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FOURTH QUARTERLY REPORT - VOLUME II
PHASE I

PHYSICAL PROPERTIES OF SOME ENGINEERING MATERIALS - UNPUBLISHED DATA FROM COMPANY SPONSORED PROGRAMS

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ABSTRACT

This is the fourth quarterly report in a program designed to collect and disseminate previously unpublished materials data obtained during recent years by Chance Vought Corporation. The information contained in this report consists primarily of physical property data on some engineering materials. Although physical property data is of primary concern in this report, there is some information presented on mechanical properties.

Materials covered in this report are ablation materials, electrical coatings, fiberglas, graphite, laminates, potting compounds, steel and zirconia.

This is Volume II of a three volume report, the remaining volumes are as follows:

Volume I - CVC Report No. 2-53420/2R373 "Mechanical Properties of Some Engineering Materials - Unpublished Data From Company Sponsored Programs"

Volume III - CVC Report No. 2-53420/2R375 "A Correlation of Properties for Various Formulations of Sintered Zirconia - Unpublished Data From Company Sponsored Programs"

CONTENTS

Physical Properties
Ablation Materials

SCREENING OF ABLATION MATERIALS IN A 40 KW PLASMA JET USING THE COLD WALL SHIELD MOVING SPECIMEN TECHNIQUE

INTRODUCTION

According to the literature, reinforced plastic materials exhibit ablation characteristics capable of withstanding extreme temperature for short periods of time.

OBJECT

To determine the ablation characteristics of a group of reinforced plastic materials.

CONCLUSIONS

As shown by the very high Q* value (ablation efficiency), the cross-ply laminated quartz phenolic material is superior to all other materials tested under the conditions of this evaluation.

PROCEDURE

All specimens were tested in the 40 KW plasma jet with an 80% nitrogen, 20% oxygen gas mixture. The exposure time was 120 seconds at an incident flux of 250 BTU/ft^2-second.

MATERIALS

1. R-120 phenolic resin - Coast Mfg. and Supply Co., Livermore, California.
2. 181 fiberglass F-120 phenolic pre-pregnated - Coast Mfg. and Supply Co., Livermore, California.
3. 581/12-00 fiber quartz cloth - Hess Goldsmith and Co., Inc., New York, N.Y.
4. C-100-29 refrasil cloth phenolic rod - H. I. Thompson Fiber Glass Co., 1733 Cordova Street, Los Angeles 7, California.
5. Fiberglass phenolic rod; formica FF-34 - Standard Parts and Equipment Corporation, 904 North Main, Ft. Worth, Texas.
6. Fiberglass silicon rod - H. I. Thompson Fiber Glass Co., Los Angeles, Cal.
7. Nylon cloth - Wellington Sears, Mdse. Mart, Dallas, Texas
8. Chopped fiberglass F-122 phenolic - Coast Mfg. and Supply Co., Los Angeles
9. MX-1801 ceramic fiber reinforced phenolic

10. MX-1344-67 quartz fiber reinforced phenolic

11. MX-1370 graphite and ceramic fiber reinforced phenolic

12. MX-2549 quartz fiber zirconium oxide and carbon reinforced phenolic

13. MX-2625 silica fabric reinforced phenolic

14. MX-2630 graphite cloth reinforced phenolic

15. 2630a graphite cloth and mineral filler reinforced phenolic - 9 through 15 - The Fiberite Corporation, Winona, Minnesota.

SPECIMENS

The specimens were molded into a 1/2 inch diameter rod configuration. The specimens fabricated at CVC were molded in a Carver laboratory press.

Molding conditions are shown in Table I.

RESULTS

The test results are presented in Table II.

TABLE 1

Sample Description	Specimen Number	Reinforcement Direction	Molding Temperature °F	
100% Phenolic Resin	1	None	300	
Glass Phenolic	2	Cross Ply	300	
Quartz Phenolic	3	Cross Ply	300	
Refrasil Phenolic	4	Cross Ply		VE
Glass Phenolic	5	Rolled vertical Ply		VE
Glass Silicon	6	Rolled vertical Ply		VE
Nylon Phenolic	7	Cross Ply	300	
Chopped glass fabric phenolic	8	Random	300	
Chopped quartz fabric phenolic	9	Random	300	
Quartz fiber phenolic	10	Random	300	
Chopped silica fabric phenolic	11	Random	300	
Ceramic fiber phenolic	12	Random	300	
Chopped graphite cloth phenolic	13	Random	300	
Chopped graphite cloth mineral filler phenolic	14	Random	300	
Graphite ceramic fiber phenolic	15	Random	300	
Quartz fiber zirconium oxide carbon phenolic	16	Random	300	

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TABLE 1

Reinforcement Direction	Molding Temperature °F	Molding Pressure PSI	Molding Time MIN.
None	300	1000	30
Cross Ply	300	1000	30
Cross Ply	300	1000	30
Cross Ply		VENDOR FABRICATED	
Rolled Vertical Ply		VENDOR FABRICATED	
Rolled Vertical Ply		VENDOR FABRICATED	
Cross Ply	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30
Random	300	1000	30

