Table 35 shall be applied to the contacts, all of which shall be in place in the connector during the test.

TABLE 35

CONTACT RETENTION AXIAL LOADS

Contact mating end size	Minimum Axial Load (pounds)	
	Fixed type contacts	Removable crimp type contacts
16	10	25
12	15	25
8	20	40
4	20	50
0	25	50

b. The load shall be applied at a rate of ∿1 pound/second until the specified load has been reached.

c. The load shall be removed and the contacts examined for visual displacement in their inserts.

d. Procedures a. to c. shall be repeated on the specimen after the thermal aging specified in Procedure TMC-3.

5. Results

The following details are to be specified:

1) Number of contacts under test
2) Thermal history of contacts
3) Test load applied
4) Contact mating end size
5) Visual displacement

6. Specification

None cited in Statement of Work.

(This method is written in accordance with Mil-C-00515 para. 4.7.16 and para. 3.6.17 (fixed) type).

T Method TMC-5

Ozone Resistance

1. Scope

In view of the nature of the materials used in the fabrication of the high temperature resistant connectors, ozone resistance testing is inapplicable.

U Method TMC-6

Resistance to Hydraulic Fluids and Kerosene (JP-4 or JP-5)

1. Scope

This method is intended for use in determining the resistance of the mated connector to physical deterioration when immersed in hydraulic fluid or jet fuel.

2. Specimen

The specimen shall be a mated connector fitted with cable and appropriate lengths of seamless cable jacket to eliminate ingress of test fluid.

3. Apparatus

a. A suitable container shall be provided having sufficient volume to hold the test fluid so that the specimen will be totally immersed with ends of jacket above liquid level.

4. Procedure

a. The specimen shall be mated and fully immersed in either a) hydraulic fluid conforming to Mil-H-5606 or b) Kerosene Specification JP-4 or JP-5, for a period of 20 hours with ends of jacket above liquid level.

b. After removal from the fluid, the connectors shall remain for 1 hour in free air at room temperature.

c. The connectors shall then be examined in the mated mode for signs of degradation/deterioration.

d. The connectors shall then be unmated and examined for degradation/deterioration of interfacial cavity and mating surfaces.

5. Results

The following details are to be specified:

1) Test fluid
2) Immersion time

6. Specification

None cited in Statement of Work

(This method is written in accordance with Mil-C-005015 (Navy), para. 4.7.14).

V Method TMC-7

Salt Spray Resistance

1. Scope

This method is intended for use in determining the resistance of connectors to accelerated corrosion, which allows a prediction of relative service life and behaviour of the connector materials.

2. Specimen

The test specimen shall consist of a mated connector unit. Consideration should be given to using the specimen employed in the hydraulic fluid resistance test, TMC-6.

3. Apparatus

The apparatus shall conform to Mil-Std-202E, Method 101D.

4. Procedure

a. The operation of the salt spray cabinet shall be adjusted in accordance with Mil-Std-202E, Method 101D.

b. The specimen shall be suspended within the cabinet so that it may be completely exposed to the spray, and in such a manner as to allow full drainage of salt solution from the metal surfaces but with ends of jacket outside of cabinet.

c. The specimen shall be exposed continuously for 48 hours, with a visual inspection after 24 hours.

d. After exposure the specimen shall be rinsed in warm water and dried.

5. Results

a. The specimen shall be examined in the mated mode for corrosion defects.

b. The specimen shall be disconnected and examined for internal corrosion defects.

6. Specification

None cited in Statement of Work.

(This method is written in accordance with Mil-Std-202E, Method 101D, condition B).

W Method TMC-8

Corona Resistance

1. Scope

A test procedure for corona resistance is under consideration as part of the Life Cycle Test, TMD-1, once facilities availability has been established.

X Method TMC-9

Splashproof Sealing
(Interfacial and Shell)

1. Scope

This method is intended for use in determining the integrity of the interfacial sealing of connector halves and shell metal-to-metal seals.

2. Specimen

The test specimen shall consist of a mated connector unit. Consideration should be given to using the test specimen after procedures TMC-6 and TMC-7 modified for attachment to test jig.

3. Apparatus

a. A test jig shall be assembled, consisting of a pressure resistant fluid reservoir fitted with pressure gauge and pumping means and an outlet line with adapter appropriate for connection to the end bell or seamless cable jacket of the specimen.

b. A short length of test line shall be provided being fitted on one end with a means of attachment to the specimen end bell or seamless cable jacket and a means of sealing the other end.

c. The test fluid shall be water.

4. Procedure

a. The test specimen shall be modified to allow attachment to the test jig.

b. The test specimen shall be mounted to the test jig, ensuring that the connections between the specimen and feed line are fully sealed.

c. A pressure of 2 psig differential shall be placed on the fluid and held for 1 hour.

d. The specimen shall be examined for compliance.

e. One half of the specimen shall be mounted to the test jig as noted in b. and pressure applied per c.

f. The specimen interface shall be examined for compliance.

5. Results

The following details are to be specified.

1) Test fluid
2) Pressure applied
3) Test duration
4) Compliance with specification

6. Specification

Interfacial sealing and metal-to-metal sealing of mated and unmated connector halves to provide a splashproof seal. It is recommended that escaping moisture during the test period shall not exceed 5 drops.

Y Method TMD-1

Life Cycle Testing

1.

The life cycle test is a combination of a modified Flight Profile, maximum continuous potential rating, and design life tests which will be carried out on the 12 and

22 gauge star quad connected cables. This test includes 1000 hours at 1250°F under 600 Volts rated power, low temperature exposure, as well as pressures corresponding to flight altitudes of 110,000 feet.

2. Specimen

The specimen shall consist of two sections of four insulated conductors, cabled into a star quad configuration, terminated by a male and female connector respectively. These connectors shall be mated and there shall be approximately 2 feet of cable on each side of the mated connectors.

3. Apparatus

a. The apparatus shall consist of a cold chamber and a furnace.

b. The high temperature furnace shall be capable of maintaining 1250°F ± 10°F as well as a reduced pressure of 7 mm Hg. Two port holes will be necessary for potential leads attached to each end of the conductor which will be connected to a 600 Volt a.c. power source.

c. The cold chamber shall be capable of maintaining -65°F ± 2°F as well as having exit port openings for electrical leads.

d. A 100% relative humidity chamber with electrical port holes shall be required.

4. Procedure

The following life cycle test shall be used to evaluate the connector terminated preproduction wire assembly, which is to be carried out while each conductor is carrying 600 Volts a.c.

Life Cycle Test
(Carried Out Under Rated Power)

1. Expose to 1250°F for 88 hours.
2. Expose to 100% RH at room temperature for one hour.
3. Expose to -65% for one hour. All temperature changes shall take place at 2°C ± 1°C/second.
4. Expose to 1250°F and 7 mm Hg for one hour. Cool to room temperature.
5. Expose to 50% RH at room temperature for one hour.
6. Expose to 1250°F for 64 hours.
7. Repeat steps 2 through 5.
8. Repeat the above procedure for 6 complete cycles.
9. Expose the wire for an additional 76 hours at 1250°F under rated power.

At the completion of the Life Cycle Test the wire harness assembly will have been exposed to the maximum service temperature for 1000 hours while under rated power and will have been subjected to 12 cycles of a modified version of the Flight Profile Test.

During this life test, a continuous 600 V a.c. potential with a current of not greater than 1 Amp shall be applied to each conductor. This may be provided by means of four separate power supplies or by soldering the conductors into one long series circuit. An ammeter shall be used to indicate any fluctuations or irregularities in the current flow during the testing.

5. Results

a. Any irregularities or shorts in the current flow shall be recorded, and an attempt shall be made to identify the cause of failure.

b. A description of any physical deterioriation shall be recorded.

c. The wire/harness assembly shall be required to meet the insulation resistance and dielectric withstand specifications as per methods TMB-1 and TMB-3 respectively after completion of the Life Cycle Test.

d. These wire assemblies shall be retained for vibration and shock testing after Life Cycle Testing.

6. Specification

The 12 and 22 AWG wire/harness assemblies shall be required to complete the Life Cycle Test without fault, and to pass the specifications for insulation resistance and dielectric withstand after life testing.

Z Method TMD-2

Vibration Resistance

1. Scope

This method is intended for use in determining the effect on the mated connector/wire harness of vibration in the frequency range of 10 to 2000 Hertz as may be encountered in aircraft or missiles.

2. Specimen

The test specimens shall consist of mated connector and joined star quad sheathed 12 and 22 AWG cables of total length of approximately 4 feet. The vibration resistance testing shall be carried out after the Life Cycle (method TMD-1) is completed.

3. Apparatus

The mounting jig and vibration system as described in method TMB-9 will be used.

4. Procedure

The prototype assembly shall be mounted, vibrated and tested in the manner described in the test method TMB-9.

5. Results

a. A description of the mounting jig shall be given.

b. The method of determining resonance shall be specified.

c. The results of the dielectric withstand test shall be given.

6. Specification

Samples from this test will be tested for shock resistance in the same test jig immediately subsequent to this test, at which time it must pass a dielectric withstand of 1.2kV/5 seconds according to test TMB-3.

(This test method is based on Method 204B of Mil-Std-202D).

AA Method TMD-3

Physical Shock Resistance

1. Scope

This method is intended for use in determining the ability of the mated connector/wire harness to withstand shocks such as those which may be expected as a result of rough handling and in-use operations.

2. Specimen

The test specimens shall consist of the mated connector/wire harness assemblies which are joined star quad sheathed 12 and 22 AWG cables of total length of approximately four feet. The physical shock resistance testing shall be performed after completion of the life cycle (method TMD-1) and vibration resistance (TMD-2) tests.

3. Apparatus

The mounting jig and shock machine shall be used as described in test methods TMB-9 and TMB-10 respectively.

4. Procedure

The prototype assembly shall be mounted, shock tested and evaluated according to test method TMB-10.

5. Results

a. There shall be no indication of a short in the monitoring circuit during or after the shock test.

b. The results of the dielectric withstand tests shall be recorded.

c. The wire/connector harness shall be removed from the test jig after shock testing to identify any signs of damage or deterioration to the wire or connector. The connector shall be disassembled to evaluate whether any internal damage has occurred.

d. A detailed description of the clamping jig shall be given.

6. Specification

The connector/wire harness assembly shall be capable of passing a dielectric withstand test of 1.2kV for 5 seconds from conductor to sheath, as well as conductor to conductor.

(This test is based on test Method 213A of Mil-Std-202D).

BB Method TMD-4

Flexibility

1. Scope

This method is intended for use in determining the flexibility of the cabled star quad 12 and 22 AWG connected harnesses.

2. Specimen

The specimen shall consist of a length of at least 36 inches of star quad cable, of both the 12 and 22 AWG wire size. Enough sample is required to allow at least two full turns around a 20X mandrel.

3. Apparatus

a. A mandrel of diameter 20 times the diameter of the cable to be tested is required.

b. A ten pound tensioning weight is required.

c. A suitable dielectric tester with variable voltage at a frequency of 60 Hertz will be required for dielectric testing and flexing.

4. Procedure

a. This test shall be conducted on cabled wire at room temperature.

b. One end of the specimen shall be secured to the 20X mandrel and the other end weighted with the 10 pound tensioning weight.

c. The mandrel shall be rotated until the full length of the specimen is wrapped around the mandrel.

d. The process shall be repeated in reverse until the full length of the specimen is again wrapped around the mandrel with the surface of the specimen previously outside during the first wrapping now next to the mandrel.

e. Procedure c. and d. shall be repeated until five flexes in each direction have been formed in the same section of the cable specimen.

5. Results

a. At the completion of the flexing, the insulation of the specimen shall be examined for fraying of the ceramic insulations, binding of the Inconel sheaths or other physical damage.

b. The test cable shall be required to pass a dielectric withstand of 2.2kV for 5 seconds, as per test Method TMB-3. The dielectric withstand shall be applied across the conductor to the sheath, as well as from conductor to conductor.

6. Specification

Both the 12 and 22 AWG cables shall be required to pass a 2.2kV dielectric withstand for 5 seconds after the cable flexing test.

Program Conclusions

A service temperature of 1250°F., as specified in the Statement of Work, creates a severe performance stress; when this is coupled with a requirement of 200 thermal cycles, an especially damaging environment can result. It was concluded early in the program that materials commercially available at that time, and wire configurations assembled from them, were totally unsuitable for service at 1250°F. This necessitated the development or procurement of a totally new family of components to fulfill the requirement of the Statement of Work.

A. Components

Considering the conductor, it has been demonstrated that careful selection of an appropriate conductive core and an oxidation resistant sheath combination is necessary to meet and exceed all design requirements; the results reported significantly extend and support the results of other studies in this area.

Recognizing the potentially incompatible requirements of thermal resistance, flexibility and long term dielectric strength, it was concluded that an insulation package based on multiple layers of inorganic braids surrounding a core of inorganic primary dielectric tapes would offer the best opportunity for success. This in turn led to the development of a novel organometallic tape made from mica impregnated with poly(carboranesiloxane)s, which satisfactorily met all design requirements. Further the development of this tape demonstrated the first potentially commercial application of this polymer, which heretofore has been little more than a laboratory curiosity. Although it was recognized that the approach of using braided yarn over and under the primary dielectric was sound, the existing inorganic yarns suffered from poor strength and workability. Fortuitously, a new family of continuous filament yarns became available during the program, which largely overcame these objections. One particular yarn, an alumina boria silica compound was particularly satisfactory in meeting the performance requirements.

Finally, it was demonstrated that satisfactory abrasion resistance and flexibility could be achieved using a knitted, fully-fashioned Inconel alloy sheath.

As a subprogram of the components development it was felt that significant cost and performance advantages would accrue if commercial conductors such as copper could be upgraded using alternate approaches to sheathing which is a present but unsatisfactory practice. This conclusion was successfully substantiated by the development of a copper conductor having a surface phase stabilized with an intermetalloid of copper, which shows early promise as a means of significantly extending the service ceiling of copper.

B. Assembly

All performance requirements of the Statement of Work were met or exceeded by a design configuration of a sheathed conductor with multiple layers of inorganic braids and organo-metallic polymer impregnated mica tapes, sheathed and protected in turn with knitted Inconel wire. This substantiates the original conclusions made as to the appropriate direction of the program efforts.

Concurrently, and under subcontract, a family of connectors was successfully developed which are compatible with all wire/cable configurations, and which meet or exceed all design requirements. The performance of these connectors successfully substantiates all design configurations and material decisions made by the subcontractor.

In conclusion, it has clearly been demonstrated that electrical wire and connectors can be designed and fabricated to meet the Statement of Work for a cyclical thermal environment of 1250°F., coupled with other performance requirements expected from harnesses intended for aerospace vehicles. The design of these components is advantageous in that it does not necessitate a significant change from present assembly practice; rather, the performance improvement accrues from significant advances in subcomponent developments.

Recommendations

In light of the technical successes in both cable and connector development, it is strongly recommended that further work be initiated to complete the program to demonstrate the ability to both manufacture commercial lengths of wire, and mate these cables to the connectors. Further, performance testing of harness assemblies is also recommended.

Serious consideration should be given to the applicability of the silver core/nickel alloy sheathed wire developed in this program to other ultra high temperature conductor requirements.

Further research and development is recommended in the bringing of the poly(carborane siloxane)/mica composite as a new, stable, general purpose composite dielectric for high temperature aerospace and other end-use applications.

Serious consideration should be given to continuing support for the development of commercially useful conductors developed from the surface stabilized copper demonstrated as a prototype during the program.

Since both the cable and connector have been shown by the contractor and subcontractor, under self-funded development, to possess unusual characteristics under direct flame and nuclear environments, a strong recommendation is made to initiate modification developments to yield a safe harness system for fire emergency and nuclear applications.

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APPENDIX A

DEVELOPMENT OF A NICKEL CLAD, BARRIER COATED COPPER CONDUCTOR

A-1 INTRODUCTION

During the conductor development section of Phase I of this program, various other approaches were considered which might allow the use of less expensive conductors such as copper. One such approach, the development of a protective and stable high temperature coating for copper, was very briefly examined by members of the Faculty of Engineering Science of the University of Western Ontario.

It was proposed that an adherent, thermally formed oxide film be developed on the surface of copper conductor to act as a diffusion barrier between the wire and the cladding or any other form of corrosion resistant coating, thus effecting a significant cost saving by allowing the replacement of the present silver core.

Many ceramics, especially oxides, are known to be thermodynamically more stable than metals and consequently less susceptible to interfacial reactions with metal substrates. However, under appropriate conditions, thermodynamically stable oxides will dissolve in metals. If one considers the displacement reaction between Cu and Ni and their lowest oxides, Cu_2O and NiO,

$$Ni + Cu_2O \rightarrow NiO + 2Cu$$

then examining the Gibbs free energy for this reaction suggests that spontaneous interaction should result from the contact of a Ni/Cu_2O couple, if the temperature is high enough ($\sim$1800°F). However, at the service temperature of 1250°F, such an interaction is highly unlikely as the free energy of reaction is extremely low. This results from the fact that the cation diffusion in NiO is reaction rate controlling rather than the oxygen transport in copper.

The approach taken was to form a continuous adherent Cu_2O film on a Cu wire substrate, reduce the surface of the oxide to Cu to regain conductivity, electrodeposit a layer of Ni and then heat treat to reoxidize the Cu interlayer to Cu_2O and form an NiO layer between the Ni and Cu_2O. Very preliminary experiments suggested that the barrier system appeared feasible, but other aspects of performance such as mechanical strength, thermal shock resistance and resistance to thermal oxidation were not evaluated.

A contract was awarded to the University of Sherbrooke, Sherbrooke, Quebec, to apply this innovative technology to the development of potential alternative high temperature resistant conductors for the program.

A-2 PROCEDURE

A-2.1 Preliminary Experiments

Test samples of copper wire (0.040" dia) were polished with 600 SiC grit paper and cleaned in alcohol. The samples were heated in vacuo at 800°C for 2 hours and then oxidized in the same apparatus to produce a 5-15 μm thick oxide film by introducing O_2 through a leak valve at a pressure of 1-2 torr.

In the first qualitative series of experiments the samples were rapidly cooled to room temperature to retain the Cu_2O type oxide formed at 800°C. However, attempts to Ni-coat the preoxidized samples using an electroless process failed due to rapid dissolution of the oxide film by all of the electroless solutions utilized. To overcome this problem subsequent samples were surface reduced immediately after oxidation, at the temperature of oxidation, in a mixture of 10:1 Ar/H_2 gas for 5-10 seconds. This step produced a 1-2 μm thick layer of free copper over the oxide film which made the surface conductive. These samples were then coated with a Ni layer of about 1-5 μm thick using a standard electrodeposition procedure. It was found that the oxide film retained its adherence and integrity while the Ni layer had excellent adhesion to the thin surface layer of copper.

A-2.2 Diffusion of the Cu_2O-Ni Couple

The Ni-plated, (pre-oxidized), samples were heat-treated in vacuo at temperatures of 880-930°C for periods of 3 to 8 hours to produce the series arrangement theoretically predicted.

Metallographic examination clearly revealed a NiO layer between the outer Ni layer and the Cu layer adjacent to the Cu_2O film surface. X-ray diffraction analysis performed on this sample confirmed the presence of the couple reactions observed metallographically.

A further series of experiments with copper coupons consisting of a short oxidation period in a mixture of argon and oxygen followed by a short reduction period in argon and hydrogen were carried out at 740, 800 and 840°C respectively. Subsequent metallographic study on the sectioned coupons enabled correlation of the oxide film thickness formed on the copper core and that of the free copper overcoat with the experimental conditions, viz., temperature, duration of oxi-

dation and reduction, and partial pressures of oxygen and hydrogen respectively. The pre-oxidized coupons were then plated with nickel and annealed in an inert atmosphere at 900°C until the diffusion barrier completely formed. The variables (oxide film thickness, plated nickel film thickness, annealing time) were studied to optimize the conditions of formation of the diffusion barrier.

A-2.3 Dynamic Studies

Concurrently an apparatus was designed for the continuous formation of the coating on copper wire. A schematic representation of the apparatus is shown in Figure A-1. It is comprised of three zones, the central zone serving to isolate the oxidation from the reduction zone. Constrictions in the inner glass tube supporting the wire serve to separate the three units of the system and as a positive argon gas pressure is maintained in the central zone, the gas mixture of either of the two end zones is prevented from entering the neutral zone. A gas train was specially designed to control the pressure, the flow rate and the gas mixture of both the oxidizing and reducing atmospheres. Figures A-2 to A-4 show the actual apparatus. The temperature of each zone is controlled by a separate furnace and its associated temperature controller. The temperatures at four critical points along the reaction chamber are monitored with chromel-alumel thermocouples. The system connects to a Pyrex-glass electroplating tank through a teflon seal. A variable speed motor provides the drive necessary on a 12-inch diameter spool to pull the wire through the system and to coil it.

The data obtained from the preliminary static experiments were found to have limited use in the dynamic system and the experimental conditions had to be revamped on the basis of several trials with the dynamic system. The following conditions (Table A-1) proved to be well within the range to produce the desired duplex coating on the copper wire.