TABLE II

ABLATION RADIATION IN 40 KW PLASMA JET E

Sample Description	Specimen No.	Incident Flux BTU/Ft^2-Sec	Exposure Time Seconds	Initial Weight Gms.	Final Weight Gms.	Weig Loss Gms.
100% Phenolic Resin	1	250	120	6.8836	4.6489	2.23
Glass Phenolic * ▤	2			11.7450	8.2576	3.487
Quartz Phenolic ▤	3			12.6749	12.5124	0.16
Refrasil Phenolic ▤	4			10.4240	10.2386	0.185
Glass Phenolic ▥	5			10.3059	7.6094	2.701
Glass Silicone ▥	6			17.5219	13.4511	4.070
Nylon Phenolic ▤	7			7.7567	4.7777	2.979
<u>Random Reinforcement</u>						
Chopped Glass fabric phenolic	8			12.43975	7.57674	4.853
Chopped quartz fabric phenolic	9			14.44572	14.22560	0.220
Quartz fiber phenolic	10			13.92748	13.66622	0.261
Chopped silica fabric phenolic	11			15.73774	15.56697	0.1707
Ceramic fiber phenolic	12			14.94837	11.96858	2.9897
Chopped graphite cloth phenolic	13			12.89602	11.60514	1.290
Chopped graphite cloth mineral filler phenolic	14			17.16597	16.22300	0.9429
Graphite ceramic fiber phenolic	15			18.84325	17.98890	0.8543
Quartz fiber zirconium oxide carbon phenolic	16			16.02490	15.70444	0.3204

* Indicates ply direction

TABLE II

40 KW PLASMA JET EXPOSURES IN 80% NITROGEN 20% OXYGEN

Final Weight Gms.	Weight Loss Gms.	Weight Loss Gms/Sec	Initial Length In.	Final Loss In.	Length Loss In.	Q* BTU/Lb.	Optical Temp. °F
4.6489	2.2347	0.01862	1 3/4	1 1/4	1/2	8,303	3840
8.2576	3.4874	0.02906	1 15/16	1 3/8	1/2	5,320	3340
12.5124	0.1625	0.00135	2 3/8	2 3/8	0	114,520	3160
10.2386	0.1854	0.00155	1 15/16	1 15/16	0	99,740	3180
7.6094	2.7010	0.0225	2 3/4	2 1/16	11/16	6,871	3340
13.4511	4.0708	0.03392	2 7/8	2 1/8	3/4	4,557	3000
4.7777	2.9790	0.02399	2 1/8	1 5/16	1/2	6,444	3260
7.57674	4.85301	0.04044	2	1 3/16	13/16	38,229	3500
14.22560	0.22012	0.00183	2 11/16	2 11/16	0	84,481	3080
13.66622	0.26126	0.00218	2 1/2	2 1/2	0	70,917	3050
15.56697	0.17077	0.00142	2 3/4	2 3/4	0	88,732	3050
1.96858	2.98979	0.02491	2 3/4	2 1/4	1/2	62,063	3360
1.60514	1.29088	0.01076	2 15/16	2 3/4	3/16	14,368	3460
6.22300	0.94297	0.00786	2 13/16	2 3/4	1/16	19,669	3100
7.98890	0.85435	0.00712	3 1/8	3 1/16	1/16	21,713	3060
5.70444	0.32046	0.00267	2 13/16	3 13/16	0	57,903	3140

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EVALUATION OF PROTECTIVE ELECTRICAL COATINGS

INTRODUCTION

Electronic circuits function more reliably when protected from moisture, corrosion, fungus, and dirt. This protection may be obtained by coating electronic circuits with materials possessing suitable electrical and physical properties. Various protective materials have been screened at CVC for use on terminal and printed circuit boards. Of the materials screened, PR 905 and Eccocoat VE appear to satisfy the majority of the desired properties. In addition, a new product, PS 798 (previously XWC45-41), appeared very promising in preliminary tests and is also evaluated herein.

OBJECT

To determine the best material for use in protecting printed circuit and terminal boards.

CONCLUSION

All the materials tested appear to be satisfactory for this purpose. Each material has certain advantages and disadvantages over the other two. From a production standpoint, however, PS 798 and Eccocoat VE were favored because of their longer working times.

MATERIALS

PR 905 protective coating material, manufactured by Products Research Co., 3126 Los Feliz Blvd., Los Angeles 39, California.

Eccocoat VE coating material, manufactured by Emerson & Cuming Corporation, 869 Washington Street, Canton, Mass.

PS 798 coating material, manufactured by Coast Proseal Co., Los Angeles, California.

PROCEDURE

I. Physical

a. Specific Gravity: Specific gravity was determined by water displacement method by the following formula:

$$\text{Specific Gravity} = \frac{(\text{Weight in Air})}{(\text{Weight in Air}) - (\text{Weight in Water})}$$

b. Water Absorption: Three specimens of each material were prepared as follows:

A 10 mil film with an area of 2 sq.in. was cast on a 2 3/8 in. x 2 in. x 0.064 in. alodined 7075-T6 aluminum panel. The specimen panels were weighed to the nearest 0.1 milligram before and after application of the coating material. The specimens were totally submerged in distilled water at room temperature for 24 hours. After the soak period the specimens were removed from the water, blotted, and reweighed.

c. Adhesion: Three specimens of each material were prepared per paragraph 6.9, reference (a). Bondline thickness was held to approximately 10 mils. Tail material was 6-ounces Dynel. Panel material was 3/32" MIL-P-18177 laminated board. Each specimen was tested in 90° peel on a Scott tester jaw separation of 2" per minute.

d. Low Temperature Flexibility: Three specimens of each material were prepared by applying a 10-mil film on 1" x 6" x 0.064" alodined 7075-T6 aluminum panels. The cured specimens were soaked at -65°F for 2 hours. While still at -65°F, the specimen ends were deflected 1/8" from the center plane.

e. Cycling: Three specimens of each material were prepared by applying a 10-mil film on the circuit side of printed circuit boards (MIL-P-18177). After curing, each specimen was given five test cycles. Each cycle consisted of the following:

1. Humidity - One hour in humidity chambers at 92% relative humidity and 160°F temperature.

2. High temperature - one hour at 300°F

3. Low temperature - one hour at -65°F

II. Electricals

a. Insulation Resistance (500 VDC): Nine specimens of each material were prepared as follows:

Two bare #20 AWG copper wires were dipped to a depth of one inch in the compound being tested. Wire spacing was maintained at 10 mils and the coating thickness on the outside of the copper wires was 10 mils. Insulation resistance was determined on the cured specimens. Three specimens of each material were subjected to each of the following environmental conditions:

1. Humidity: 92% relative humidity at 160°F. Specimens were then removed from chamber to measure insulation resistance.

2. High Temperature (300°F): Specimens were tested while in the oven at 300°F.

3. Low Temperature (-65°F): Specimens were tested in cold box at -65°F.

Insulation resistance of each specimen was measured at 2-hour intervals for a total of 8 hours at each condition.

c. Volume and Surface Resistivity: Volume and surface resistivity was determined on approximately 10-mil thick specimens cured on 1/4" aluminum plates per MIL-S-8516.

d. Dielectric Strength: Two specimens of each material 10 and 20 mils thick, respectively, were prepared on a non-adherent surface. Dielectric strength was determined.

RESULTS

Results of the test appear in Table I.

TABLE I

EVALUATION OF PROTECTIVE ELECTRICAL COATI

MATERIAL	DIELECTRIC STRENGTH		RESISTIVITY		SAMPLE THICKNESS	D.C. INSULATION RESISTANCE	
	VOLT/MIL	SAMPLE THICKNESS (in.)	Volume (OHM-CM)	Surface (OHM)		As-Received	After 8 h
Eccocoat VE	250	0.020	0.686 x 10^{12}	8.44 x 10^{12}	0.013 x 10^{12}	50,000	
Eccocoat VE	810	0.008	0.785 x 10^{12}	7.85 x 10^{12}	0.015	50,000	
Eccocoat VE	810	0.008	-	-	-	50,000	
Eccocoat VE	935	0.008	-	-	-	50,000	
PR-905	870 870	0.023	11.62 x 10^{12}	12.55 x 10^{12}	0.014		
PR-905	1075	0.013	23.1 x 10^{12}	12.55 x 10^{12}	0.018		
PS-798	905	0.021	10.1 x 10^{12}	8.44 x 10^{12}	0.019	50,000	2
PS-798	1250	0.010	11.32 x 10^{12}	7.59 x 10^{12}	0.018	50,000	2

PHYSICAL PROPERTIES

MATERIAL	SPECIFIC GRAVITY	Water Absorption g/sq. in.	ADHESION lb/in.	-65°F Flex.	CYCLING
Eccocoat VE	1.0387	0.0116	10 AF	Passed	Darkened Hardened
PR-905	1.0702	0.0058	0 AF	Passed	Slight darkening; hardened
PS-798	1.036	0.0024	7 AF	Passed	Darkened Hardened

MATERIAL	CO
Eccocoat VE	Cl am
PR-905	Cl an
PS-798	Cl an

(1) Coast Proseal claims to have eliminated the air entrapment problem. This fact was evidenced by a later sample.

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TABLE I

CTRICAL COATINGS - ELECTRICAL PROPERTIES

N RESISTANCE (MEG-OHM)	60 cycle		1 kilocycle		1 megacycle		SAMPLE THICKNESS
After 8 hours at 300°F	Dielectric Constant	Power Factor	Dielectric Constant	Power Factor	Dielectric Constant	Power Factor	
1.5	6.24	0.277	2.73	0.1085	2.95	0.065	0.013
1.5	6.63	0.248	4.84	0.0980	3.37	0.061	0.015
1.5	-	-	-	-	-	-	-
1.5	-	-	-	-	-	-	-
3	4.73	0.054	4.43	0.0153	3.79	0.049	0.014
3	3.72	0.047	3.62	0.0156	3.19	0.056	0.018
20	5.5[illegible]	0.046	5.57	0.0480	3.71	0.1135	0.019
20	5.95	0.096	5.78	0.0530	4.31	0.065	0.018

OBSERVATIONS

ERIAL	COLOR	RIGIDITY	CURE	WORK LIFE	MIX A:B BY WEIGHT	HANDLING CHARACTER-ISTICS	HANDLING DISADVANTAGES
coat VE	Clear amber	Slightly flexible	8 hours at 75°F or 1 hour at 150°F	3-4 hours	1:1	Good	No apparent dis-advantages
-905	Clear amber	Slightly flexible	48 hours at 75°F or 1 hour at 180°F	25-30 min.	12:11	Good	Does not wet surface easily
-798	Clear amber	Slightly flexible	24 hours at room tempera-ture	2 hours	3:1	Good	Hard to remove air in mix(1)

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THERMAL CONDUCTIVITY OF HONEYCOMB SANDWICH PANELS

INTRODUCTION

In order to retard heat buildup from aerodynamic heating, a protective low conductivity structure is beneficial. To establish criteria for design, the thermal conductance of proposed materials must be determined. The purpose of this test is to determine the thermal conductance of fiberglass honeycomb structure.

OBJECT

To determine the thermal conductance of two fiberglass honeycomb specimens with different face thicknesses and the same overall thickness.

CONCLUSION

The thermal conductance increased with increased face thickness (decreased core thickness). Apparently, most of the insulation effect of the honeycomb sandwich is due to the core; hence, a thinner core has a lower insulation value (higher conductance) for a given overall thickness.

PROCEDURE

I. Fiberglass Reinforced Phenolic Honeycomb Fabrication

A. Skin Fabrication

The following laminates were laid up parallel to the warp and cured at 300°F.

(1) One 6-ply F-120-11 9" x 17"
(2) One 3-ply F-120-11 9" x 17"
(3) Two 1-ply F-120-11 9" x 17"

B. Core Preparation

Two 9" x 17" 5.5 pound density HRP fiberglass reinforced phenolic cores were milled to the following thicknesses respectively - 0.116" and 0.146".

C. Sandwich Fabrication

Panels were fabricated by bonding the skin laminates to the core sections using Bloomingdale Rubber Company H2424 Adhesive.