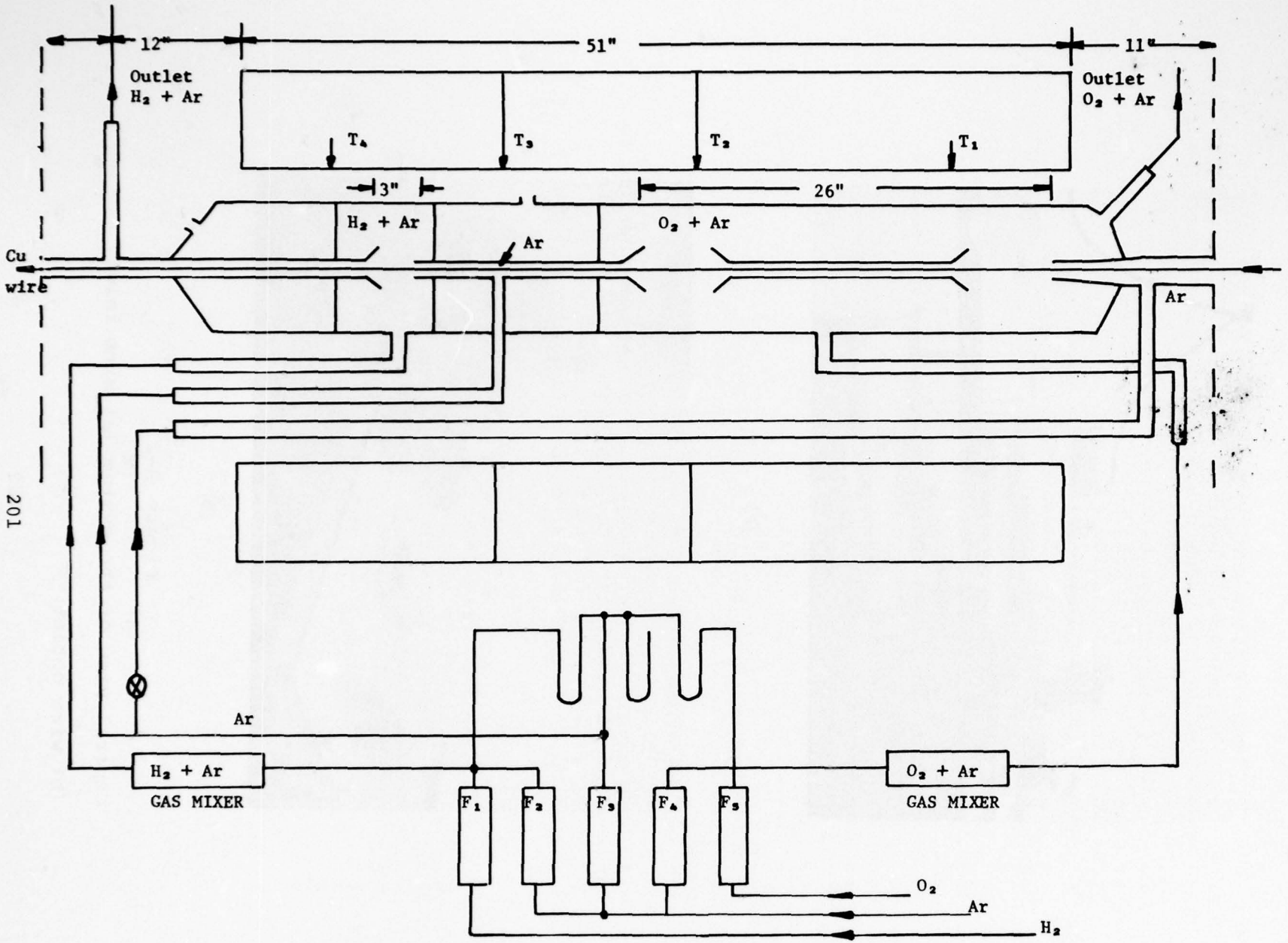
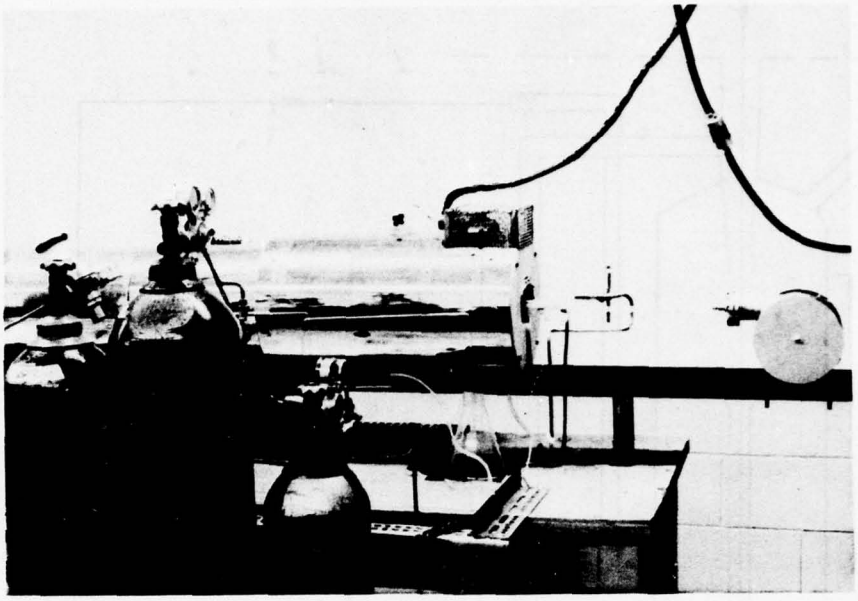
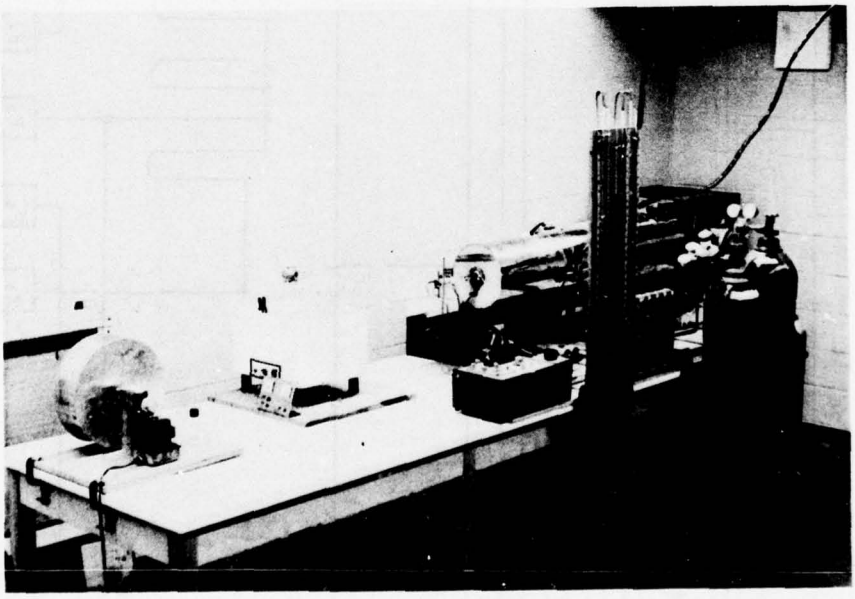


Figure A-1 Schematic representation of the system to accomplish the first step process.



(a)

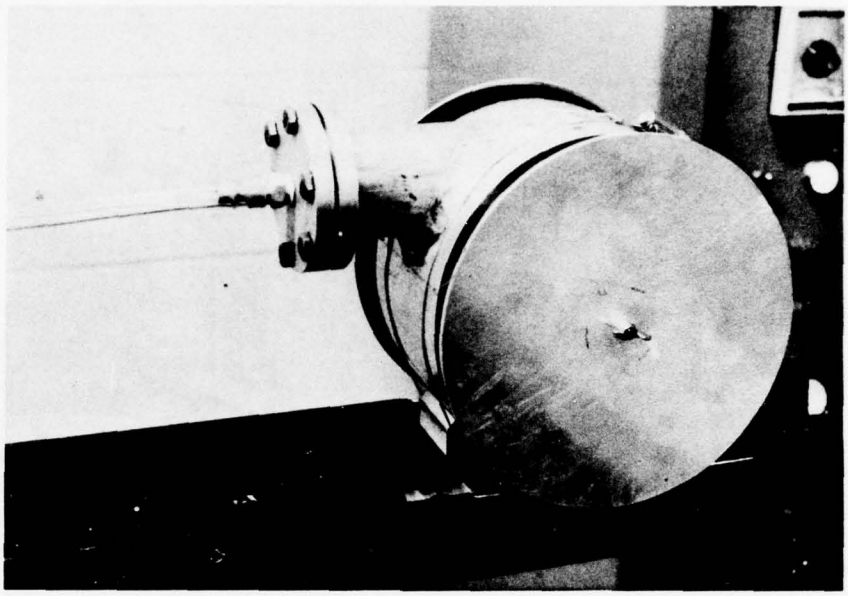


(b)

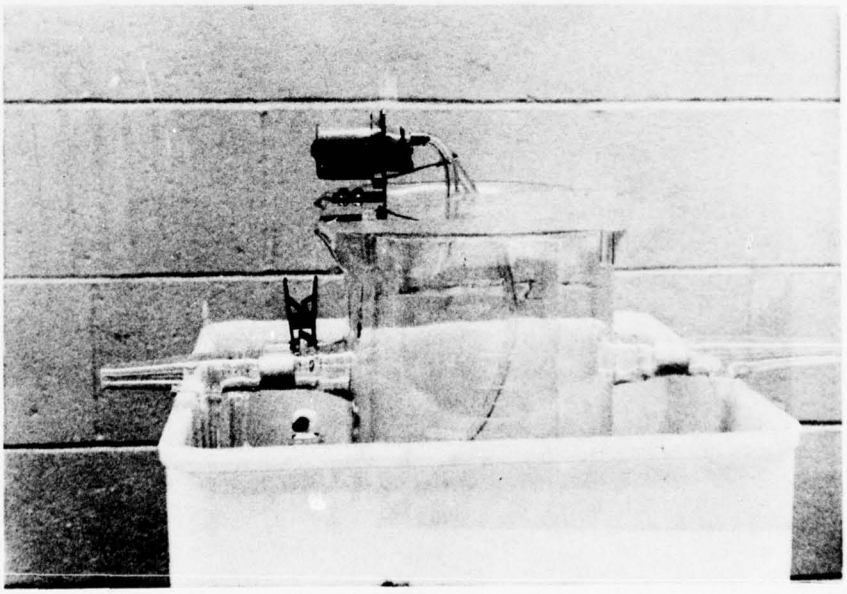
Figure A-2

General view of the system (a) wire inlet

(b) wire outlet.



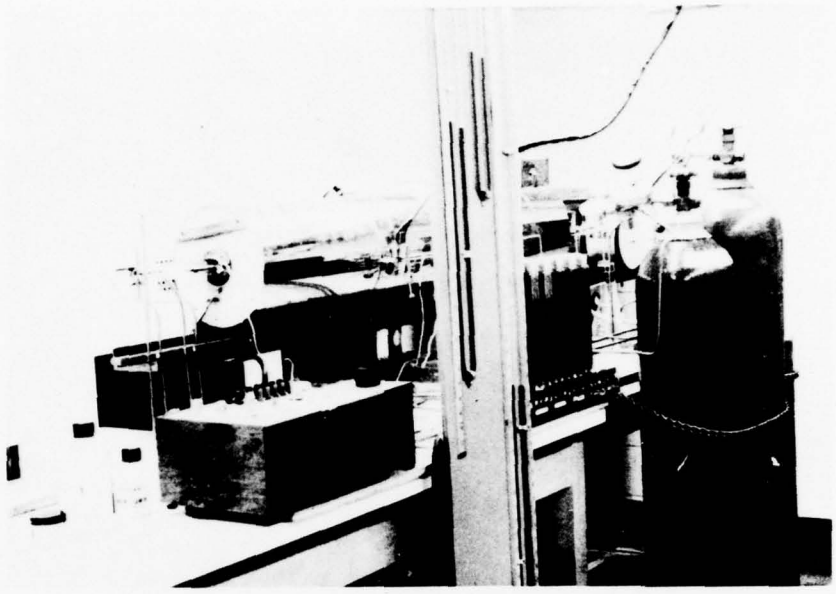
(a)



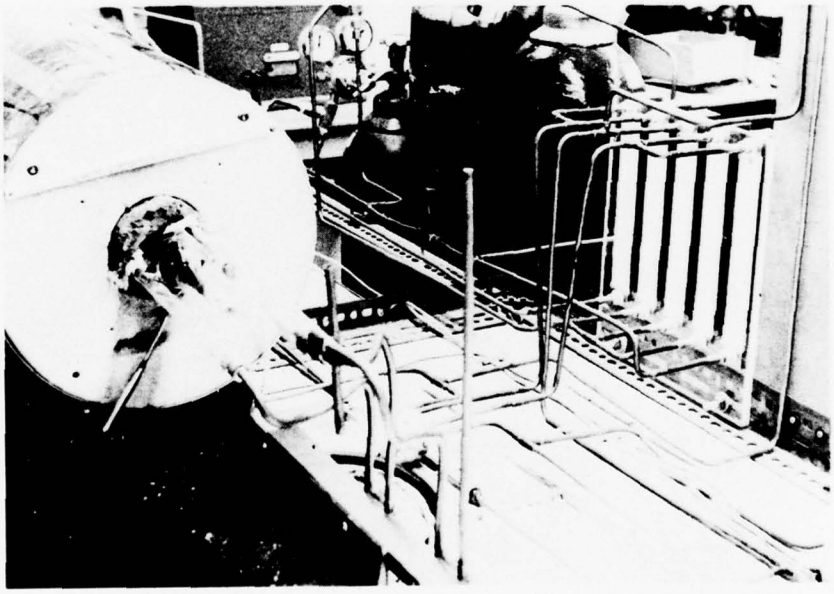
(b)

Figure A-3

(a) Protective casing for the coil of wire (inlet).
(b) Electroplating tank.



(a)



(b)

Figure A-4

Gas train (a) general view of manometers and gas flow meters.
(b) system gas feed lines.

TABLE A-1

EXPERIMENTAL CONDITIONS FOR PRODUCTION OF DUPLEX COATINGS ON COPPER CONDUCTOR

Operating Zone	Zone Temperature °C	Gas Mixture and Flow Rate cc/minute	
Oxidation	750	Argon Oxygen	45 6
Neutral	730	Argon	500
Reduction	750	Argon Hydrogen	45 3

A metallographically polished section of the copper wire that was processed through the first step is shown in Figure A-5. The cupric oxide layer sandwiched in between the outer layer (nickel) and the substrate, is about 5 μm thick and uniform. The nickel overcoat is about 10 μm thick and generally uniform. Short lengths of the coated wire were given a 6-hour diffusion anneal in vacuo at 900°C to produce the NiO diffusion barrier. Metallographic observations of the sectioned heat-treated wires suggested that the Ni-Cu_2O couple was completely reacted. The diffusion layer (NiO) was about 1 μm thick and difficult to resolve on the optical microscope (Figure A-6).

Following the success of these preliminary dynamic experiments, work continued to optimize the oxidation/reduction step by varying the oxygen and/or hydrogen partial pressures in the system. This caused alterations in the relative thicknesses of the copper oxide and reduced copper oxide layers, which in turn affected the adhesion of the subsequently applied Ni layer as well as the integrity of the final vacuum annealed wire.

Table A-2 summarizes the results of these experiments. The conditions listed for Runs #10 and 17 have been adopted permanently, as they give a sound, adherent coating. A wire feed of 5 cm/minute through the system was retained as in previous tests, as it allows the optimization of the

TABLE A-2

SUMMARY OF EXPERIMENTAL CONDITIONS AND RESULTS OF SERIES OF TESTS CONDUCTED WITH THE DYNAMIC SYSTEM

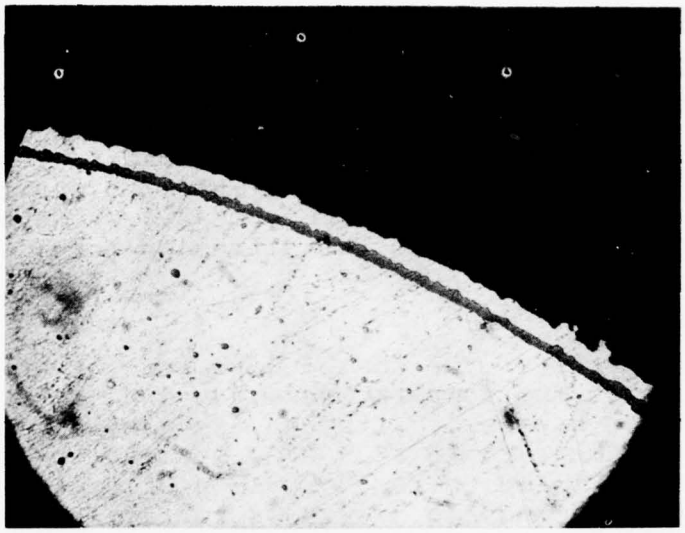
Run No.	Temperature of zones (°C) Oxid	Neut.	Red	Gas flow (CC/min) O_2*	Ar	H_2*	Thickness of layers (μm) CuO+ Cu	Cu	Ni-overcoat	Comments
10	750	750	750	6.0	500	1.5	7	3	8	Good adherence and sound coating
11	750	750	750	2.5	500	0.5	-	-	-	Cu layer too thin for Ni-plating
12	750	750	750	2.5	500	1.0	2.5	2	8	CuO layer too thin
13	760	755	770	4.0	500	1.0	4	3	8	CuO layer too thin
14	770	755	770	6.0	500	1.0	5	4.5	8	Poor adherence of Ni-plated onto CuO
15	770	770	775	10	500	1.0	8	7	8	Reduced CuO layer too thick
16	755	770	775	10	500	1.0	8	7	16	Reduced CuO layer too thick
17	750	750	750	6	500	1.5	7	2	8	Good adherence and sound coating.

* Flow of gaz mixed to argon flow of 45cc/min.

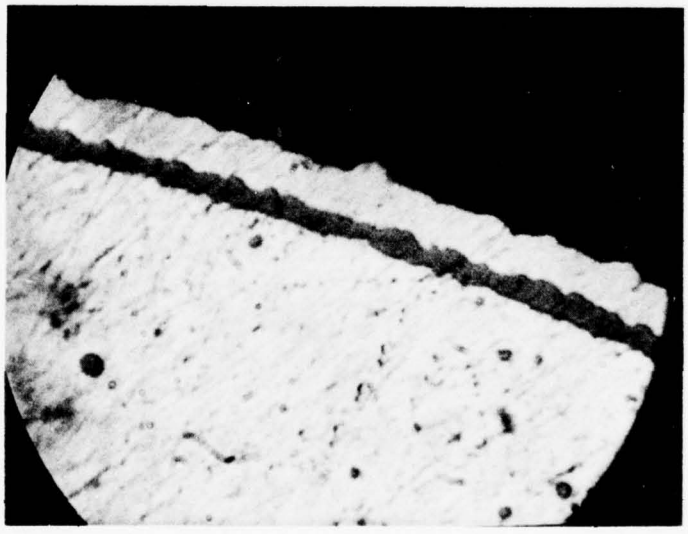
Ni-plating conditions. Some minor problems such as inhomogeneous nucleation and a preferential penetration of free copper into the CuO layer were not resolved. These did not affect the soundness of the final product and could be rectified only by major alteration of the gas entry into the quartz reaction chamber. The photomicrograph in Figure A-7 shows a cross-section of the coating before the diffusion anneal.

A-2.4 Diffusion Anneal

Short lengths of the coated wire were given a diffusion anneal in vacuo at two temperatures, 800 and 900°C, to produce the NiO diffusion barrier. Although the development of the barrier was much more rapid at 900°C, heating the coated wire to this temperature led to the formation of cracks at the CuO layer interface and the growth of surface pustules. The 800°C anneal did initiate the nucleation of the NiO barrier but the rate of growth was impractically slow. Figure A-8 shows a photomicrograph of the coated wire following a 20-hour vacuum anneal at 810°C; a thin NiO layer adjacent to the Ni overcoat can be seen. Further annealing of these samples at 900°C for one hour did not, however, affect the soundness of the coating. It would thus appear that an initial heating at 800°C followed by more rapid diffusion anneal at 900°C could be a practical solution.



(a) X 400



(b) X 1000

Figure A-5

Metallographic section of the wire following the first step process.

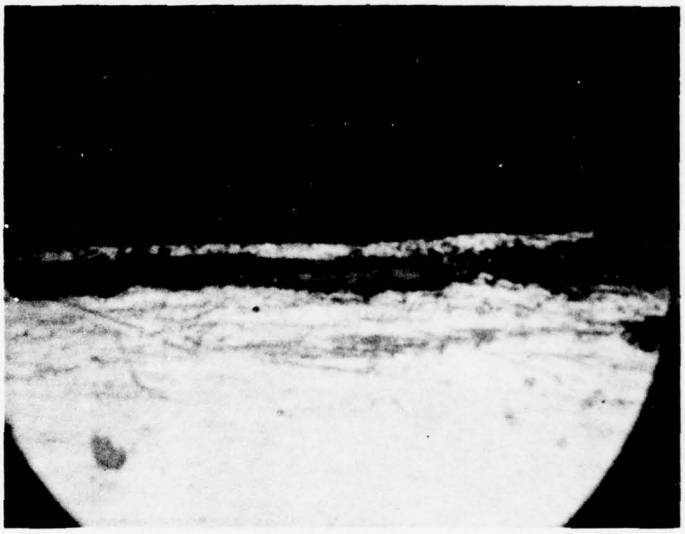


Figure A-6

Metallographic section of the coated wire following a vacuum anneal at 900°C for 6 hours.