One surface of each core section was the one ply skin. The six ply laminate was bonded to the 0.116 core and the three ply laminate was bonded to the 0.146 core, thus the overall thickness of both specimens was approximately the same.

II. Thermal Conductivity Measurements

A. The thermal conductance of all specimens was measured on a guarded hot plate apparatus conforming to ASTMC-177-45.

B. The guarded hot plate unit consists of a central metered heater section four inches square. This section is heated by a chromel "A" resistance wire of approximately 19-ohms resistance, maximum wattage is approximately 720 watts at 120 volts. A 1,000 watt guard heater section surrounds the central heater section. A 1/8" air gap separates the guard and central heater face sections. A precise temperature balance between the metered and guard sections is achieved by a Brown drive amplifier actuated by a differential thermocouple. The drive amplifier regulates an autotransformer supplying power to the guard ring heater. This maintains the same temperature in both the metered guard sections.

C. Identical specimens of the honeycomb panels were placed on each side of the hot plate with the thicker face of each panel toward the hot plate. The outer faces of each specimen were cooled by 8" x 8" square water cooled copper cold plates. Iron-constantan thermocouples were attached to each specimen face with catalysed epoxy resin or ceramic cement (Rusco 749).

Intimate thermal contact of the specimen face and heater surface was achieved by insertion of soft asbestos paper layers. The cold surface interlayer was also asbestos in the first test of this report but was changed to aluminum foil when it became apparent that inadequate cooling resulted from use of asbestos inserts at the cold face.

Sufficient power was applied to the central heater to maintain the desired temperature differential between hot and cold faces. The system was allowed to come, to thermal equilibrium, and measurements were made on the hot and cold face temperatures as well as the power input. Thermal conductivity "K" was calculated as follows:

$$K = \frac{ql}{A \quad T}$$

Where: -

K = The thermal conductivity in

$$\frac{\text{BTU - In.}}{\text{ft.}^2 \text{ - hr. - }^{o}\text{F}}$$

q = power input to central heater in BTU/hr.

l = thickness of specimen in inches

A = area of the central heater (both hot faces)

T = the temperature differential between the hot and cold face of the specimen (average)

RESULTS

Test data are recorded in Table I and Graphs I and II.

TABLE I

RESULTS OF THERMAL CONDUCTIVITY TEST ON FIBERGLASS HONEYCOMB PANELS

Test I 0.030" Face Panel 0.2023" Total Panel Thickness						Test II 0.060" Face Panel 0.1979" Total Panel Thickness					
Hot Face Temp. °F	Cold Face Temp. °F	T °F	Mean Temp. °F	Power Watts	K BTU - In. / ft^2 - hr - °F	Hot Face Temp. °F	Cold Face Temp. °F	T °F	Mean Temp. °F	Power Watts	K BTU - In. / ft^2 - hr - °F
147	100	47	124	8.84	0.585	127	100	27	114	8.84	0.996
193	98	94	146	18.24	0.597	150	100	50	125	16.92	1.03
297	112	185	205	36.70	0.617	211	100	111	156	39.76	1.09
385	130	255	258	58.14	0.709	310	105	205	208	73.34	1.06
517	170	347	344	92.45	0.828	395	109	286	252	107	1.14
604	197	407	400	115.68	0.886	503	111	392	307	149	1.16
						564	123	441	344	186	1.28

FIGURE 1

THERMAL CONDUCTIVITY OF HONEYCOMB SANDWICH PANELS

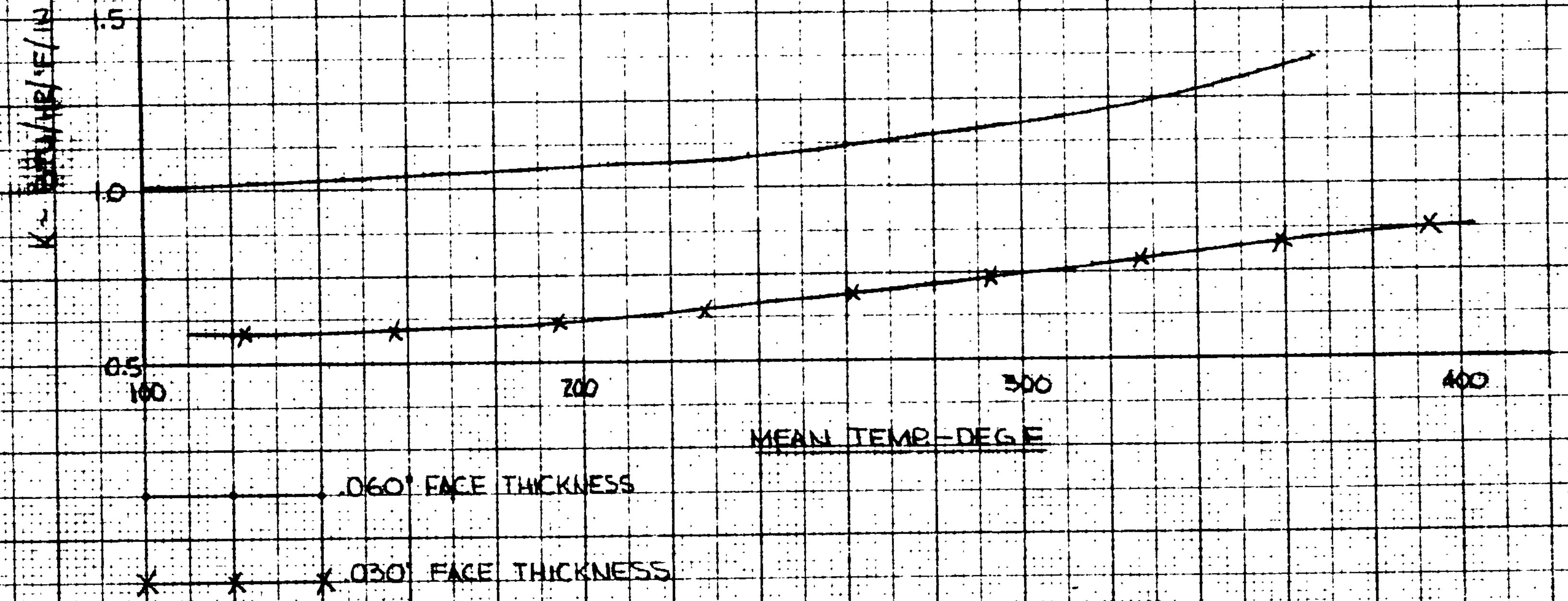
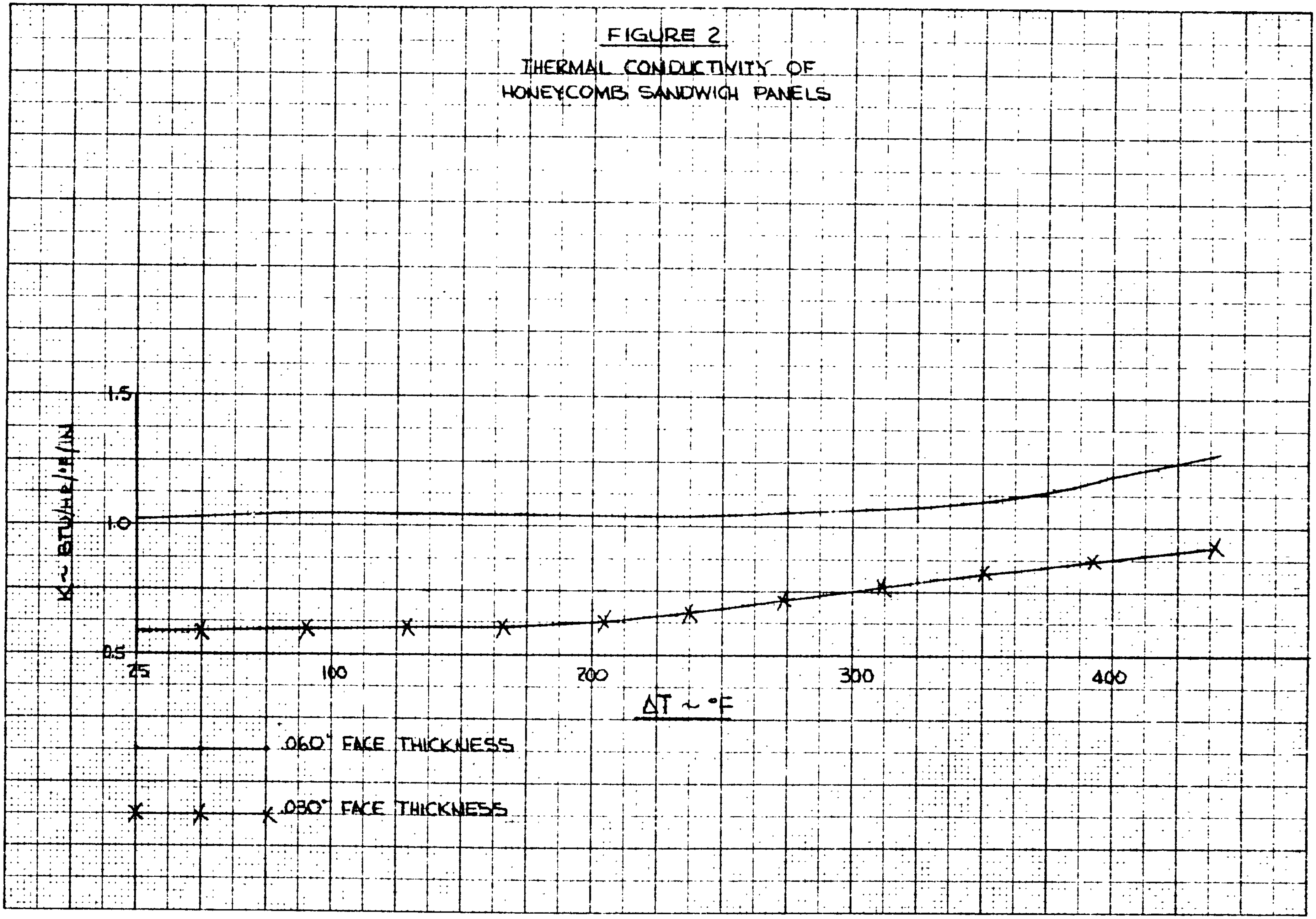


FIGURE 2

THERMAL CONDUCTIVITY OF HONEYCOMB SANDWICH PANELS



THERMAL SHOCK TEST OF COATED GRAPHITE

INTRODUCTION

The purpose of this evaluation is to determine the thermal shock resistance of siliconized coated ATJ graphite.

OBJECT

To determine if siliconized coated ATJ graphite can resist a thermal shock rate of 150 degrees per second when heated from room temperature to 3000°F and also resist rapid cooling.

CONCLUSION

Siliconized coated ATJ graphite can resist a thermal shock rate of 150 degree per second when heated from room temperature to 3000°F and also a rapid cooling rate of 43.5 degrees per second from 3000 to 2000°F.

PROCEDURE

All thermal shock testing was performed in the 40 KW Plasma flame. Temperatures were measured by radiation pyrometer and times were determined by stop watch. The specimens were rods of ATJ graphite 1/2 inches in diameter and 1 1/2 inch long which had been siliconize coated at National Carbon by the pack diffusion process.

Specimens Number 5 and 7 were preoxidized for 5 minutes at 3000°F in the plasma flame using a gas mixture of 80% nitrogen and 20% oxygen.