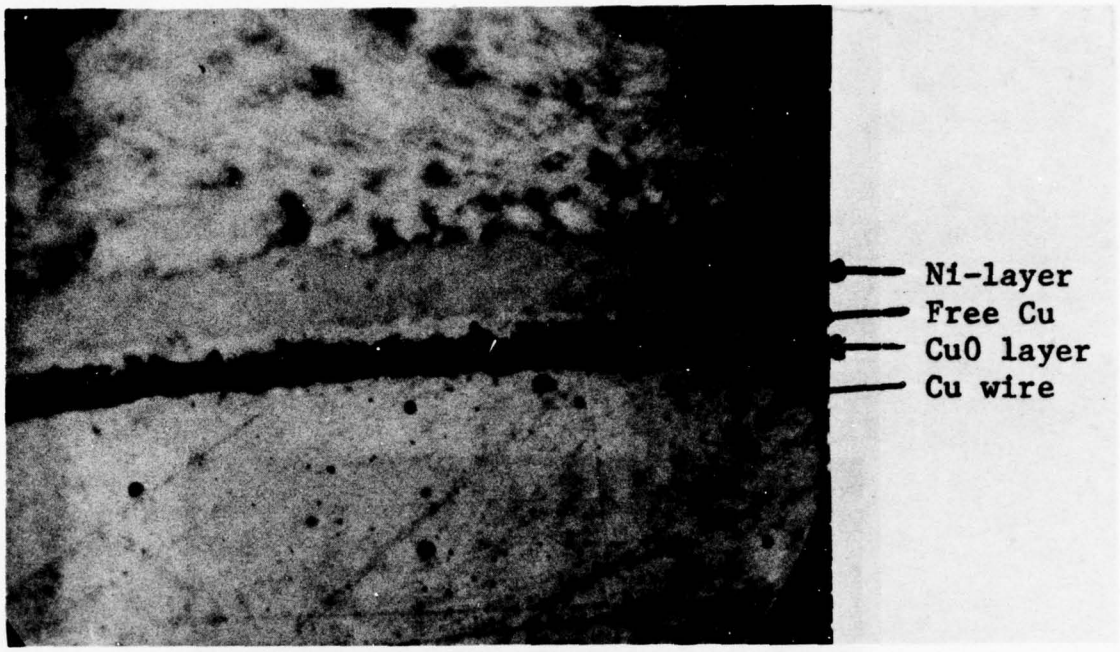


Figure A-7

Polished section of the coated wire - First step process. X1100

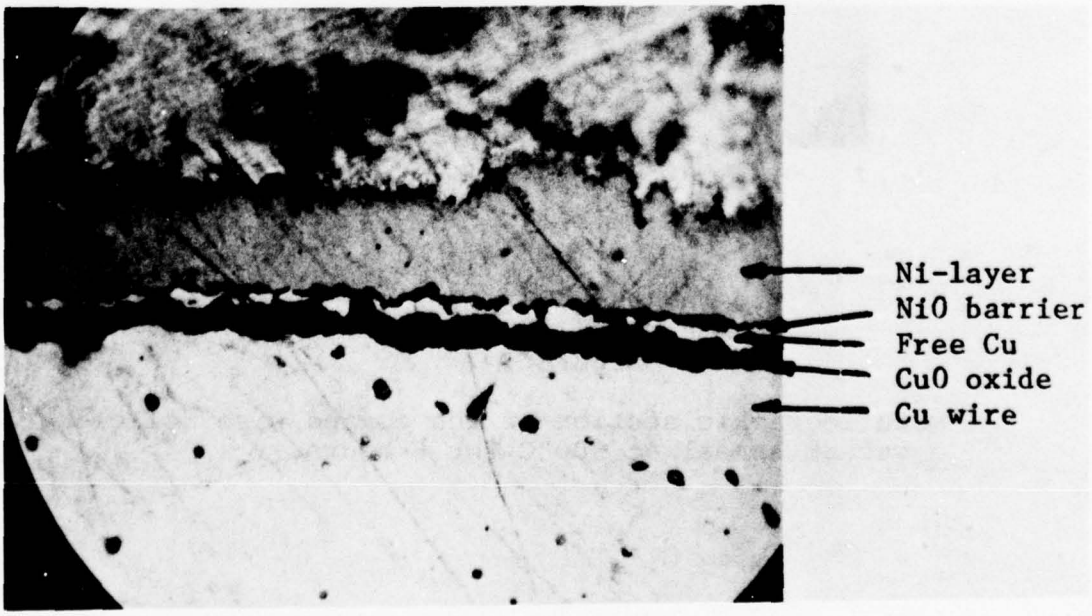


Figure A-8

Polished section of the coated wire following a vacuum anneal. (Nucleation of the NiO barrier can be seen as a dark layer adjacent to the Ni-plated layer. X1100)

APPENDIX B

CONTACT RESISTANCE AND CONTACT SPRING RELAXATION INVESTIGATION (PART A)

B-1 OBJECT

To investigate the effect on the millivolt drop across mated pairs of unplated stainless steel type 308L contacts due to relaxation in contact retention spring at room ambient temperature.

B-2 SUMMARY

As Inconel X750 material was not available, and in order to expedite the test program, Inconel X was chosen because it has similar creep properties to the former up to temperatures of +600°F.

The Inconel X springs were mounted on the unplated stainless steel socket contacts, (see Fig. B-1), so as to exert a retention force (Appendix D) on the pin contacts.

The millivolt drop across the mated pair of No. 16 contacts was measured with the help of a Contact Resistance Tester at various contact retention forces.

This procedure was carried out at ambient temperature (78°F) for three samples of mated contacts.

Of prime importance was the determination of the nature and degree of change due to spring relaxation rather than the magnitude of the measured results.

B-3 INTRODUCTION

B-3.1 Contact Resistance

Contact between two solid conductors is established when the highest of the minute asperities on each surface meet. As the force pressing the surfaces together is increased, the asperities are deformed to increase their contact areas. Consequently, the resistance to the flow of current across the junction is called the constriction resistance([22]). The contact resistance therefore includes the bulk and the constriction resistances. The contact resistance is determined by measuring the millivolt drop from tail to tail for the mated set of contacts.

The degree of electrical resistance or, conversely, conduction between two surfaces designed to make or break an electric circuit is limited by the mechanical qualities and resistivities of the materials. Basically, in a contact

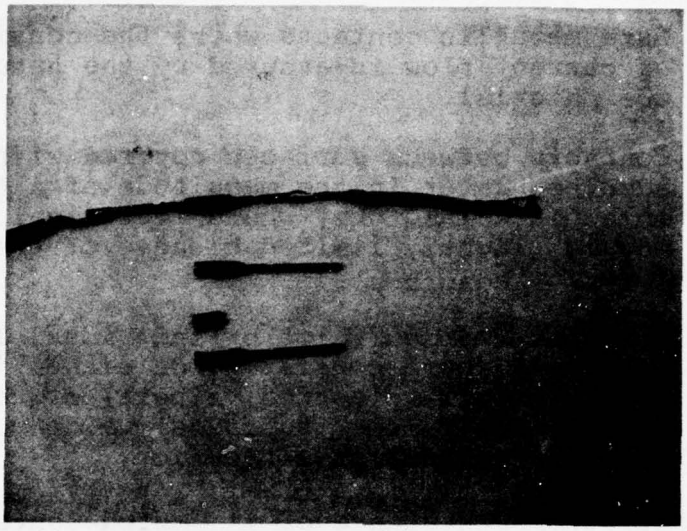


Figure B-1

Assembly of stainless steel type 308L contacts with the Inconel X retention spring.

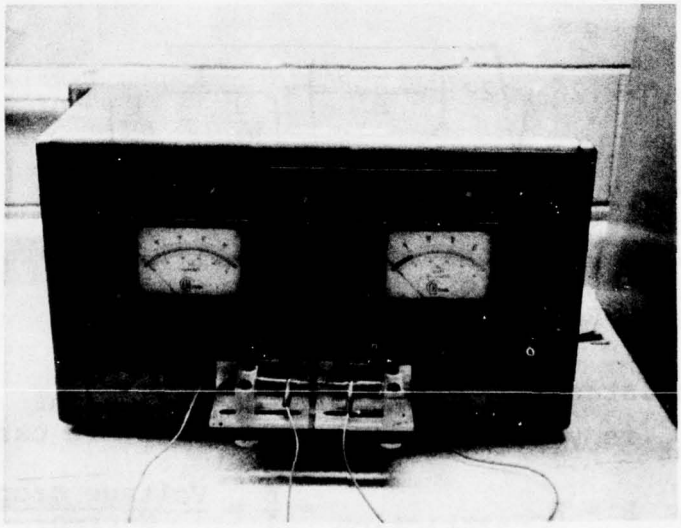


Figure B-2

Contact Resistance Test Apparatus.

system there are three controlling factors or conditions:

(i) Bare metallic contacts where the continuity of current flow is assured by the nature of the material.

(ii) Contacts between surfaces covered with an adsorbed layer in the magnitude of a few molecular diameters, where electrical flow may be slightly limited because of the impurity adsorbed.

(iii) Coherent foreign films or individual large particles, both consisting of either insulators (oil, plastics, fibers) or semiconductors (oxides, sulfides) which act as limiting factors in conduction.

B-3.2 Constriction Resistance of #16 Mated Contacts

The resistance of a pair of clean contacts depends on the bulk resistance of the material (which is so small that it can be neglected) and the resistance to the flow of current at the control junction. A cross-sectional view through the pair of mated contacts is shown in Figure B-3.

For elastic deformation the area of contact per unit length due to force F is derived as

$$A = 2.26 \sqrt{2F \left(\frac{1 - \mu^2}{E} \right) x \frac{1}{\left(\frac{1}{R_1} - \frac{1}{R_2} \right)}} \text{ -------} \quad (23,24)\ (i)$$

where, F = Load (lbs-force)
E = Modulus of Elasticity (lbs/in^2)
μ = Poisson's ratio ($\simeq .3$)
R_1 = Radius of Pin Contact (in)
R_2 = Radius of Socket Contact (in)

Contact Resistance $R = R_{Bulk} + R_{Constriction}$.
Since R_{Bulk} is very small (.001Ω or less) it can be neglected.

Therefore, $R \simeq R_{Constriction} = \frac{E}{I} = \frac{\text{Voltage drop}}{\text{Current}}$ (vii)

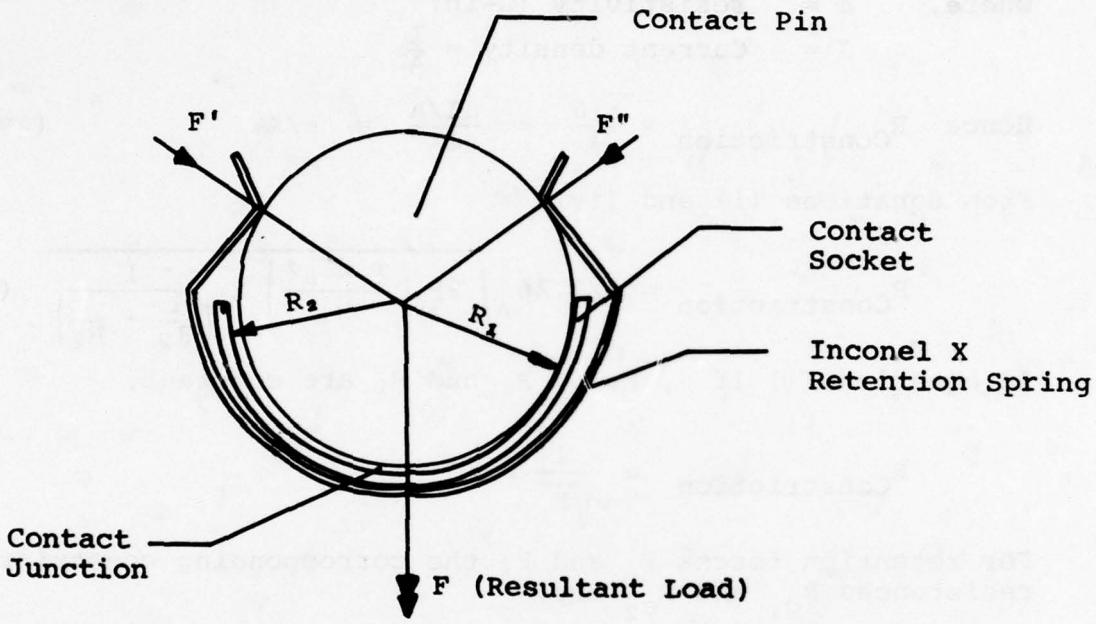


Figure B-3

Cross-section of a pair of mated contacts.

Also, $E = \rho . J$ (iii)

where, ρ = resistivity (Ω-in)

J = Current density = $\frac{I}{A}$

Hence $R_{Constriction} = \frac{\rho . J}{I} = \frac{\rho I/A}{I} = \rho/A$ (iv)

From equations (i) and (iv)

$$R_{Constriction} = \rho/2.26\sqrt{2F\left(\frac{1-\mu^2}{E}\right) \times \frac{1}{\left(\frac{1}{R_1}-\frac{1}{R_2}\right)}} \quad (v)$$

In equation (v) if ρ, E, μ, R_1 and R_2 are constant,

$$R_{Constriction} \alpha \frac{1}{\sqrt{F}}$$

For retention forces F_1 and F_2 the corresponding constriction resistances R_{c_1} and R_{c_2} are,

$$R_{c_1} \alpha \frac{1}{\sqrt{F_1}} \quad (vi)$$

$$R_{c_2} \alpha \frac{1}{\sqrt{F_2}} \quad (vii)$$

Dividing (vi) by (vii)

$$\frac{R_{c_1}}{R_{c_2}} = \sqrt{\frac{F_2}{F_1}} \quad (vii)$$

Equations (v) and (viii) have been derived using some simplifying assumptions, but they are accurate enough for practical purposes and show which material parameters influence the constriction resistance.

B-4 TEST PROCEDURE

The Inconel X retention spring mounted on a sample of the contact socket was deflected such that it exerted a maximum pressure on the contact pins. This retention force was measured with the help of a standard No. 16 pin and standard weights.

The mated pair of contacts was connected to the Contact Resistance Tester, (Figure B-4) and the millivolt drop was measured.

The retention spring was relaxed and the new retention force noted. A new reading for the millivolt drop was thus obtained. A third reading was then measured by removing the spring altogether.

Using the same contact pin, the above procedure was adopted for all three samples of contact sockets complete with retention springs.

B-5 TEST RESULTS

SAMPLE 1

Current (Amps)	Millivolt Drops Across Mated Contacts (F ≃ 12 oz)	(F ≃ 5 oz)	(Without Spring)
5	212	262	328
10	315	378	432
15	480	489	480

SAMPLE 2

Current (Amps)	Millivolt Drops Across Mated Contacts (F ≃ 12 oz)	(F ≃ 4 oz)	(Without Spring)
5	178	218	320
10	317	350	400
15	420	420	430

SAMPLE 3

Current (Amps)	Millivolt Drops Across Mated Contacts (F ≃ 12 oz)	(F ≃ 5 oz)
5	115	122
10	212	238

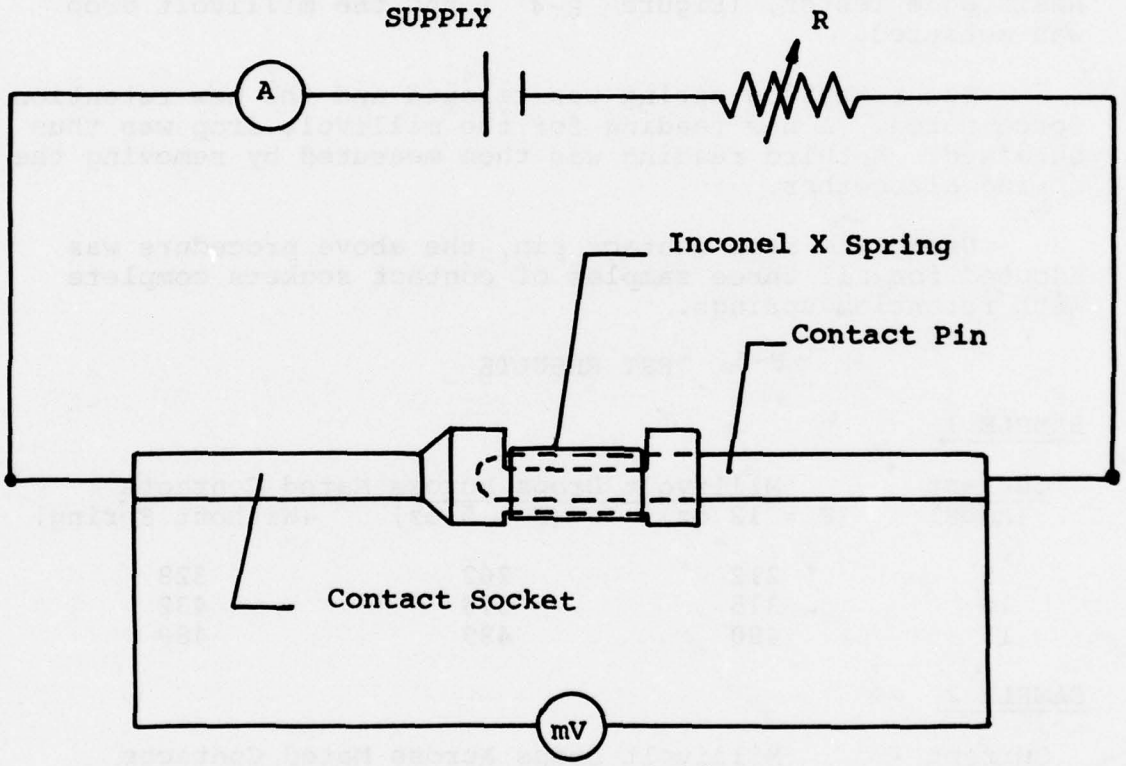


Figure B-4

Test circuit for determination of contact resistance.

B-6 CALCULATIONS

TABLE B-1

VERIFICATION OF EQUATION (viii) USING RESULTS FROM SECTION B-5

Current (A)	F_1 (oz)	F_2 (oz)	R_{c_1} (Ω)	R_{c_2} (Ω)	$\sqrt{\frac{F_2}{F_1}}$	$\frac{R_{c_1}}{R_{c_2}}$
SAMPLE #1						
5	12	5	.042	.052	.64	.80
10	12	5	.032	.038	.64	.84
SAMPLE #2						
5	12	4	.036	.044	.58	.81
10	12	4	.032	.035	.58	.91
SAMPLE #3						
5	12	5	.023	.025	.64	.92
10	12	5	.021	.024	.64	.88

B-7 DISCUSSION

During the test it was noticed that as the current was increased, the millivolt drop rose rapidly to a peak and then gradually dropped to a lower saturation value. This observation results in the fact that the contact resistance is high at low voltages and decreases to a constant value as the voltage is increased.

The results (Section B-5) indicate that at high currents, ($\geq$15A), the millivolt drop across the contacts is independent of the spring retention force and this is explained as follows:

(a) The high current heats up the contacts to a temperature greater than 450°F thereby facilitating the tunneling of electrons across the contact surfaces to a greater degree as the work function for the contact material is lowered.

(b) The heat produced causes an expansion in the contact material resulting in an increased mutual pressure

between the contact pin and the contact socket thereby reducing the constriction resistance. This, combined with (a), nullifies the effect of the retention spring.

The results also show that without the retention spring the contact resistance at low voltages is reasonably high and therefore the millivolt drop and the degree and rate of temperature rise is large even at low current values.

The differences in the calculated results in Table B-1 may be explained by

(i) Surface contamination on contacts because of failure to properly degrease them before the tests - thereby resulting in unreal magnitudes of results in Section B-5.

(ii) Human accuracy errors.

B-8 FURTHER INVESTIGATION

Tests are being conducted on the millivolt drop across mated pairs of plated and unplated stainless steel type 308L and Inconel 601 contacts using Inconel X retention springs at +600°F. Inconel X750 will eventually be used for testing at +1250°F on final prototypes.

The purpose of these tests is to determine if

(a) there is relaxation in the retention springs at the chosen temperature ranges and if,

(b) the change in the millivolt drop across the mated contacts is in close agreement to the analysis of this report.

B-9 CONCLUSIONS

From the test results the following conclusions can be drawn:

(a) At low currents, as the Inconel X retention spring relaxes, there is an increase in millivolt drop across the mated pair of contacts.

(b) At high currents ($\geq$15A) the millivolt drop appears to be independent of the spring force.

APPENDIX C

CONTACT RESISTANCE AND CONTACT SPRING RELAXATION INVESTIGATION

PART B

C-1 SUMMARY

This report is a continuation of the Contact Resistance/Contact Spring Relaxation Investigation described in Appendix B, and describes the determination of the change in contact resistance across mated pairs of #15 gold plated stainless steel type 308L contacts with Inconel X retention springs at 600°F ± 10°F for a period of 680 hours.

The final prototype contacts for use at 1250°F are intended to be #16 nickel plated stainless steel type 308L contacts with Inconel X-750 retention springs. However, at the time that these studies were made, neither the Inconel X-750 was available nor the nickel plated stainless steel contacts, thus, Inconel X springs, which have similar high temperature performance properties were employed, along with readily available gold plated contacts.

C-2 INTRODUCTION

C-2.1 Derivation of Contact Resistance

a) Constriction Resistance

It was previously shown[25] that the constriction resistance per unit length of a pair of clean contacts is:

$$R_{Constriction} = \rho/2.26\sqrt{\frac{2F(1-\mu^2)}{E} \times \frac{r_1 r_2}{r_2 - r_1}}$$

Hence, for a length ℓ,

$$R_{\ell\,(Constriction)} = \rho/\ell \times 2.26\sqrt{\frac{2F(1-\mu^2)}{E} \times \frac{r_1 r_2}{r_2 - r_1}} \qquad (i)$$

For the gold plated contact samples,

Length of contact surface	ℓ	=	.342"
Radius of contact pin	r_1	=	.0315"
Radius of contact socket	r_2	=	.035"
Poissons ratio for Gold	μ	=	.6
Average of P max & P min (Appendix D)	F_{AV}	=	.89 lbsf
Resistivity of Gold	ρ	=	1.85×10^{-6} ohms-in
Modulus of elasticity	E	=	11.5×10^{6} psi

Thus, substituting in equation (i)

$R_{\ell\,(\text{Constriction})} = .0135$ ohms

Considering the dimensioned design cross-section of a pair of #16 contacts in stainless steel 308L, (Figure C-1), it is possible to derive a bulk resistance value assuming that the contacts have a resistance pattern shown in Figure C-2.

b) Bulk Resistance

Volume Resistivity $\rho = 28.347 \times 10^{-6}$ -in

$$R = \rho \frac{L}{A}$$

For the various sections of Figure C-1,

SECTION 1

Area = .0030679 in^2

L = .598 in

$R_1 = .00552$ ohms

SECTION 2

Area = .001735 in^2

L = .062 in

$R_2 = .00101$ ohms

SECTION 3

Area = .0030679 in^2

L = .150 in

$R_3 = .001386$ ohms

SECTION 4

Area = .001735 in^2

L = .054 in

R_4 = .000882 ohms

SECTION 5

Area = .011309 in^2

L = .091 in

R_5 = .000228 ohms

SECTION 6

Area = .00746 in^2

L = .0935 in

R_6 = .002813 ohms

SECTION 7

Area = .0030679 in^2

L = .350 in

R_7 = .003234 ohms

$$R_{6,7} = \frac{R_6 \times R_7}{R_6 + R_7} =$$

$$\frac{.000009}{.006047} = .001488 \text{ ohms}$$

$R_{6,7}$ = .001488 ohms

SECTION 8

Area = .0030679 in^2

L = .060 in

R_8 = .000554 ohms

SECTION 9

Area = .0030679 in^2

L = .150 in

R_9 = .001386 ohms

SECTION 10

Area = .0030679 in^2

L = .4255 in

R_{10} = .003931 ohms

hence, R_{Total} = .0184 ohms

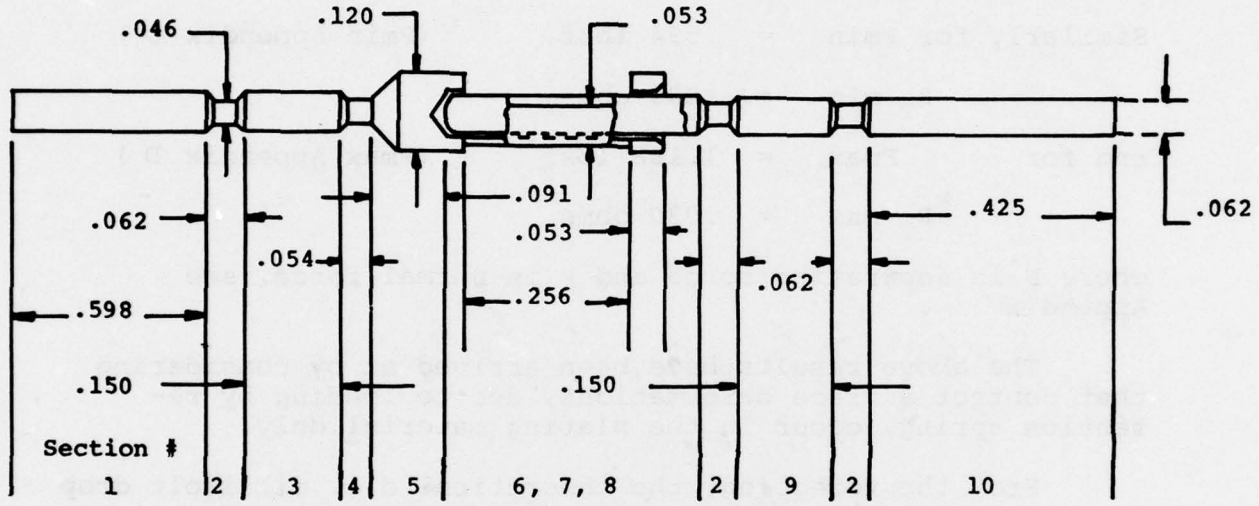


Figure C-1

Dimensioned mated pair of #16 contacts in Stainless Steel 308L (Dimensions in inches).

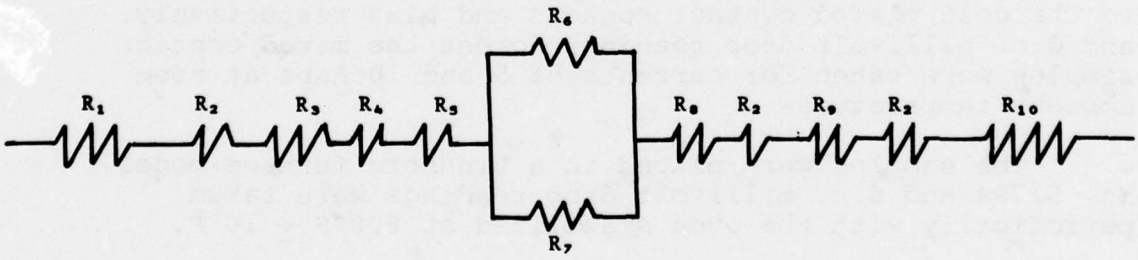


Figure C-2

Resultant resistance pattern corresponding to figure C-1.

Therefore, total contact resistance R_{TAV} equals

$$R_{TAV} = R_{\ell\,(Constriction)} + R_{Bulk} \qquad \text{(ii)}$$

$$= .0135 + .0184$$

$$= .032 \text{ ohms}$$

Similarly for Fmin = .594 lbsf (Pmin Appendix D)

R_T min = .035 ohms

and for Fmax = 1.188 lbsf (Pmax Appendix D)

R_T max = .030 ohms

where F is separating force and P is normal force, see Appendix .

The above results have been arrived at by considering that contact surface deformations, due to loading by retention spring, occur in the plating material only.

From the foregoing, the theoretical d.c. millivolt drop for a current of 5A corresponding to the minimum and maximum contact resistance values has been computed to range between 150 and 175 mV.

C-3 TEST PROCEDURE

Three foot lengths of 16 AWG copper wire were brazed to the gold plated contact sockets and pins respectively, and d.c. millivolt drop readings across the mated contact samples were taken for currents of 5 and 10 Amps at room ambient temperatures.

The samples were placed in a Lindberg furnace model No. 51794 and d.c. millivolt drop readings were taken periodically with the oven stabilized at 600°F $\pm$ 10°F.

C-4 TEST RESULTS

TABLE C-1

Millivolt Drop Across #16 Gold Plated
Stainless Steel Type 308L Contacts
with Inconel X Retention Springs

TIME IN OVEN (HRS)	TEMP (°F)	MILLIVOLT DROP ACROSS #16 GOLD PLATED CONTACTS SAMPLE NO. 1		SAMPLE NO. 2		SAMPLE NO. 3	
		5A	10A	5A	10A	5A	10A
0	76	210	420	172	350	172	350
4	596	237	500	210	430	200	410
46	598	235	488	208	420	197	402
95	603	232	467	208	420	201	408
167	600	238	480	207	418	196	400
191	598	240	473	206	416	198	400
335	600	247	480	203	414	196	402
679	592	258	500	207	418	200	408
AFTER COOLING	76	235	458	177	361	175	360

C-5 DISCUSSION

Test results of the voltage drop at room temperature for samples 2 and 3, Table C-1, lie within the theoretically estimated minimum and maximum millivolt drop range. However, the millivolt drop at 76°F for sample 1 is considerably higher when compared to the theoretical value. This large difference could be accounted for by the following.

(a) Excessive deposit of insulating contaminant on contacts because of failure to degrease samples before testing.

(b) The load due to the retention spring and consequently the pressure between mating surfaces is not of sufficient magnitude to wipe away any insulating contaminant and thus maintain good contact.

(c) Extent of the porosity of gold plating.

(d) Errors in the control and measurement of current and temperature. Inspection of the contact samples after the heat cycle showed that an oxidized film (possibly partly due to impurities in gold and base material) had deposited on the contact surfaces. Deposits of charred insulating contaminants, (i.e. grease and dust particles), were also noticed. This clearly accounts for the fact that the final millivolt drop results at 76°F are higher than the initial readings.

The variation in the millivolt drop at 600°F ± 10°F with time is negligible for samples 2 and 3, but quite prominent for sample 1. This large variation for sample 1 can possibly be accounted for by reasons (a) and (b).

C-6 FURTHER INVESTIGATION

Tests will be conducted on the millivolt drop/contact resistance of nickel plated stainless steel type 308L contacts using Inconel X-750 retention springs at a temperature of +1250°F. The purpose of these tests will be to,

(a) Investigate the change in contact resistance at 1250°F for a prolonged period in time,

(b) Obtain the exact value of contact resistance for the final prototype samples.

C-7 CONCLUSIONS

From an analysis of the test results it can be concluded that there is no appreciable change in the contact resistance across a mated pair of #16 gold plated stainless steel type 308L contacts with Inconel X retention springs at 600°F ± 10°F for a period of 680 hours. However, final contact resistance values can only be established upon completion of tests utilizing all materials/finishes identified by the most recent work in the areas of,

(a) sealing
(b) brazing
(c) plating optimization

APPENDIX D

STRESS ANALYSIS OF INCONEL X RETENTION SPRINGS

D-1 OBJECT

To determine the various stresses anticipated on the Inconel X retention spring.

D-2 INTRODUCTION

As shown in Figures B-3 and B-4 , (Appendix B), the preferred pin/contact assembly uses a positive locking retention spring made of Inconel X. Figure D-1 shows such a spring with dimensions.

D-3 CALCULATION OF STRESSES

From Figure D-2 , the maximum yield strength at a 0.2% offset is 82×10^3 psi at 1250°F.

Assuming a safety factor of 1.5 and a modulus of elasticity (E) for Inconel X of 31×10^6 psi, then the permissible working stress at 1250°F $\approx 55 \times 10^3$ psi.

D-3.1 Permissible Deflection (f)

$$f = \frac{\pi D^2 S_s}{4 Eh}$$

$$f = \frac{3.1416 \times .110^2 \times 55 \times 10^3}{4 \times 31 \times 10^6 \times .005}$$

$$f = \frac{2.085}{620}$$

$$f = \underline{.00337} \text{ or } \underline{.004} \text{ max.} \qquad (1)$$

Assuming a total of .002 mfg tolerance affecting f. min. f = .002 inches.

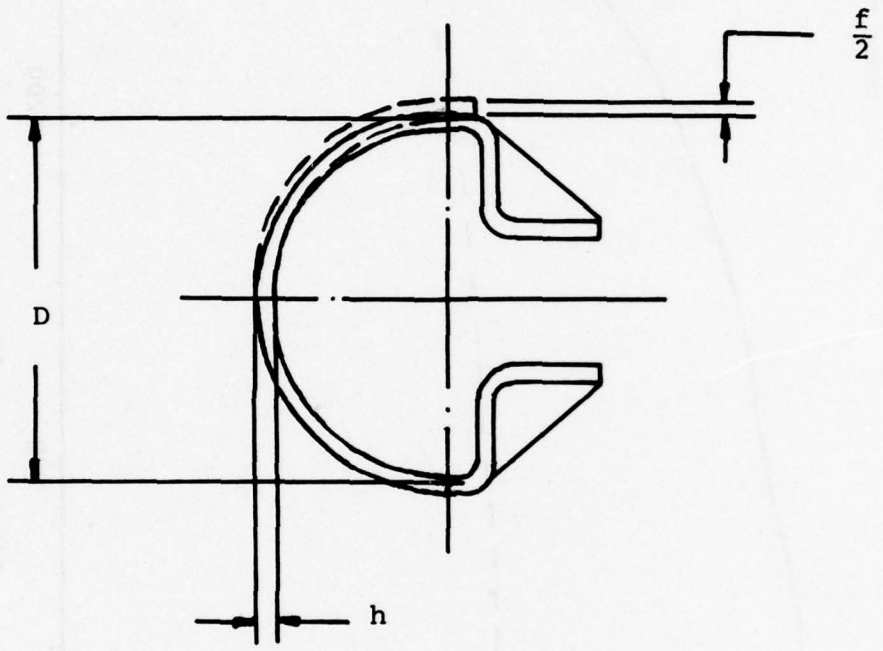


Figure D-1

Inconel Retention Spring

Diameter (D) = .110 inches

Thickness of Material (h) = .005 inches

Length (b) = .240 inches

as f = total deflection

then $\frac{f}{2}$ = single side deflection = 0.2%

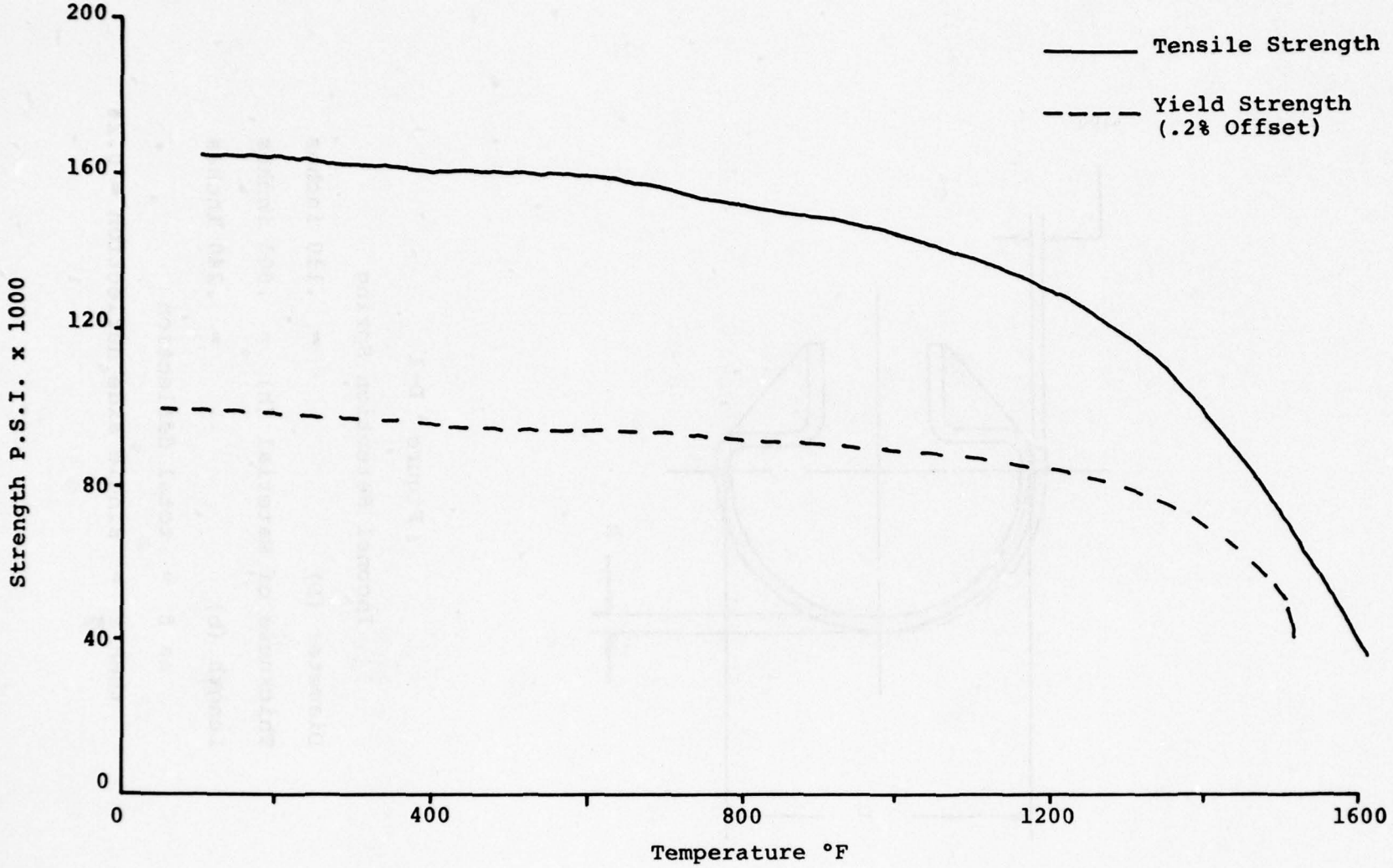


Figure D-2

Elevated Temperature Strength of Inconel X Annealed and Aged.

D-3.2 Force (Normal) P min

$$P_{min} = \frac{Efbh^3}{2.36\ D^3}$$

$$P_{min} = \frac{31 \times 10^6 \times 2 \times 10^{-3} \times 2.4 \times 10^{-1} \times 1.25 \times 10^{-7}}{2.36 \times 1.33 \times 10^{-3}}$$

$$P_{min} = \frac{.00186}{.00313} = .594 \text{ lbs}$$

$$P_{max} = \frac{.004}{.002} \times .594 = \underline{1.188} \text{ lbs} \qquad (2)$$

D-3.3 Separating Force (F)

Assuming $\mu = .5$

$F = \mu P$

$\therefore$ F min = .5 x .594 = .2970 lbs = 4.7 ozs (3)

F max = .5 x 1.188 = .5940 lbs = 9.5 ozs (4)

APPENDIX E

CONDUCTOR/CONTACT BRAZING STUDIES

E-1 INTRODUCTION

As previously noted[26], Au-Ni alloys appear to have many of the necessary brazing material properties for use in high temperature connectors. Upon canvassing three brazing alloy manufacturers, however, Johnson, Matthey & Company, Engelhard Industries, and Wall Colmonoy (Canada) Ltd., it was found that these alloys are not stocked in Canada. Thus, to test the suitability of these alloys, small amounts were made from the pure components.

E-2 PREPARATION OF Au BASED BRAZING ALLOYS

Approximately 10 gm of each Au-18 wt % Ni and Au-18 wt % Ni-5 wt % In were melted in high purity Al_2O_3 crucibles under a boric acid flux in a resistance furnace at 1150°C. The indium was added to the second alloy immediately before casting. Both alloys were cast onto graphite blocks. The Au-Ni-In was much more fluid than the Au-Ni alloy at the casting temperature, since indium lowers the freezing temperature.

Attempts to produce foil of the Au-Ni-In alloy were unsuccessful. Hot rolling reduced the thickness approximately 50%, but edge cracking also took place. Hammering while heating with a reducing flame was also inadequate for producing crack free foils. Small pieces for brazing were either broken or filed from the 10 gm alloy sheet.

E-3 NICKEL PLATING OF INCONEL COATED SILVER WIRE

The plating solution used was:

120 gm nickel chloride
63 ml hydrochloric acid
317 ml deionized water

A pure nickel rod was used as the anode.

After cleaning the wire ends in trichlorethylene and rinsing with alcohol, they were plated using currents from 0.08 to 0.01 amperes, with the wire immersed in the plating bath to a depth of approximately 0.375 inches. The most uniform plating resulted from the lowest current. Plating time for this current was approximately 20 minutes.