The specimens were examined and weighed before testing for thermal shock. The pre-oxidation treatment caused blistering of the surface contamination. The specimens were exposed to a thermal shock rate of 150 degrees per second from room temperature to 3000°F and cooled slowly. Examination of the specimens revealed no detrimental effects to the coating.

Specimens Number 8 and 12 were heated slowly to 3000°F in the plasma flame. The flame was extinguished and a cooling rate of 43.5 degrees per second from 3000 to 2000°F was determined. Inspection of the specimens after testing revealed no detrimental effects to the coating, however, some blistering of the surface contamination was observed. The specimens were then heated to 2000°F for 1 hour in the Cooley furnace as an oxidation proof test. The small weight loss is negligible and can be contributed to the oxidation of surface contamination on the specimens.

RESULTS

The test conditions and weight changes are found in Table I.

TABLE I

THERMAL SHOCK TESTING OF SILIZONIZED COATED ATJ GRAPHITE

SPECIMEN NUMBER	TEST CONDITION	BEFORE	WEIGHT GRAMS		
			AFTER	CHANGE	% CHANGE
5	Pre-oxidized in plasma flame for 5 minutes at 3000°F	9.29334	9.13716	.15518	-1.68
7	Pre-oxidized in plasma flame for 5 minutes at 3000°F	9.29615	9.26221	.03394	- .37
5	Heated in plasma flame at 150° per second from room temperature to 3000°F	9.13716	9.12357	.01359	- .15
7	Heated in plasma flame at 150° per second from room temperature to 3000°F	9.26221	9.25384	.00837	- .09
8	Heated in plasma flame to 3000°F and cooled at 43.5° per second from 3000° to 2000°F	9.28763	9.28562	.00174	- .02
12	Heated in plasma flame to 3000°F and cooled at 43.5° per second from 3000° to 2000°F	9.28897	9.29377	.00480	+ .05
8	Oxidation proof tested at 2000°F for 1 hour in Cooley Furnace	9.28562	9.27159	.01403	- .15
12	Oxidation proof tested at 2000°F for 1 hour in Cooley Furnace	9.29377	9.26606	.02771	- .30

OXIDATION EVALUATION OF COATED GRAPHITE

INTRODUCTION

The purpose of this evaluation is to determine the high temperature capabilities of siliconized coated ATJ graphite in an oxidizing atmosphere.

Fifteen 1-inch cube specimens of ATJ graphite siliconized by the National Carbon pack diffusion process and four 1-inch cube specimens of ATJ graphite siliconized by the National Carbon pack diffusion process but with a light green dusty surface were submitted to the Structures Materials Laboratory for this investigation.

OBJECT

To determine the oxidation resistance of siliconized coated graphite at elevated temperatures.

CONCLUSION

The siliconized ATJ graphite coated by the National Carbon pack diffusion process which formed a beta silicon carbide coating did not oxidize when exposed to 3300°F for 1 hour in an air atmosphere.

The siliconized ATJ graphite coated by the National Carbon pack diffusion process which formed a coating with beta silicon carbide as the major constituent and alpha silicon carbide as the minor constituent showed some oxidation when exposed to 3300°F for 1 hour in an air atmosphere; however, the oxidation did not penetrate the siliconized coating.

PROCEDURE

The specimens were weighed, photographed and X-rayed before and after each test.

A. Specimen No. 1 was tested in the plasma flame with a gas mixture of 80% nitrogen and 20% oxygen at 3000°F for 10 minutes. The flow of free surface contamination was observed at approximately 2200°F and continued throughout the duration of the test. The graphite specimen did not oxidize, however the slight weight gain was contributed to the oxidation of the free contamination on the surface. No detrimental effects to the specimen were shown by X-ray analysis.

B. Specimen No. 4 was tested in the plasma flame with a gas mixture of 80% nitrogen and 20% oxygen at 3300°F for 10 minutes. The flow of the surface contamination was again observed at 2200°F and continued throughout the duration of the test. It should be noted that bubbling of this surface contamination was observed on the sides of the specimen during testing;when the specimen was cooled, the bubbles solidified leaving blisters on the surface. The grapite specimen did not oxidize, however the slight weight gain was contributed to the oxidation of the contamination of the surface. No detrimental effects to the specimen were shown by X-ray analysis.

C. Specimen No. 5 and No. 17 were tested in the plasma flame with a gas mixture of 80% nitrogen and 20% oxygen at 3300°F for 30 minutes. Specimen No. 5 had a coating of beta major and alpha minor silicon carbide. The flow of surface contamination was again observed at 2200°F. The heated face of the specimen contained small oxidation pits which had not penetrated the coating. A slight weight gain was measured which was due to the oxidation of the surface contamination. The surface pits were evident by X-ray analysis.

Specimen No. 17 had a coating of beta silicon carbide. No oxidation occurred during testing. The slight weight loss is due to the volitalization of the surface contamination. X-ray analysis showed no detrimental effects to the specimens.

Specimen No. 17 was superior to Specimen No. 5.

D. Specimen No. 9 and No. 16 were tested in the graphite resistance furnace for 1 hour with an air atmosphere. Specimen No. 9 which had a beta major and alpha minor silicon carbide coating exhibited severe blistering on the surface. The blisters were removed from one face of the specimen revealing pit oxidation of the specimen surface. X-ray analysis revealed the presence of the pit oxidation under the surface blisters.

Specimen No. 16 had a beta silicon carbide coating. This graphite specimen did not oxidize, however a slight weight increase was found which was due to the oxidation of the contamination on the surface. X-ray analysis showed no detrimental effects to the specimen.

Specimen No. 16 with the beta silicon carbide coating was superior to Specimen No. 9 which had the beta major alpha minor silicon carbide coating.