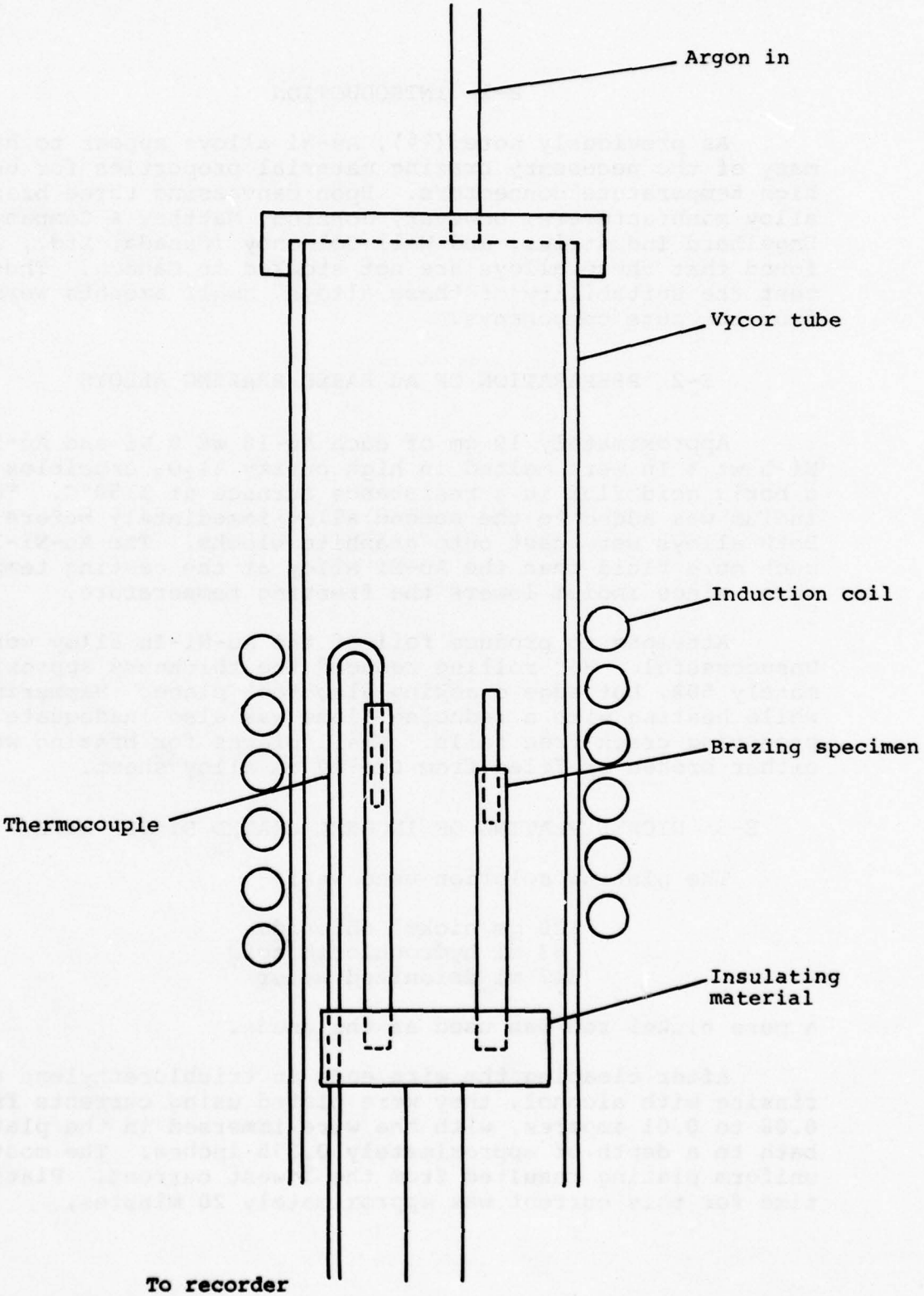


Figure E-1. Schematic diagram of induction brazing apparatus.

E-4 BRAZING EXPERIMENTS

E-4.1 Approaches

(a) Using Fluxes and Oxyacetylene Torches.

Although it was recognized that fluxes may be undesirable if they are difficult to remove([26]), the brazing operation may be simplest if a flux can be employed.

Nickel-plated 308L stainless steel pin contacts, with 0.085" ID holes drilled to a depth of 0.27 in, were brazed to Inconel 600 clad silver wire plated as described in Para.(E3), using the Au-18 wt % Ni-5 wt % In brazing alloy. Brazing was carried out using an oxyacetylene torch with a reducing flame.

Boric acid flux was employed first, using two application methods. Particles of the brazing alloy and some boric acid-water paste were placed inside the contact brazing pot, the plated wire inserted, and more flux paste spread around the flat end surface of the brazing pot (shoulder) touching the wire. Secondly, both the contact and plated wire were dipped into a saturated solution of boric acid, allowed to dry, and then the wire inserted into the contact brazing pot after preplacing particles of brazing alloy in the brazing pot. In all cases the molten brazing alloy did not flow easily into the gap, apparently because this flux has a high surface tension or viscosity which slows the flow of the molten metal.

The use of a commercial flux, Nicrobraz, Wall Colmonoy (Canada) Ltd., normally used for nickel-based brazing materials, did allow the brazing alloy to flow into the gap. After brazing with the oxyacetylene torch, an attempt was made to remove the flux by placing the brazed specimen in water and ultrasonically cleaning for 10 minutes. Some flux remained after this cleaning attempt. The specimen was then fired at 680°C to determine possible detrimental effects in service.

(b) Using an Induction Furnace.

Heat input to a brazement can be controlled more accurately than with torches by using an induction furnace with a variable output. For these initial experiments a 1kW induction furnace was found to be adequate. The specimen to be brazed was held vertically in an insulating material (Figure E-1). The approximate temperature was monitored

using a chromel-alumel thermocouple inserted into a second contact. This latter contact does not contain any of the coated silver wire, and does not reach the same temperature as the brazing specimen, but monitoring its temperature allows good control over heating and cooling rates, as well as maximum temperature reached. Both pins were enclosed in a Vycor tube, and a slight flow of argon was used to provide an inert atmosphere.

A mixture of Nicrobraz cement and coarse particles of the Au-Ni-In alloy were placed inside the contact brazing pot, the plated wire inserted and a paste of Au-Ni-In filings and Nicrobraz cement placed around the contact/wire shoulders. Initial experiments employed commercial purity argon. Two specimens brazed using this procedure are shown in Figures E-2 and E-3 . Specimen A (Figure E-2) was made using a greater argon flow than specimen B (Figure E-3). The increased argon flow resulted in more braze material left at the shoulder. Sections through these specimens, (Figures E-4,E-5) showed much better flow of braze material into the gap when the flow rate of argon was reduced. Figure E-6 shows a third specimen brazed with high purity argon. In this case, the brazing alloy flowed so well that it was able to flow out the vent hole, leaving a deficiency of brazing material around the wire (Figure E-7).

E-5 HIGH TEMPERATURE OXIDATION RESISTANCE

Two brazed specimens were conditioned in air at 680°C, for 240 hours. One specimen was brazed using the Nicrobraz flux (Para. E-4.1-a) the other brazed in argon without flux, (Para. E-4.1-b).

Definite evidence of increased oxidation could be seen in the area where flux was present before this high temperature test (Figure E-8). Since the flux is not easily removed, its use is highly undesirable. The other specimen, however, showed a uniform black oxide coating.

E-6 FURTHER INVESTIGATION

Further experiments will be undertaken to optimize the amount of brazing material and the brazing procedure.

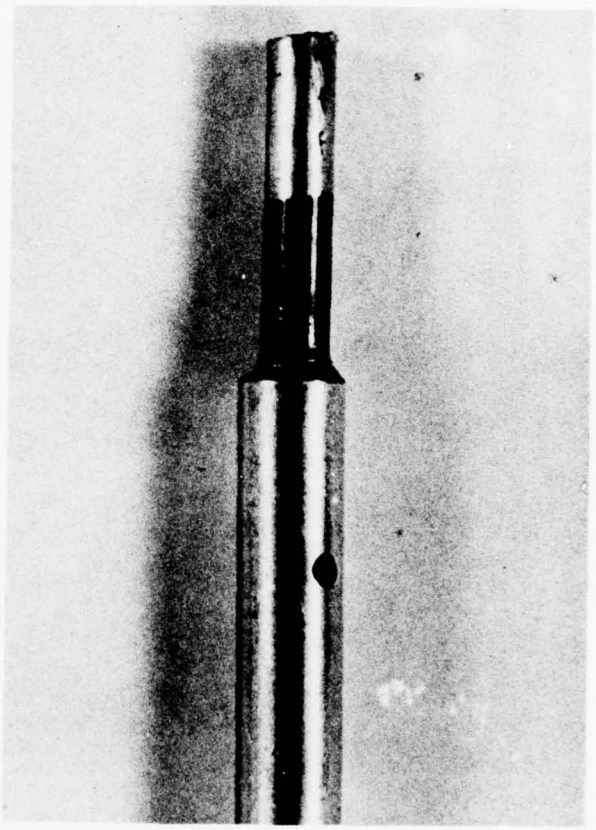


Figure E-2

Plated stainless steel pin contact brazed to wire using high flow rate of commercial purity argon, induction heating (Specimen A).
4.8X

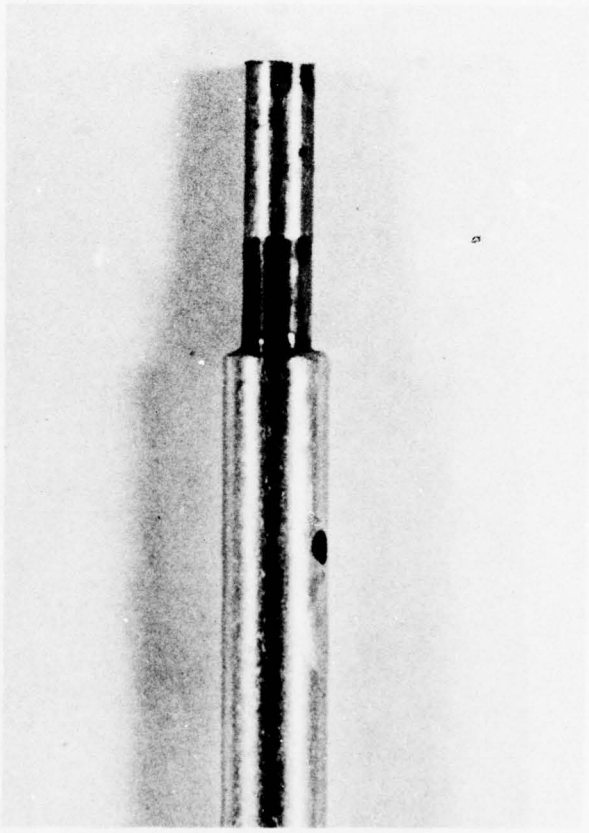


Figure E-3

Plated stainless steel pin contact brazed to wire using low flow rate of commercial purity argon, induction heating (Specimen B).
4.8X

Figure E-4

Section through Specimen A. (Figure E-2)
26X

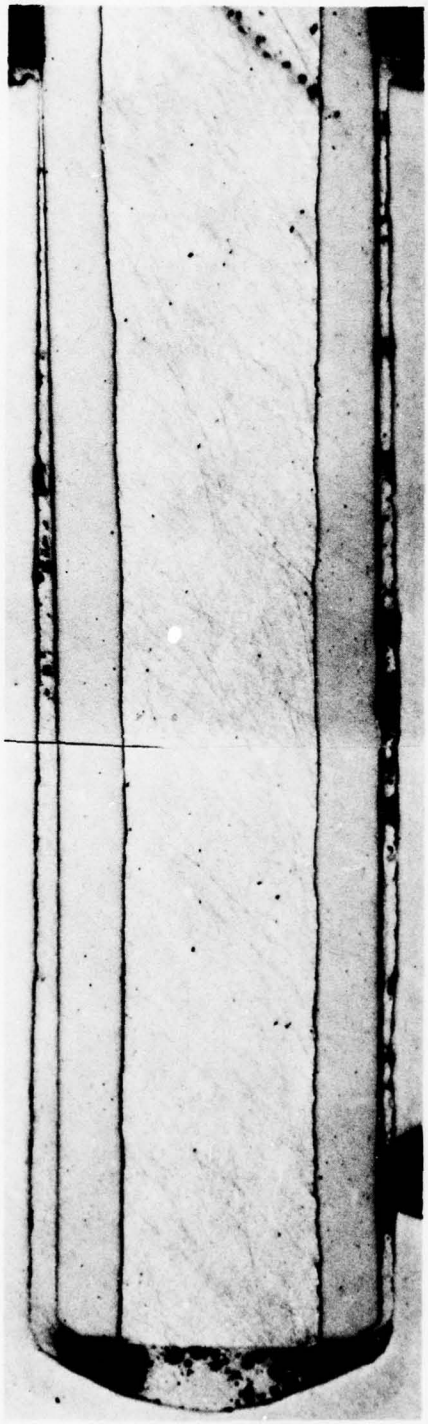


Figure E-5

Section through Specimen B (Figure E-3)
26X

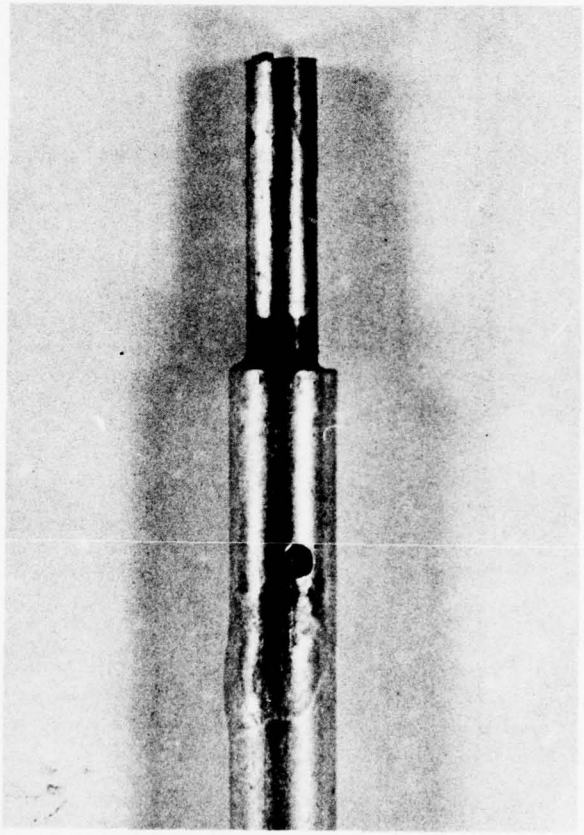


Figure E-6

Plated stainless steel pin contact brazed to wire using High Purity Argon, Induction (Specimen C).

4.8X

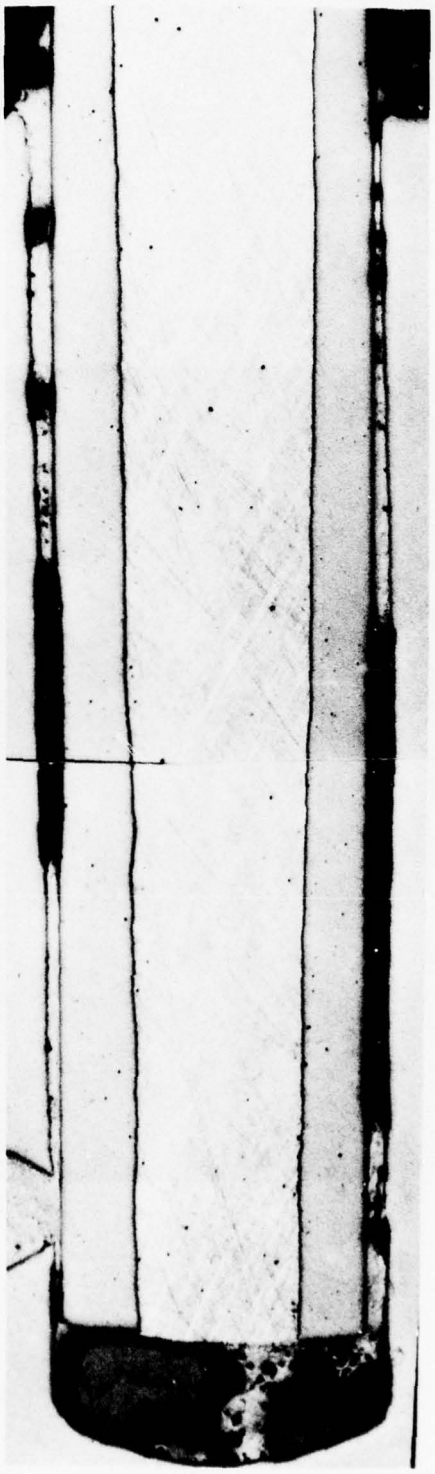


Figure E-7

Section through Specimen C. (Figure E-6)
26X

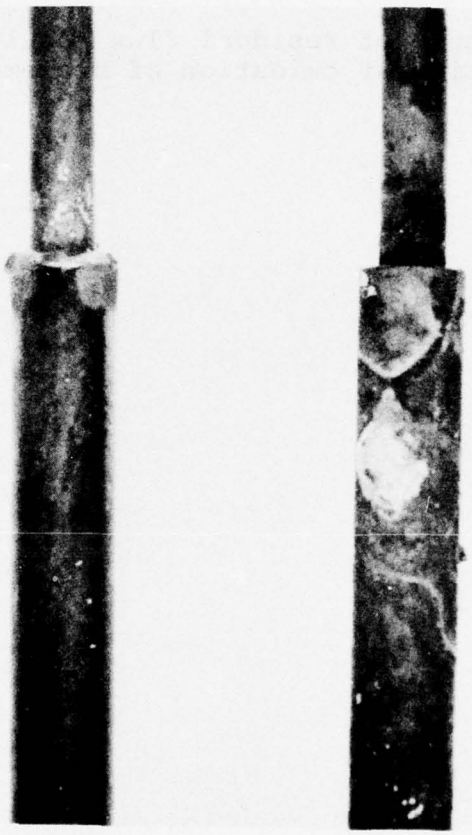


Figure E-8

Brazed specimens after 240 hours at 680°C.
4.8X

E-6 CONCLUSIONS

From the test results the following conclusions are drawn:

(a) Wire and contacts may be brazed together using an Au-Ni-In alloy, without flux, in an argon atmosphere.

(b) The presence of residual flux results in an undesirable acceleration of oxidation of brazements.

APPENDIX F

METALLIZATION OF 99.5% ALUMINA INSERTS

F-1 APPARATUS

The apparatus shown schematically in Figure E-1 was used to metallize pure Al_2O_3 inserts. This tube assembly was placed into a silicon carbide element furnace such that the end of the tube fitted with the watercooled flange protruded from the furnace. The tube was first evacuated and then filled with a 96:4 v/v N_2:H_2 gas mixture. The pressure of the gas mixture was maintained by using a water trap. The gas flow through the system was kept maintained at a very low rate of one bubble every second issuing from a 3/8" pipe against a 3" head of water.

F-2 APPLICATION OF METALLING COMPOUND

Transene®([17]) type 300 Moly-Manganese Paint was used as the metallizing compound. In initial experiments, extra Mo-powder was added to this commercial compound. Since this addition did not effect noticeable improvements in the metallized product, this procedure was discontinued and the as-received Type 300 paint was used for further work. The paint was applied by brush to ultrasonically cleaned 99.5% Al_2O_3 insulating beads. Due to the volatility of the solvent in the Type 300 paint, the painted beads dried very rapidly in air.

F-3 FIRING

The painted beads were placed on the zircon porcelain plaque which was positioned in the cold zone, (the water-cooled end of tube), of the system. The hot zone of the tube was raised to ∿800°C. The system was then closed, evacuated and the N_2-H_2 gas atmosphere was established. The temperature of the hot zone was then raised to 1250°C. When this temperature was reached the porcelain plaque with the samples was inserted into the hot zone using the push rod, and kept there for 2-1/2 hours. The furnace was then allowed to cool to 950°C. At this temperature, the tube assembly was pulled out of the furnace and cooled in air. The gas flow was maintained until the tube had cooled to room temperature.

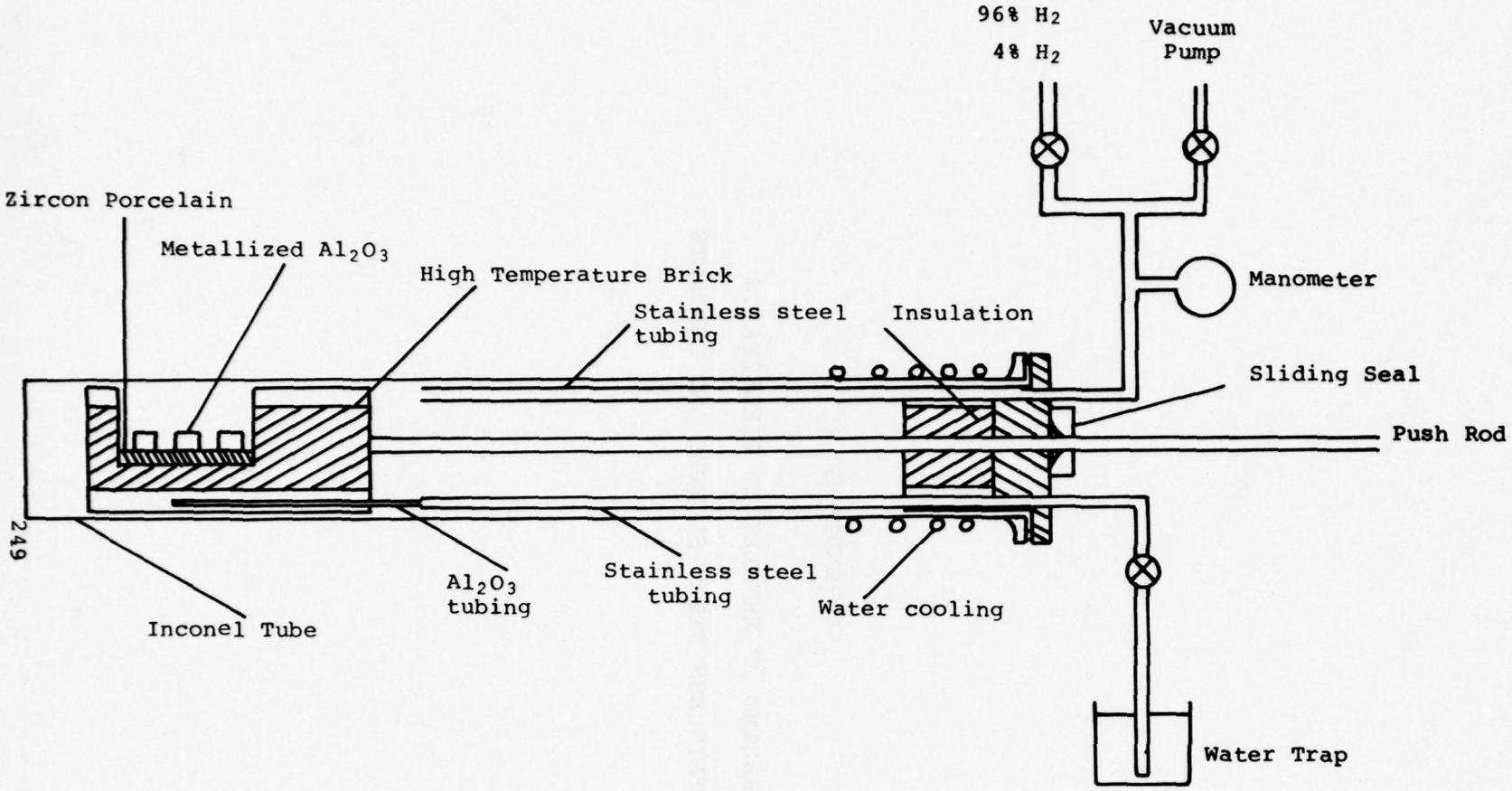


Figure F-1

Schematic cross-section of metallization apparatus.

APPENDIX G

BRAZING OF METALLIZED DIELECTRICS TO STAINLESS STEEL SLEEVES AND CONTACTS

G-1 METHOD

G-1.1 Cleaning of Components

The Ni plated sockets and pins were soaked in trichlorethylene and absolute ethanol respectively for 5 minutes each and blown dry in a warm air jet.

The front ceramics (insulators) and housings were ultrasonically cleaned in trichlorethylene and absolute ethanol respectively for 5 minutes each and dried in a warm air jet.

G-1.2 Brazing Sockets and Pins into Rear Insulators

Water base Green Stop-off was applied around the socket (pin) circumference immediately in front of the rear insulator. (Figure G-1)

The sockets, pins and insulators were then assembled and mounted on their respective brazing jigs. (Figure G-2)

Water base Green Stop-off was applied around the socket (pin) circumference approximately 1/16" behind the rear insulator. (Figure G-3)

N-30 brazing powder was then applied around the circumference of the sockets (pins) and binder (Nicrobraz Cement, viscosity 500) was applied to keep the powder in place. Figure G-4 shows the braze material prior to the brazing operation.

The assembly was positioned in the induction coil such that the field was concentrated on the rear insulator. (Figure G-5)

The vacuum chamber was evacuated to 4×10^{-6} Torr and the heating cycle started. Figure G-6 shows the equipment used.

Because of the large gap between the induction coil and the sockets (pins), approximately 2 kW of power was required to melt the N-30 braze material. A heat-up time of 1-1/2 hours was employed with a 5 minute hold time at 1150°C, followed by a 4 hour cool-down to room temperature.

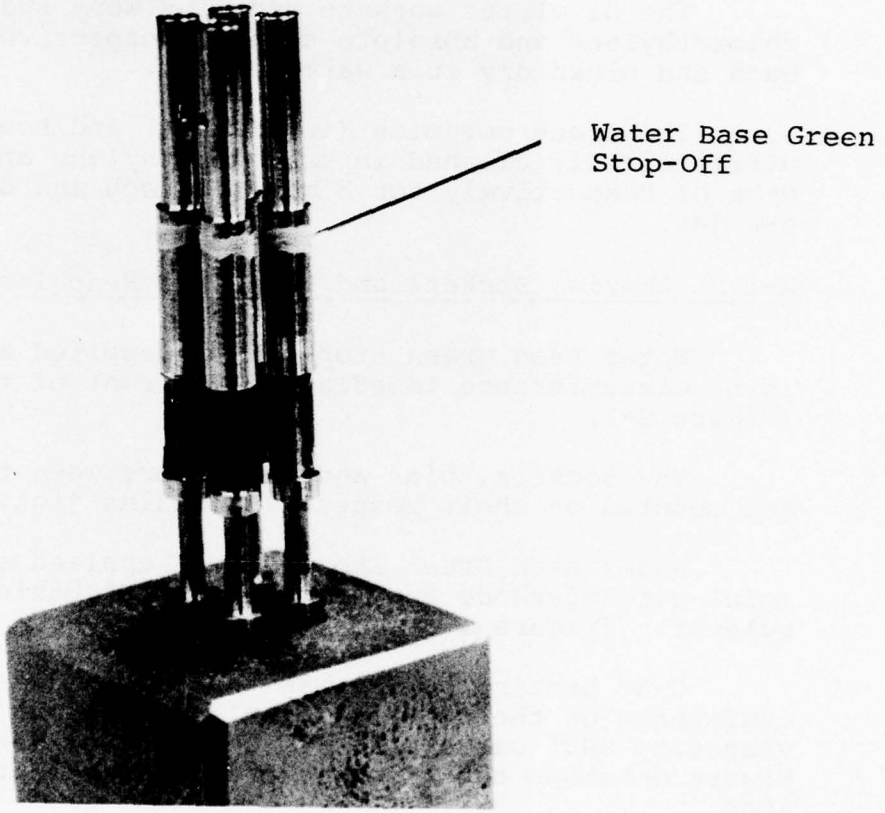
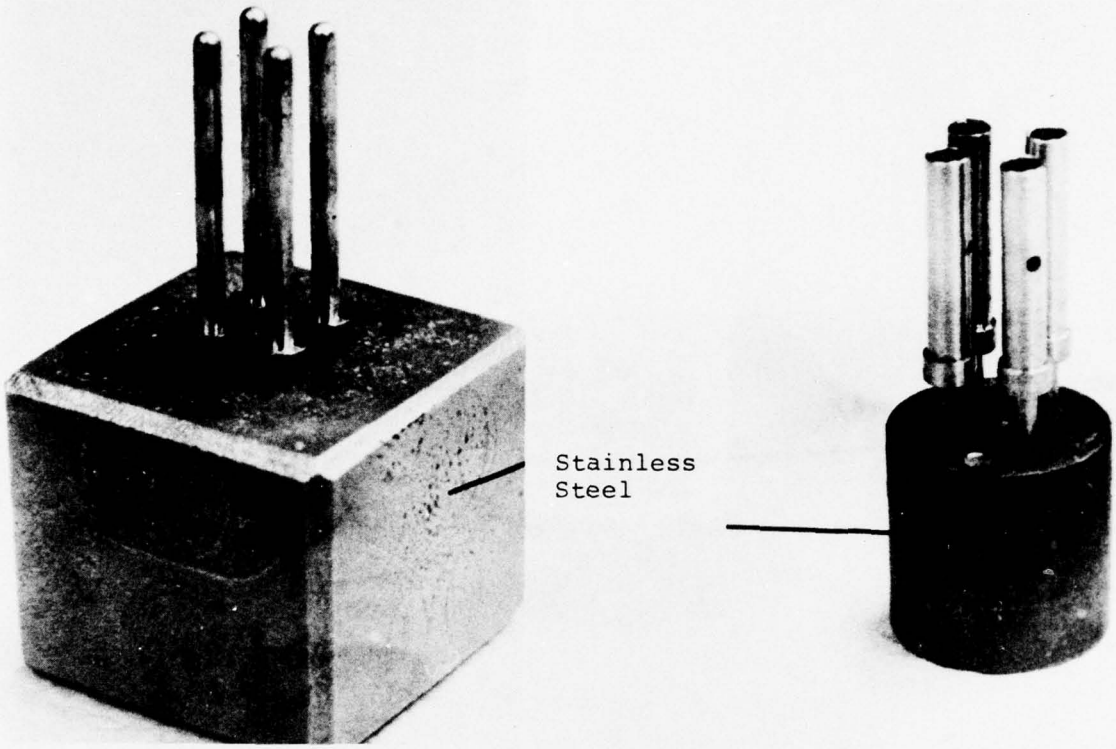


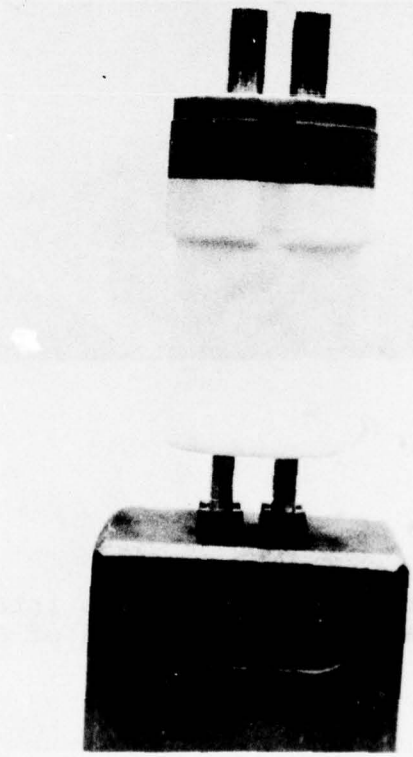
Figure G-1

Brazing sockets and pins into rear insulators - application of Stop-Off to pins.



Socket Jig

Pin Jig



Sockets and Insulator Assembly

Figure G-2 Brazing sockets and pins into rear insulators - jig assemblies.

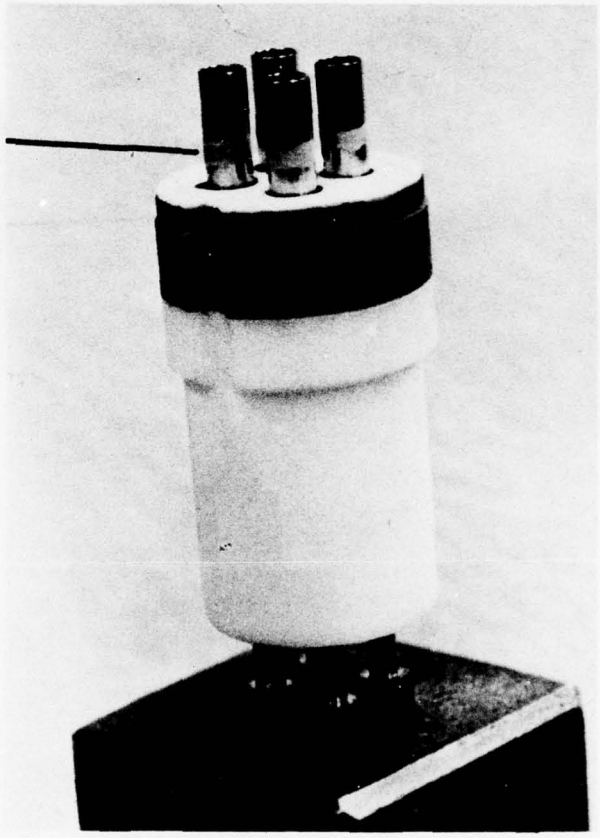


Figure G-3

Brazing sockets and pins into rear insulators - application of Stop-Off.

N-30 + Binder

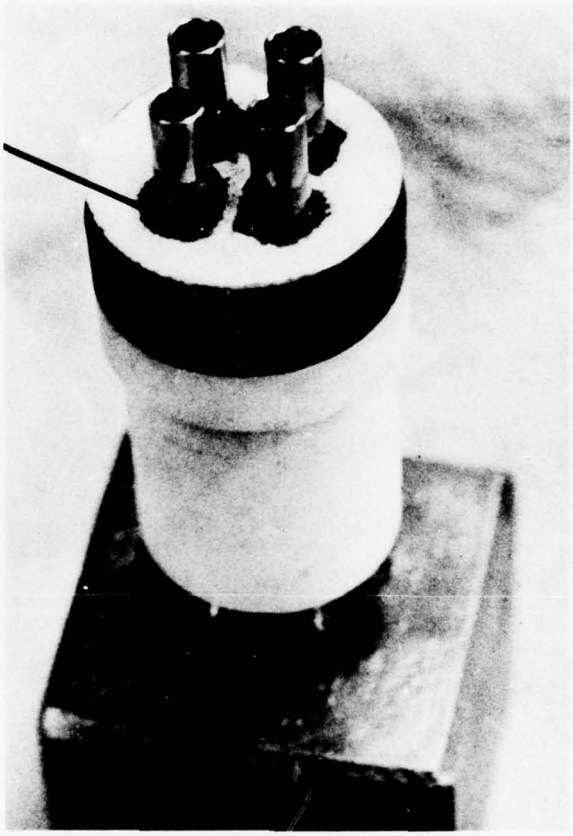


Figure G-4

Brazing sockets and pins into rear insulators - assembly prior to firing.

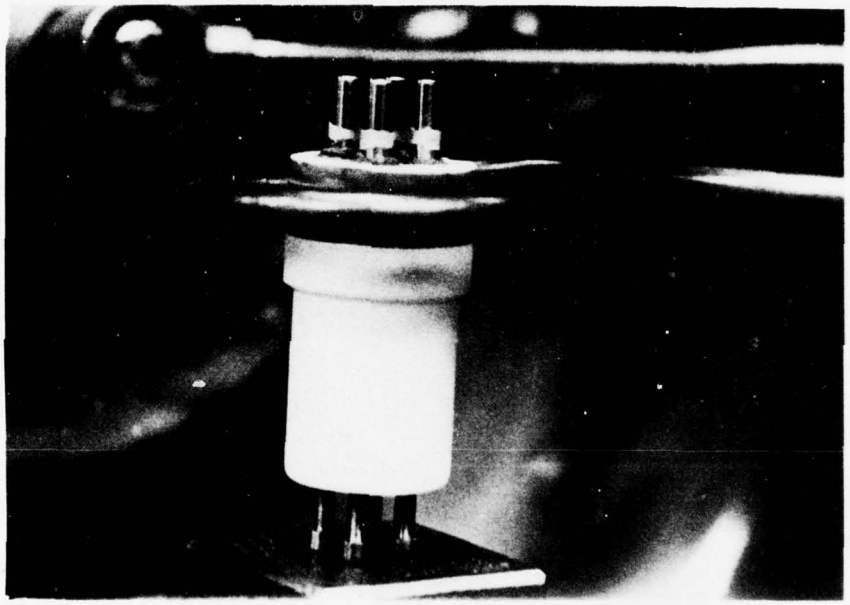


Figure G-5

Brazing sockets and pins into rear insulators - Assembly positioned in induction coil.

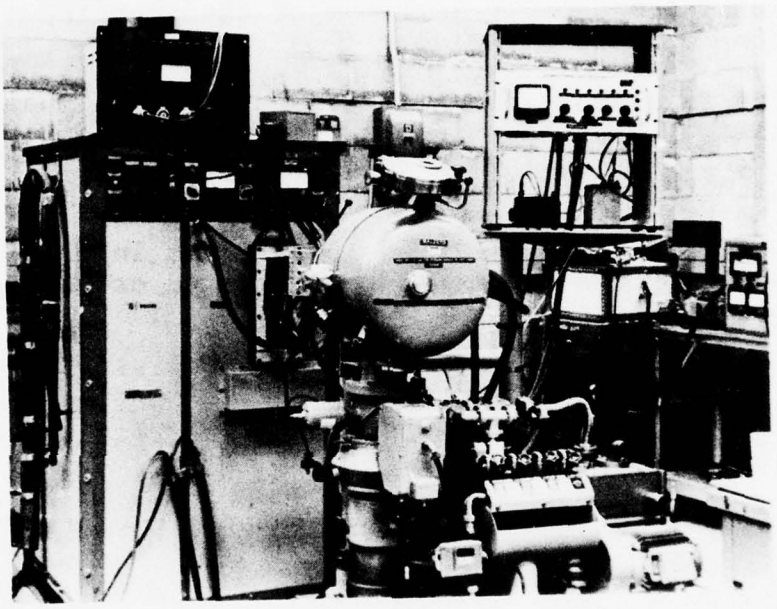


Figure G-6

Brazing sockets and pins into rear insulators - overall view of vacuum furnace.

G-1.3 Brazing Housings to Rear Insulators

Oxide build-up from the spot welding of the key was mechanically removed using a fine scraper and jewellers files.

Green Stop-off was applied around the inside diameter of the housing immediately in front of the rear insulator and along the rear edge. (Figure G-7)

The insulator-socket (pin) assembly was positioned in the housing. N-30 and binder were applied along the inside diameter of the housing to form a large fillet of braze material. (Figure G-8)

The housing assembly was positioned in the induction coil such that the field was concentrated around the rear insulator. (Figure G-9)

The same thermal cycle was employed as described in Section G-2. Since the gap between the housing and induction coil was small, only 0.8 kW of power was required to melt the N-30 braze material.

Figure G-10 shows a dummy pin assembly after brazing.

G-2 DISCUSSION

G-2.1 Brazing of Pins and Sockets into Rear Insulators

After nickel plating the metallized holes in the rear insulators, pins and sockets were brazed into their respective insulators. No difficulties were encountered in this operation. Although brazing powder, rather than the more desirable braze preforms were used, filling of the brazing gap was achieved. It is felt that ease of brazing is mainly due to the creation of a very small gap between the mating faces to be joined which results in capillary action drawing the molten braze material into the interstice. The reduction of the gap between the stainless steel contacts and the insulator during brazing is due to the significant difference in their thermal expansion coefficients.

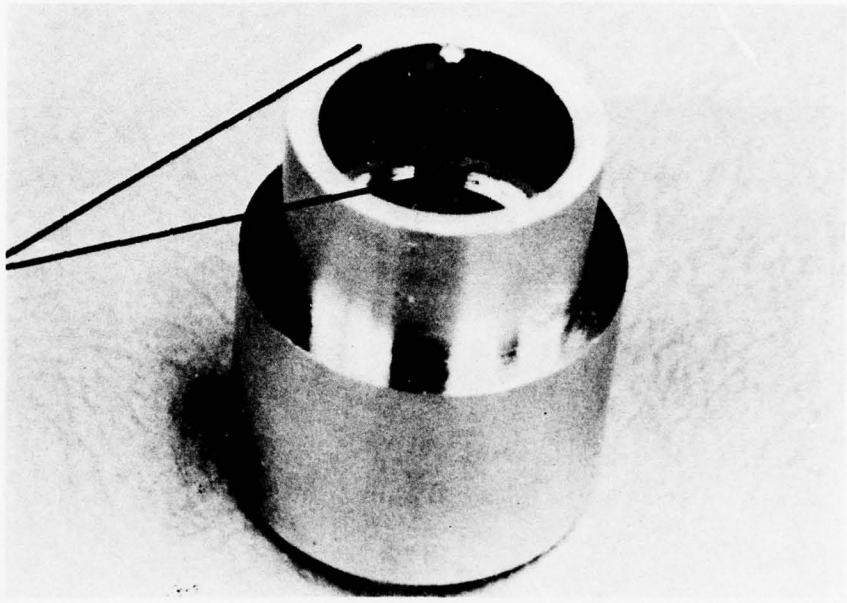


Figure G-7

Brazing sockets and pins into rear insulators - dummy shell.

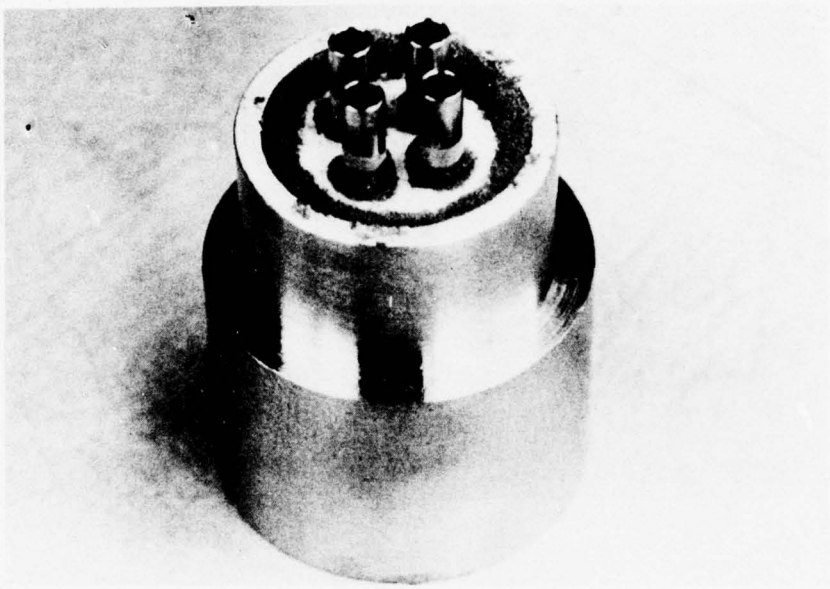
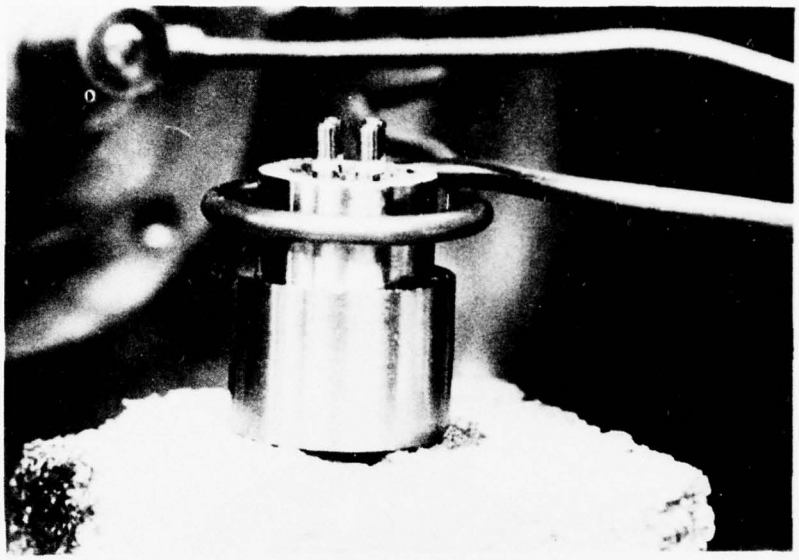
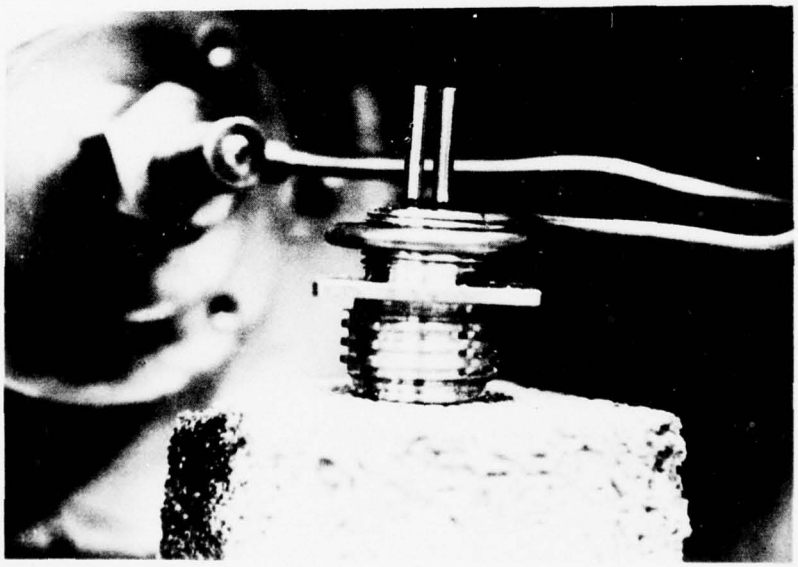


Figure G-8

Brazing sockets and pins into
rear insulator/housing - insulator
socket assembly inserted.