E. Specimens No. 10, 11, 19, and 18 were tested in the zirconia pot furnace for 1 hour with an air atmosphere. Specimens No. 10 and No. 11 which had the beta major alpha minor silicon carbide decoating exhibited severe blistering on the surface. The surface contamination was removed revealing pit oxidation on the specimen surface. It should be noted that the pits did not penetrate completely through the siliconized coating. Specimen No. 10 had a weight loss of 3.5% and Specimen No. 11 had a weight loss of 3.43%. This weight loss was due to oxidation. X-ray analysis revealed the presence of pit oxidation.

Specimen No. 18 and No. 19 had the beta silicon carbide coating. These specimens did not oxidize although a small weight loss was measured. This weight loss is apparently due to the volitization of the surface contamination. The surface of the specimen retained a small amount of SiO_2. X-ray analysis showed no detrimental effects on the specimens.

RESULTS

Table I shows the test method and per cent weight change of the specimens.

TABLE I

RESULTS OF OXIDATION EVALUATION OF SILICONIZED GRAPHITE

Specimen Number	Test Conditions			Weight Grams			% Change
	Temp. °F	Time Min.	Method	Before	After	Change	
1	3000	10	Plasma flame	29.59346	29.60586	+ .01240	+ .042
2			Not tested	30.14412			
3			Not tested	29.92438			
4	3300	10	Plasma flame	29.84831	29.88254	+ .03423	+ .12
5	3300	30	Plasma flame	29.90901	29.98339	+ .07438	+ .25
6			Not tested	29.69796			
7			Not tested	30.04678			
8			Not tested	29.54447			
9	3000	60	Graphite furnace	30.09382	29.69172	- .40210	-1.34
10	3300	60	Zirc. Pot Furn.	29.51908	28.4832	-1.03276	-3.50
11	3300	60	Zirc. Pot Furn.	29.78738	28.77034	-1.01704	-3.43
12			Not tested	30.33786			
13			Not tested	29.73793			
14			Not tested	30.13563			
15			Not tested	29.77567			
16	3000	60	Graphite furnace	31.90355	31.98754	+ .08390	+ .26
17	3300	30	Plasma flame	30.50052	30.42025	- .08027	- .26
18	3300	60	Zirc. Pot Furn.	30.41420	30.29027	- .12392	- .41
19	3300	60	Zirc. Pot Furn.	30.38185	30.32111	- .06074	- .20

THERMAL CYCLING TEST OF SILICONIZED GRAPHITE

INTRODUCTION

This report presents the results of thermal cycling tests performed on specimens of siliconized graphite.

OBJECT

The purpose of this test is to verify the thermal integrity of siliconized graphite under temperature and constant load conditions.

CONCLUSIONS

Tests were performed on three specimens. One of these completed four cycles without failure and the remaining two failed late in the fourth cycle.

MATERIAL AND SPECIMENS

Three siliconized graphite specimens for thermal cycling tests were made as shown in Figure 1.

PROCEDURE

Three specimens were tested according to the 3000°F temperature-time data (Table I). The center cross-section of the specimen was measured. The specimen was the installed in the test fixture so as to maintain a constant compression stress of 845 psi. Test cycle was as follows: (1) apply computed load, (2) apply heat according to temperature-time data (0 to 60 minutes) (3) allow specimens to cool to room temperature and remove load, (4) apply four cycles per specimen, (5) weigh test specimen before and after tests.

RESULTS

Table II presents the results of the thermal cycling tests.

TABLE I

TEST SCHEDULE

TIME (Min)	TEMPERATURE (°F)
0 - 10	1350 - 2420
10 - 20	2420 - 2770
20 - 30	2770 - 2980
30 - 35	2980 - 3000
35 - 40	3000 - 2980
40 - 50	2980 - 2630
50 - 60	2630 - 1490

TABLE II

TEST RESULTS

SPEC NO	DIA	AREA	WEIGHT (GRAMS) BEFORE	AFTER	REMARKS -
1	.510	.2043	53.1322	50.1348	Completed 4 cycles
2	.507	.2019	53.2018		Failed after 55 min. of 4th cycle
3	.509	.2035	53.4213		Failed after 40 min. of 4th cycle