Dummy Pin Assembly



Prototype Socket Assembly

Figure G-9

Brazing sockets and pins into rear insulator/housing - assemblies inserted into induction coil.

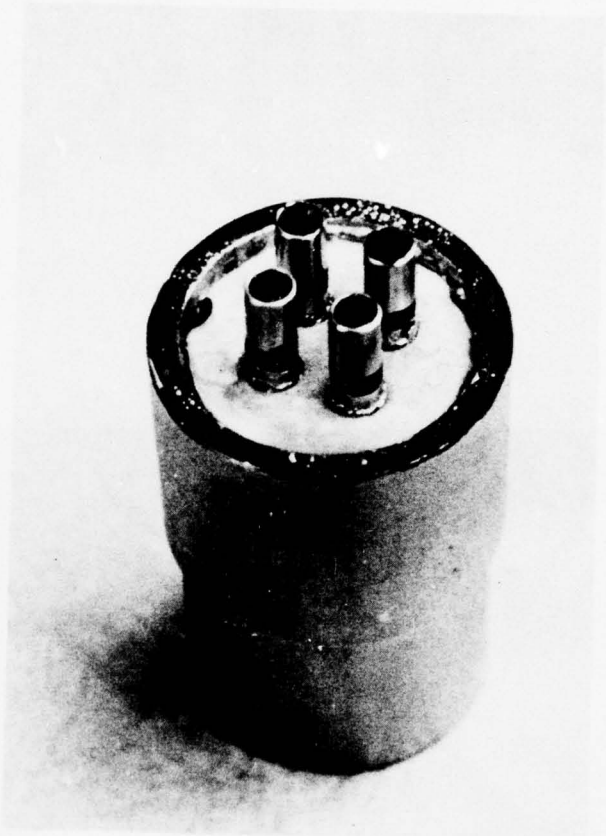


Figure G-10

Brazing sockets and pins into rear insulator/housing - dummy pin assembly after brazing.

G-2.2 Brazing of Insulators into Connector Housings

To establish working parameters, rear insulators were brazed into dummy shells. In these experiments it was found that, contrary to the effect described above, on heating to brazing temperature the clearance between the insulator and shell increased. Due to this expansion, the clearance between shell and insulator became so large that insufficient powder braze material could be introduced to produce a continuous fillet. Furthermore, because the dummy shells had no key but the insulators had a keyway, much of the braze material was drawn into the open keyway. Typical brazed prototype and dummy shells are shown in Figures G-9 and G-10.

Although the increase of the shell-insulator clearance suggested possible brazing problems might be encountered, it was decided to proceed with the brazing of prototype insulators into prototype connectors. Because the prototype shell housing has a key, it was hoped that acceptable fillets could be obtained in spite of the expansion effect. As the socket housing has a mounting flange on which the induction field concentrated, a different induction coil had to be designed and the control electronics of the generator had to be changed to give much better control in the low power region.

To braze the insulators into the prototype connector housing, generous amounts of Nicrobraz 30 powder was applied to the brazing joint. Poor fillets resulted, however, due to the gap opening effect and the fit between key and keyway. This brazing operation did not produce the desired joint between insulator and connector housing. To improve the joint, more brazing powder was added and brazing was repeated. In this attempt the insulator developed a crack running from one hole radially outward towards the keyway. This cracking is probably due to the expansion on heating of the contacts and the shell which combine to exert a tensile force in the insulator. Although the second brazing operation gave more fill to the brazing gap, the fillet shape suggested that incomplete wetting of the alumina occurred. It is suspected that this is due to the metallized layer not being strongly bonded to the alumina due to the low metallizing temperature. Thus, during the initial brazing operation, regions of the metallized layer which are not immediately wetted by braze metal recede along its edge exposing bare alumina. This latter is very poorly wetted by brazing powder.

These results indicate that successful joining of the insulator to the connector housing must be done in one brazing operation i.e. one heating cycle, which would be possible if enough brazing material could be supplied to the joint by using preforms instead of powder. The use of powder may be possible, however, if the insulator-housing clearance is kept small - i.e. .0002 to .0004 inches diametral clearance at room temperature, which at brazing temperatures would yield a very small gap.

Throughout the efforts to produce acceptable brazes, the key-keyway configuration between the connector housing and insulator was found to interfere with the production of acceptable brazes. The key is spot-welded to the shell which results in an oxidized gap between the shell and key. During brazing the filler material does not wet the oxide and thus does not flow into this gap, resulting in a cavity in the joint between shell and insulator. Furthermore, standard key and keyway fit tolerances necessitate the introduction of relatively large amounts of brazing material in this region.

Before brazing the last connector housing, the oxide associated with the key was removed by mechanical and chemical means. The gap between the key and the shell was then closed by an initial brazing operation. The resulting fillets were then machined to conform closely to the keyway in the insulator. The insulator was then brazed into the shell housing. Unfortunately, the initial braze under the key became very hot and fluidized before the brazing powder for the shell-insulator bond reached its flow point. This led to braze flow-out from under the keyway resulting in an imperfect joint.

During the experiments it was found that thermal expansion of the housing led to a permanent expansion in the region of the brazed in rear insulator. This may be due to the fact that as the brazing material solidifies at ∿1150°C and the coefficient of expansion of the insulator is very low, little further contraction of the housing occurs on cooling below 1150°C.

G-3 ACTION

Modifications will be made to the second set of prototype connectors to reduce the tolerance between the insulator and housing and to eliminate a portion of the connector housing key in the area of the braze.

All brazing will be performed in one operation, preferably using brazing preforms.

Discussions will be held with a potential supplier to use their production furnace facilities which would allow metallization in the optimum 1450°C-1600°C range.

APPENDIX H

OXIDATION RESISTANCE OF

BRAZED CONTACTS/CONDUCTORS

Selected specimens of the contact-conductor combinations discussed in Appendix E were sectioned after thermal exposure and tensile strength testing to study the effects of high temperature exposure on oxidative stability.

a) 308L Stainless Contact-Conductor Combination

A section of one side of this braze is shown in Figure H-1 and is typical of all the circumference of the conductor. The original brazed joint is visible as a straight line with a few small regions of porosity. Diffusion has permitted a large fraction of the braze material to move into the 308L contact, while relatively little diffusion into the Inconel 601 coating on the conductor has taken place.

To show the resulting distribution of elements, the grey rectangular area in Figure H-1 was examined by electron microprobe. The resulting scans are shown in Figure H-2 and a higher magnification optical microscope photo of the scanned area in Figure H-3. In comparing Figures H-2 and H-3 it must be remembered that (a) the left and right sides are reversed and (b) the microprobe scan is compressed laterally by the electronics, in order to display a wider region on the cathode ray tube. Comparison of Figures H-3, H-2(a), and H-2(b) shows that the grey areas of the optical microscope pictures correspond to regions rich in gold, and thus have high conductivities. Figure H-2 (b) - H-2 (d) clearly show that where the gold content is high, there is a low nickel and iron content. This is consistent with limited solid solubilities of nickel and iron in gold, and vice versa, at 680°C.(27). The chromium distribution also appears to indicate limited solubility of this element in gold. The chromium content is higher further away from the original brazed joint, but the nickel, and iron contents are higher closer to the brazed joint than in the original 308L stainless steel (Figure H-2, (c)-(e)). Thus the gold has diffused into the contact, with a counter flow mainly of iron and nickel. The irregular pattern of the gold distribution suggests that it may be occurring by grain boundary diffusion. Despite the complexity of this diffusion pattern, the absence of large voids and oxides in this specmen suggests that both the mechanical and electrical properties of this combination would be acceptable after 1000 hours at 680°C (1250°F). Tensile specimens to test this assertion were prepared.

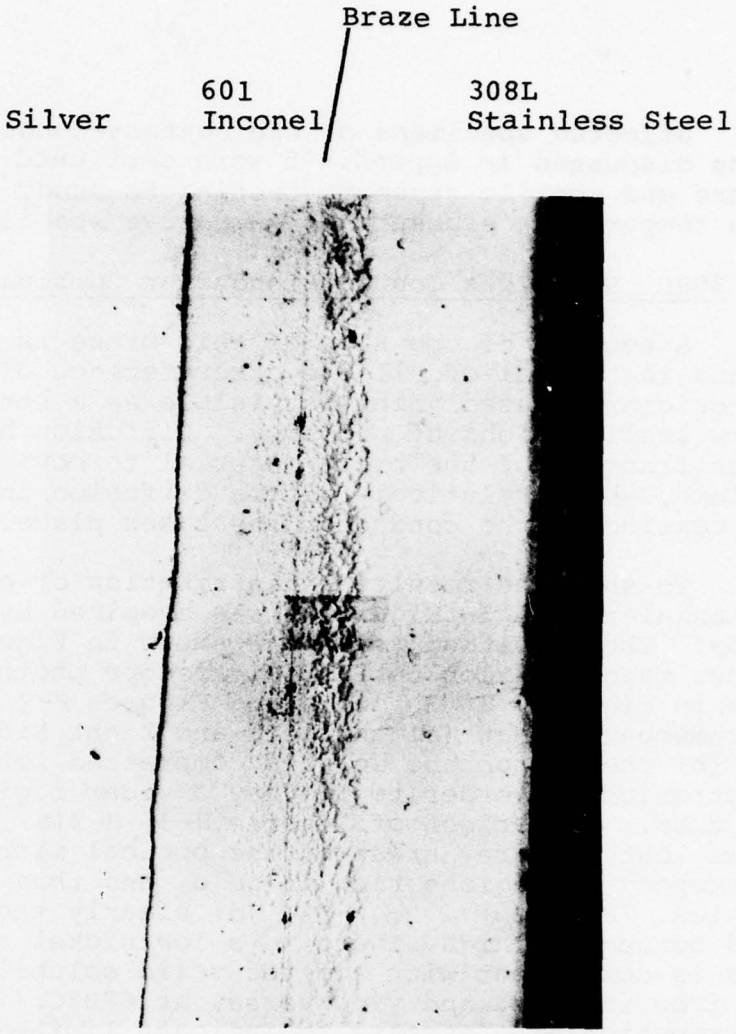


Figure H-1

Cross-section of stainless contact/ experimental conductor.

Cross-section of 308L stainless steel Ni plated contact brazed with (AuNiIn) alloy to Ni plated Inconel 601 Clad Ag conductor, and aged 1010 hrs at 680°C. The grey rectangle is the area of micro-probe analysis. 53X

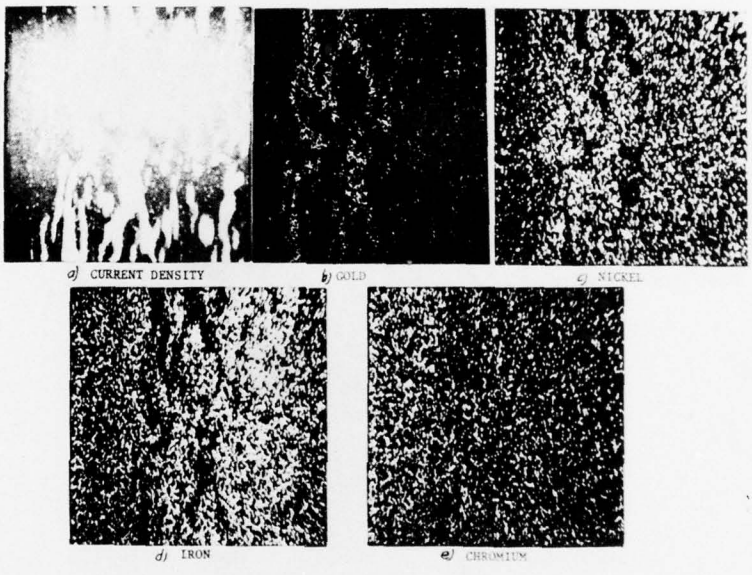


Figure H-2

Microprobe analysis of area in Figure H-1
400X

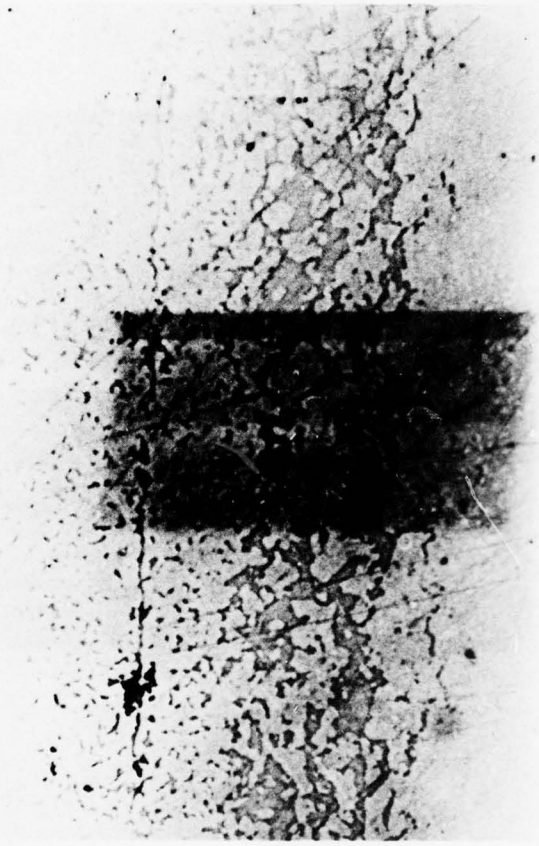


Figure H-3

Micrograph of microprobe analysis area in Figure H-1. 210X

AD-A046 406 CANADA WIRE AND CABLE TECHNOLOGY DEVELOPMENT DIV POI--ETC F/G 9/1
DEVELOPMENT OF HIGH TEMPERATURE (1250 F) WIRE AND CONNECTORS.(U)
JUL 77 M A DUDLEY, F D BAYLES, P O SCADDING F33615-75-C-2040

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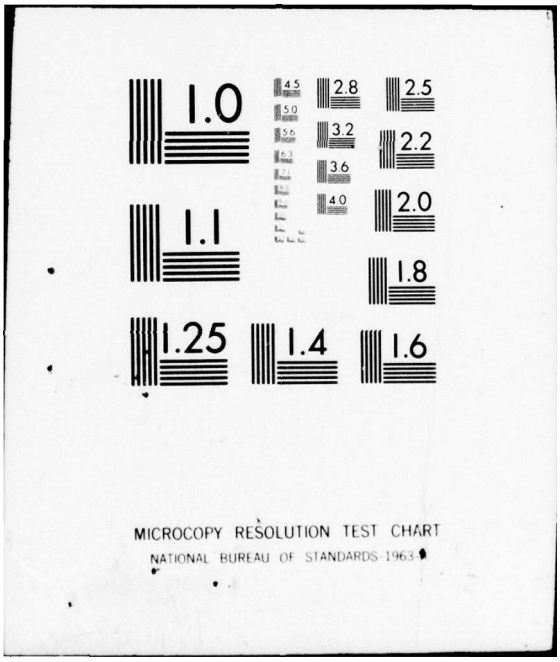

1.0
4.5
5.0
5.6
2.8
3.2
3.6
4.0
2.5
2.2
2.0
1.8
1.1
1.25
1.4
1.6
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963

b) Inconel 601 Contact-Conductor Combination

This specimen appeared to have a complete fillet both directly after brazing and after 1010 hours at 680°C. Micro-examination, however, showed a second phase, plus some small cracks, along the original brazed joint, (Figure H-4). The number of particles of this second phase decreased as the distance from the fillet increased, suggesting that the phase was formed during the high temperature oxidation test. A region close to the fillet, containing both this new second phase and some cracks, was examined by electron microprobe. An optical microphoto of this region is shown in Figure H-5 , and the corresponding electron microprobe scans are shown in Figure H-6 . (As for Figures H-2 and H-3, the sides of the microprobe-optical microscope photos are reversed, and the microprobe scans compressed laterally). The grey areas within the contact and along the brazed joint are again rich in gold, and have resulted from gold diffusion into the contact, with very little gold in the conductor cladding, despite the fact that both are Inconel 601. The low chromium, nickel and iron contents in gold rich areas are clearly seen in Figure H-6(c)-(e). As in the 208L stainless contact-conductor specimen, the diffusion of gold has resulted in the counter movement of nickel and iron, but the chromium content shows the highest concentration adjacent to the original brazed joint. In addition, although its content within Inconel 601 is only approximately 1%, a high aluminum content was found in the new second phase at the brazed joint (Figure H-6(f)). Since the aluminum is easily oxidized, this suggests that the new phase is an oxide, and that chromium has also diffused to the surface in response to the presence of oxygen. The formation of an oxide in this specimen is believed to be due to the presence of small microcracks along the brazed joint allowing the movement of oxygen down the joint. As oxides form, the resulting increase in volume may also generate stresses severe enough to allow micro-cracking to propagate further down the braze. Some small cracks were observed further down the brazed joint where oxide formation had not yet taken place. This oxide formation re-emphasizes the importance of eliminating micro-cracking during the brazing operation. These specimens were made when the formation of microcracks was still being investigated. As discussed in previous sections, careful nickel plating on the contact and conductor is required. Besides controlling the plating current conditions, ultrasonic cleaning of the plated contacts appears to be detrimental. In addition, brazements are now cooled slowly after manufacture, in order to reduce thermal gradients and thus

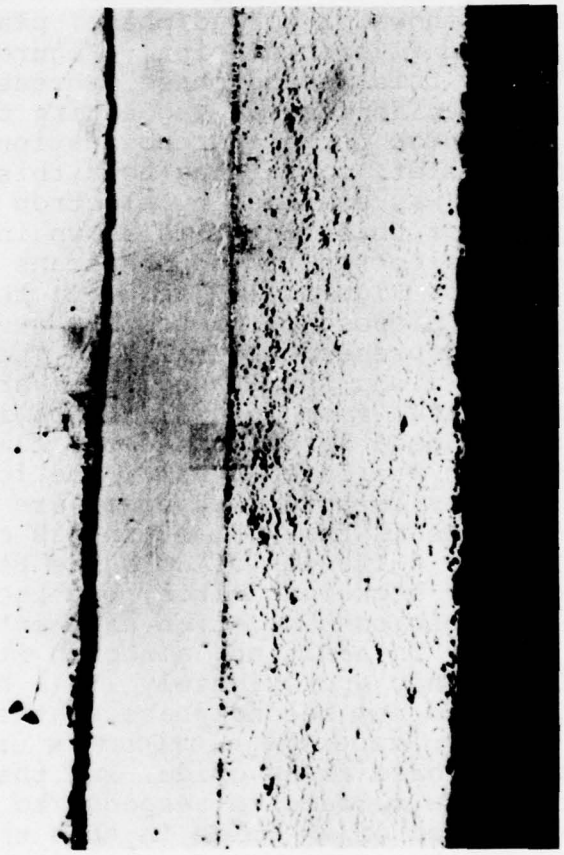


Figure H-4

Cross-section of Inconel 601 contact/ experimental conductor.

Cross-section of Inconel 601 Ni plated contact brazed with AuNiIn alloy to Ni plated Inconel 601 clad Ag conductor and aged 1010 hrs at 680°C. The grey rectangle is the area of microprobe analysis. 53X

Figure H-5

Micrograph of microprobe analysis area in Figure H-4. 533X

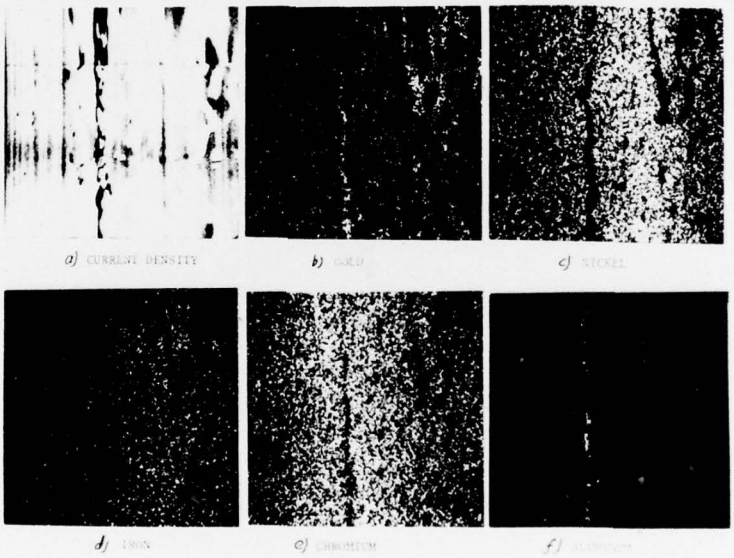


Figure H-6

Microprobe analysis of area in Figure H-4. ≈400X

Figure H-7

Cross-section of microcrack free braze joining Ni plated Inconel 601 (0.090" I.D.) to Ni plated Inconel 601 clad Ag conductor. 213X

the thermal stresses during solidification of the braze. This careful control of plating and brazing appears successful in eliminating microcracks (Figure H-7) and should therefore aid oxidation resistance.

APPENDIX I

TENSILE STRENGTH TESTING OF BRAZED CONDUCTOR/CONTACT JOINTS

I-1 METHOD

Tensile tests of brazed conductor/contact assemblies were performed at room temperature and at 1250°F. Tensile samples were prepared by brazing contact pins to the ends of a 1.4 inch length of the Inconel clad silver conductors. The tests were performed on an Instron machine. For the high temperature test, 8" lengths of 0.125" diameter Inconel wires were welded to the ends of the contact pins so that the tensile grips could be located outside the furnace. The temperature profile of the tubular furnace used was flat over a 1.5" zone. The test sections of the specimens were located in this zone. The temperature was recorded by a thermocouple contacting the test specimens at the central point of their test section.

A crosshead speed of 0.005"/minute was used for the room temperature test and 0.2"/minute for the 1250°F test. The higher strain rate for the higher temperatures was used to forestall any possibility of recovery creep during the tensile test.

I-2 RESULTS

A typical load-elongation curve for room temperature is given in Figure XXII. Microphotographs of typical conductor failure are shown in Figures XXIII and XXIV. Figure XXV illustrates a typical conductor failure after a test at 1250°F. Discussion of the results may be found in the body of the text.

APPENDIX J

CALCULATION OF EXPECTED LOADS FOR ROOM TEMPERATURE AND 1250°F TENSILE TESTS OF CONDUCTOR/CONTACT PIN ASSEMBLIES

O.D. of Inconel cladding = 0.081"

I.D. of Inconel cladding = 0.055"

Cross-sectional area of Inconel in conductor:

$$A = \frac{\pi}{4} [(O.D.)^2 - (I.D.)^2]$$

$$= \frac{\pi}{4} [(0.081)^2 - (0.055)^2]$$

$$= \frac{\pi}{4} [0.0035]$$

$$= 0.00278 \text{ in}^2$$

Temp. °F	Expected Weld[28] UTSx1000 psi	Expected Weld Load (lb)	Actual Conductor UTSx1000 psi	Actual Conductor Load (lb)
72	85-120	236-330	110	305
1250	50-70	138-195	40	110

APPENDIX K

AN IMPROVED INTERNAL BRAZE POT PLATING TECHNIQUE

K-1 METHOD

To obtain an improved nickel plate layer on the inside walls of contact braze pots, a special in-house technique was developed.

To promote good throwing power inside the cavity, a nickel tube, resembling a syringe needle, was inserted into the contact braze pot. The pot was also provided with a pierced side vent. The electrolyte was injected into the pot through the needle from an external reservoir providing a 12-inch pressure head, exiting through the vent. Using the tube as the anode, plating proceeded in the usual manner.

APPENDIX L

TENSILE TESTS OF AGED BRAZED CONTACT/CONDUCTOR SPECIMENS

L-1 METHOD

Tensile specimens were prepared by brazing contacts to each end of a 1.4 inch length of Inconel 600 clad silver conductor, and then exposing the samples for 1073 hours at 680°C (1250°F) in air. Upon removal from the furnace, 8 inch lengths of 0.125 inch diameter Inconel wire were welded to the ends of the contacts to act as leads for the tensile tester jaws. The wires were placed in the jaws and a tubular furnace was arranged around them to allow the tensile strength to be determined at 680°C (1250°F). The tensile tester crosshead speed was 0.2 inches per minute.

Four specimens were tested, with contact combinations as shown in Table L-1. This table also lists the ultimate loads achieved as well as the locations and types of failure. The unaged ultimate load for this temperature was found to be 110 lb. ([29]). The load elongation curves are shown in Figure L-1 . Three out of the four specimens tested after aging showed only small decreases in ultimate load, compared to the unaged specimen. These specimens included both stainless steel type 308L and Inconel alloy 601 contacts.

It was previously reported([30]) that oxide formation can take place in brazes between the Inconel-clad conductor and the Inconel alloy 601 contacts. Micro-examination of these brazes after aging and tensile testing again revealed oxide formation (Figures I-2 and I-3). Greater quantities of oxide appeared to be associated with the absence of a well developed fillet (Figure I-2 vs Figure I-3). These specimens were made before the arrival of stop-off material. In some cases, (e.g. Figure I-2), the liquid braze material was not constrained when it reached the top of the contact, and continued to run up the conductor, forming a braze-covered area on the conductor without forming a complete fillet at the top of the contact. The absence of a fillet with a large radius of curvature would concentrate thermal stresses at the top of the braze inside the gap between conductor and contact. As the gap is small, the resulting radius of curvature is also small thus the thermal stresses will be high, resulting in more microcracking and thus more oxidation. It is expected that the stop-off materials will result in more uniform fillets with larger radii of curvature. It is important to note, however, that the presence of oxide did not appreciably reduce the braze strength. Even when the braze was so poor that the conductor pulled out of the contact, its strength was 101 lbs. compared to 110 lbs for an ideal and unaged specimen. One specimen broke at a load of 61 lbs, adjacent to a fillet with a very small radius of curvature

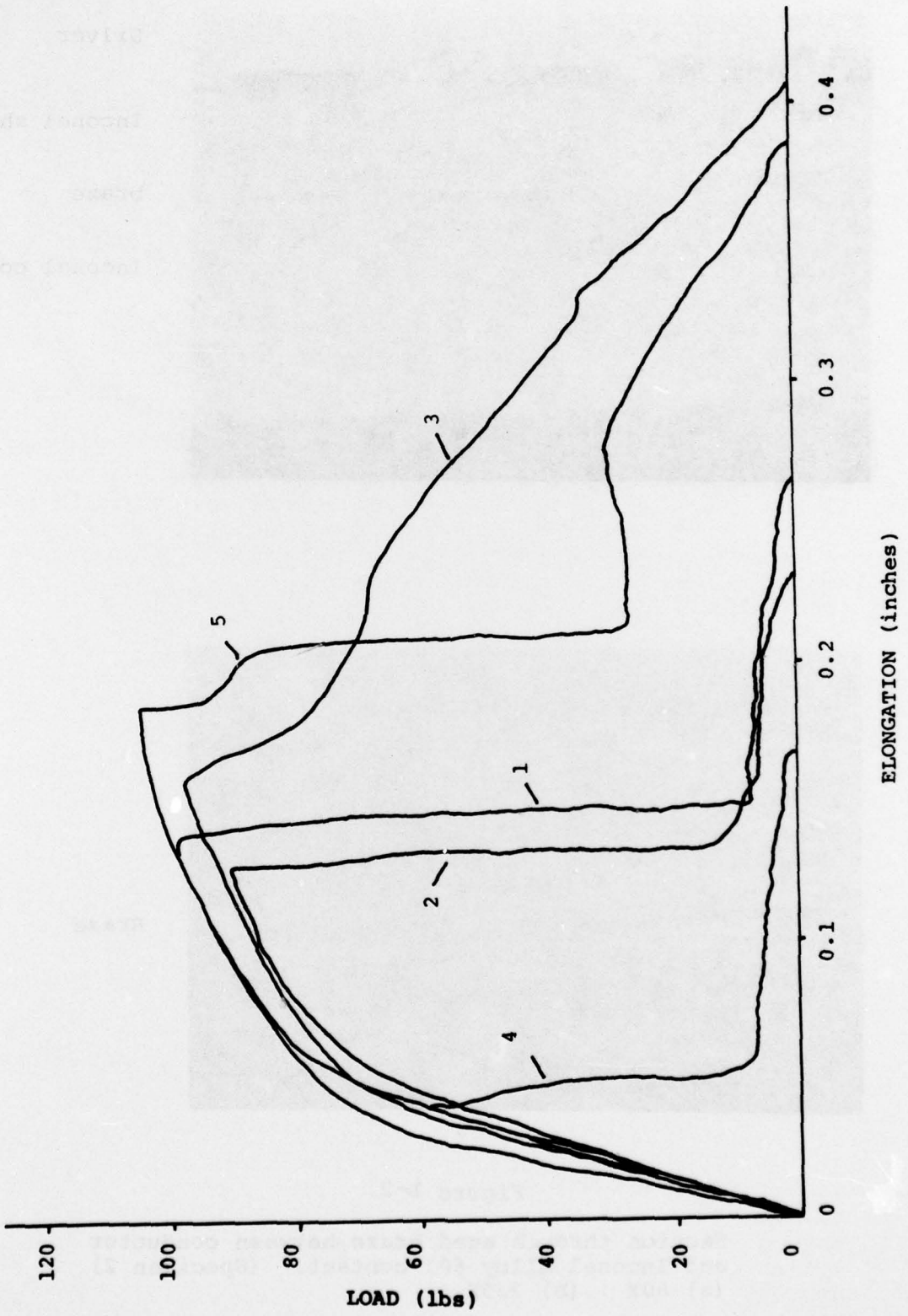
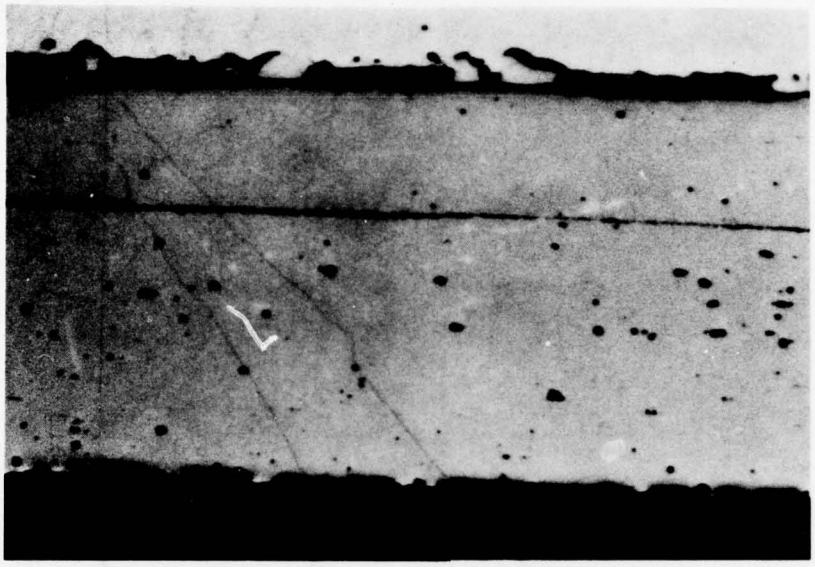


Figure L-1 Load vs Elongation curves for the contact-conductor tensile specimens.

(a)

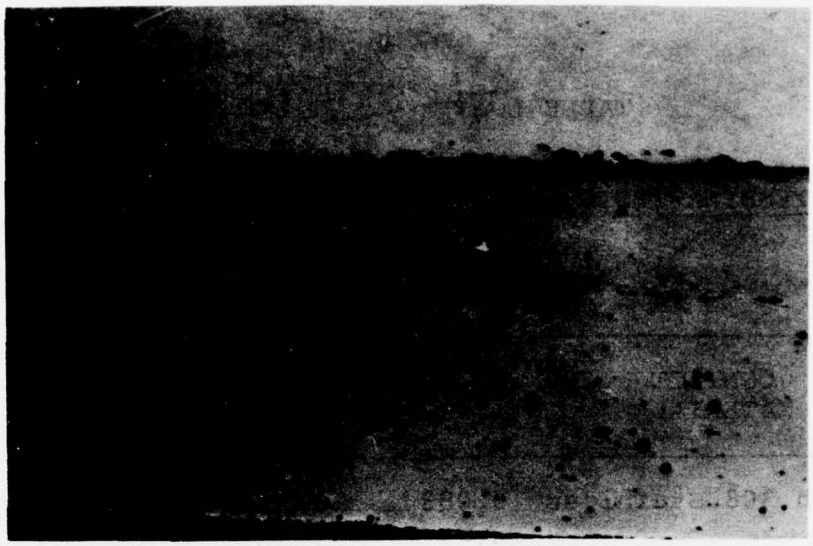


(b)

Figure L-2

Section through aged braze between conductor and Inconel Alloy 601 contact. (Specimen 2)
(a) 60X (b) 235X

(a)



(b)

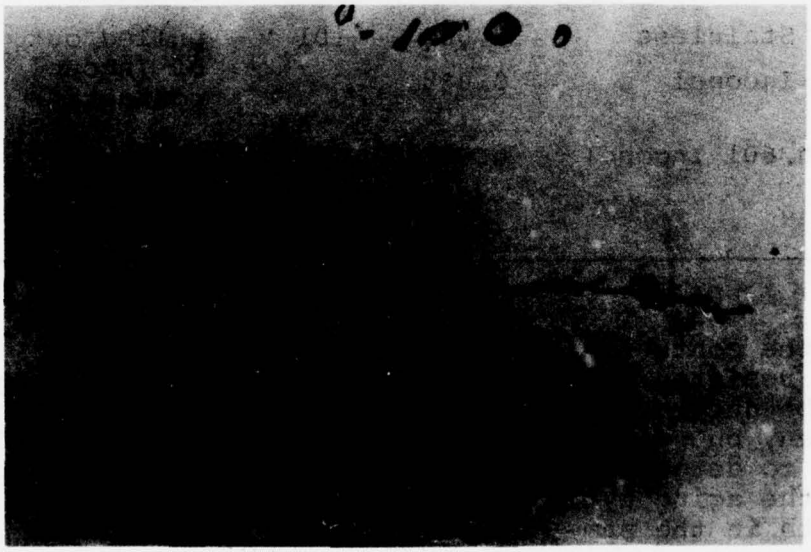


Figure L-3

Section through aged braze between conductor and Inconel Alloy 601 contact. (Specimen 4)
(a) 60X (b) 235X

TABLE L-1

RESULTS OF TENSILE TESTS AT 1250°F, AGED SPECIMENS

SPECIMEN	CONTACT MATERIAL		ULTIMATE LOAD (lbs)	FAILURE TYPE & LOCATION
1	both 308 Stainless	0.083	101	conductor at fillet
2	308 Stainless	0.083	92	conductor, inside stainless steel contact
	601 Inconel	0.085		
3	308 Stainless	0.083	101	pulled out of Inconel contact
	601 Inconel	0.085		
4	both 601 Inconel	0.085	61	conductor, at smaller fillet

at the top of the contact. Since other Inconel alloy 601 contact brazes resisted much higher loads, this lower strength cannot simply be due to the combination of materials. Another similar specimen, but with larger fillets, was aged for 1000 hours at 680°C to determine the role of fillet geometry. Upon completion of the aging cycle, the specimen was tested for tensile strength in the same manner as the previous samples.

The load/elongation curve for this specimen is number 5 in Figure L-1. It is seen that this specimen fractured under a load of 107 lbs, which is close to the ideally attainable load of 110 lbs([29]). Failure of this specimen was by inter-granular fracture in the Inconel sheath of the wire. This fracture occurred below one fillet so that final separation was a pull-out of a short length of the conductor from

the brazing pot of the contact. This fracture mode could only have occurred if some degradation in the upper end of the brazing joint had taken place. Metallographic examination showed indeed that oxidation in the contact/braze interface was present. Its metallographic appearance was quite similar to that shown in Figure L-2 .

L-2 CONCLUSIONS

While a degree of degradation of the brazed joint is evident after 1000 hours at 680°C (1250°F), this degradation does not detrimentally affect conductor/contact integrity.

With proper brazing practices, connections between Inconel contacts and conductors can be made which withstand tensile loads of at least 100 lbs.

APPENDIX M

STOP-OFF MATERIALS

M-1 STOP-OFF MATERIALS

As previously discussed, nickel plating of the contacts and conductors is necessary to ensure reproducible wetting by braze material. A side vent hole in the contact allows through flow of the electrolyte during plating, but also allows the molten braze material to flow out during the brazing operation. To prevent this latter flow, and also unnecessary flow up the sides of the conductor, stop-off materials were investigated.

Three forms of stop-offs were purchased from Wall Colmonoy (Canada) Ltd.,; Liquid Nicrobraz Green Stop-Off, Type II Liquid Nicrobraz Green Stop-Off, and Stix Nicrobraz Stop-Off. It was quickly recognized that the latter, which is in a solid crayon form, is difficult to apply uniformly, especially in small areas. Both of the other stop-offs were easy to apply, even to the inside circumference of the vent hole. The Type II Stop-Off has a water base([29]) and the other an organic base, but both were effective in stopping the flow of the braze material out of vent holes and up the sides of conductors beyond the ends of the contacts. After brazing, the stop-offs were green or grey solids, which could be removed even by a fingernail. Despite the ease of removal, brazed specimens which had stop-off on the surface were aged at 680°C for 1000 hours, to determine whether the stop-off had any detrimental effect to the conductor or contact. No effects could be detected by either visual or microscopic examination.

APPENDIX N

LEAK TESTING OF DUMMY CONNECTOR ASSEMBLIES

N-1 METHOD

Four brazed contact/insulator/dummy barrel assemblies were leak tested by measuring the flow rate of water from one side of the insulators to the other under a pressure differential of 15 psig.

N-2 RESULTS

Two specimens brazed using Au/Ni powder braze had leak rates of 9 and 15 cc/minute. The assembly brazed with wire preforms and the use of a superior alignment jig had a leak rate of 0.0022 cc/minute.

APPENDIX O

MEASUREMENT OF INSULATION RESISTANCE OF CERAMACAST 528 COMPOUND

O-1 OBJECT

Measurement of the insulation resistance at +1250°F of the given sample of Ceramacast 528 specimen (a) in comparison with room ambient readings.

O-2 TEST SAMPLES

Quantity Tested	Description
1	Ceramacast 528 of composition 10/2.3 and cured to 1250°F, contained in a metal shell with two pin contacts consisting of a small length of high temperature wire.

O-3 TEST SPECIFICATION

The insulation resistance both from pin to pin and pin to shell, shall exceed 500 megohms when measured at 1250°F and at room ambient temperatures.

O-4 APPARATUS

(i) G.R. megohm bridge, Type 544B; serial no. 2371; calibrated June 1976.

(ii) Lindberg furnace, Model No. 51892.

O-5 TEST PROCEDURE

A one-foot long conductor of CWC high temperature wire (Inconel clad silver) was brazed to the metal shell and two lengths (approximately 1 ft) of the same wire were inserted in each of the cavities for the pin contacts. The test sample was positioned inside the Lindberg furnace and the temperature was raised to +1300°F. Insulation resistance readings were taken in accordance with Method 302, Test Condition B of MIL-STD-202 at temperatures of +1300°F and +270°F, after cooling to room ambient temperature, and after 48 hours at room ambient temperature.

The insulation resistance was measured separately between all adjacent pins, and between the shell and all pins adjacent to the shell.

O-6 TEST RESULTS

Connections	Initial Room Amb.	+1300°F	+270°F	Room Amb.	After 48 hr at Room Amb.
Pin to pin	2.8×10^3	90.0	10^6	10^6	3×10^3
Pin to shell	1.3×10^3	18.5	10^5	4×10^5	9×10^2
Pin to shell	1.8×10^3	70.0	10^6	10^6	2.2×10^3

O-7 DISCUSSION

O-7.1 Bonding of Insulating Compound to Pin Contacts

This was found to be very poor, for the pin contacts had broken loose inside the cavities due to some light handling, i.e. clamping of crocodile clips for electrical connections, etc.

O-7.2 Insulation Resistance

From the test conducted, the insulation resistance was found to have dropped to a very low value at high temperatures (1300°F), whereas on cooling to room ambient temperature, it had reached an infinitely higher value, and then gradually dropped off with time to reach the initial values.

O-7.3 Moisture Absorption and Evaporation

Heating of the insulating compound had evaporated all of the initially absorbed moisture, resulting in an absolutely dry specimen and hence a phenomenally high insulation resistance, at room ambient temperature, immediately after the heat cycle. The test sample, being fairly porous, absorbed moisture from the surrounding air to reach saturation in approximately 48 hours, thereby resulting in a gradual decrease of the insulation resistance.

O-8 CONCLUSION

The test sample failed to meet the specifications.

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