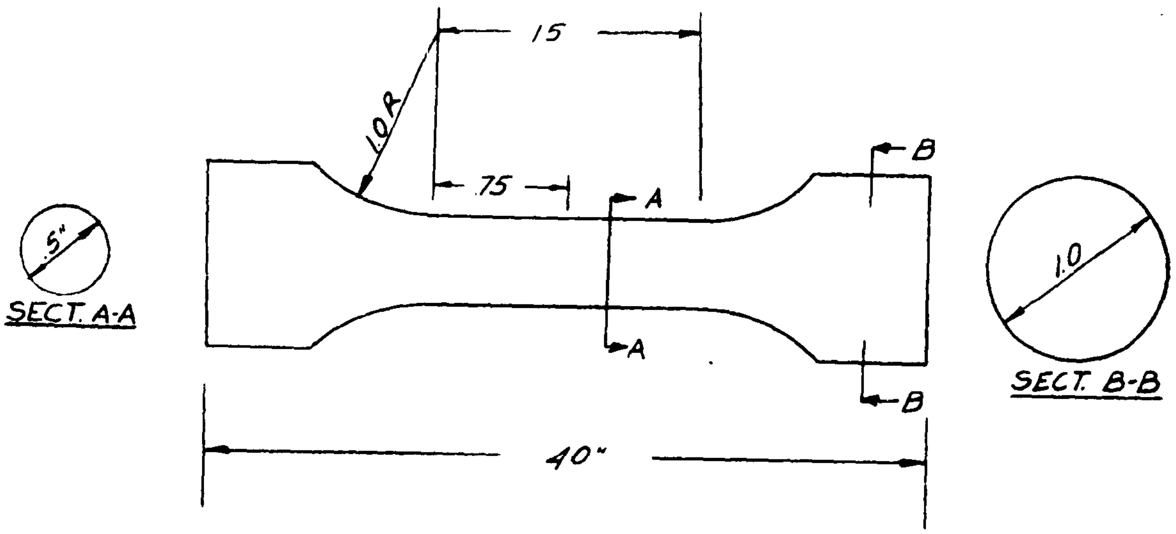


FIGURE 1

TYPICAL GRAPHITE SPECIMEN

ABLATION STUDIES OF QUARTZ REINFORCED PHENOLIC IN A 40 KW PLASMA JET AT VARIED TEST CONDITIONS

INTRODUCTION

Screening tests of ablation materials indicated that laminated quartz phenolic specimens were superior to all other specimens when tested under the conditions stated. In order to more clearly evaluate the above material, additional testing under controlled conditions is required.

OBJECT

To determine the ablation characteristics of cross ply laminated fiber quartz phenolic material under controlled conditions.

CONCLUSIONS

In all cases, fusion occurred at the surface of the fiber quartz reinforced specimens. However, at the 500 BTU/ft^2-second heat flux level, ablative flow of the quartz was observed. This is shown by the lower Q* value at the 500 BTU/ft^2-second level as compared to the values obtained at the other flux levels during the 120-second exposure.

It is also shown that at the 250 BTU/ft^2-second level, ablation times of 20 minutes or more can be expected.

MATERIALS

1. R-120 phenolic resin, Coast Manufacturing and Supply Company, Livermore, California.
2. 581/12-00 fiber quartz cloth, Hess Goldsmith and Co., Inc., 1400 Broadway, New York 18, N. Y.
3. 181 fiberglass F-120 phenolic pre-pregnated, Coast Manufacturing and Supply Company, Livermore, California.

SAMPLE PREPARATION

1. Fiber quartz phenolic rods

   The fiber quartz cloth was impregnated with R-120 phenolic resin by the spray method in the CVC plastics shop. The resin was then "B" staged at 180°F for 3 hours. One-half inch diameter disks were then cut from the pre-impregnated material, placed in the 1/2 inch diameter mold and cured. The curing conditions used were 1,000 psi molding pressure, 300°F molding temperature, for 30 minutes.

2. Fiberglass phenolic rods

The fiber glass phenolic material was purchased in the pre-impregnated "B" staged condition. One-half inch diameter disks were cut from the pre-impregnated material, placed in the 1/2 inch diameter mold and cured under the same conditions used in molding the fiber quartz phenolic specimens.

TEST CONDITIONS

Three groups of specimens were tested in this evaluation. For the first group, a heat flux of 250 BTU/ft^2-second was held constant while the exposure time was varied. For the second group, an exposure time of 120 seconds was held constant while the heat flux was varied. In the third group, one specimen was held at a 45° angle to the flame and exposed to a heat flux of 250 BTU/ft^2-second for 120 seconds. A second specimen was placed in an insulation block of zirconia and exposed to a heat flux of 250 BTU/ft^2-second for 120 seconds.

Testing was performed in the 40 KW plasma jet with a gas mixture of 80 per cent nitrogen and 20 per cent oxygen. Except for the insulated specimen, all specimens were tested by the cold wall shield moving specimen technique. All calculations were based on the calibration of the Milpar calorimeter. Optical temperature was recorded for all specimens.

RESULTS

Results of this investigation are shown in Table I.

TABLE I

ABLATION RADIATION IN A 40 KW PLASMA
EXPOSURES IN 80% NITROGEN 20% OXYGE

Sample Description	Specimen No.	Incident flux BTU/ft^2 sec	Exposure Time	Initial Weight Gms.	Final Weight Gms.	Weight Loss Gms.	Weig Loss gms/se
Glass Phenolic	1	250	5 sec	16.3649	16.15024	0.2147	0.042
Glass Phenolic	2	250	50 sec	16.3346	14.50055	1.8340	0.036
Quartz Phenolic	3	250	5 sec	14.4835	14.33794	0.1456	0.029
Quartz Phenolic	4	250	50 sec	15.2683	14.98036	0.2879	0.005
Quartz Phenolic	5	250	20 min	12.48505	12.21800	0.2671	0.000
Quartz Phenolic	6	65	120 sec	11.27018	11.2161	0.0541	0.000
Quartz Phenolic	7	100	120 sec	14.29660	14.15391	0.1427	0.001
Quartz Phenolic	8	300	120 sec	14.92740	14.8331	0.0937	0.000
Quartz Phenolic	9	500	120 sec	12.88022	11.96689	0.9133	0.007
Quartz 45° Angle	10	250	120 sec	11.96689	11.87494	0.0920	0.000
Quartz Insulated	11	250	120 sec	9.74190	9.16159	0.5803	0.004

1

ABLATION RADIATION IN A 40 KW PLASMA JET
EXPOSURES IN 80% NITROGEN 20% OXYGEN

Initial Weight Gms.	Final Weight Gms.	Weight Loss Gms.	Weight Loss gms/sec.	Initial Length In.	Final Length In.	Length Loss In.	Q* BTU/lb.	Optical temp °F
16.3649	16.15024	0.2147	0.0429	2 13/16	2 13/16	0	3,400	3200
16.3346	14.50055	1.8340	0.0367	2 5/8	2 3/8	1/4	4,250	3200
14.4835	14.33794	0.1456	0.0291	2 1/2	2 1/2	0	5,666	3200
15.2683	14.98036	0.2879	0.00576	2 3/4	2 3/4	0	28,333	3200
12.48505	12.21800	0.2671	0.000223	2 3/8	2 3/8	0	680,000	3200
11.27018	11.2161	0.0541	0.000451	2 13/16	2 13/16	0	106,080	2200
14.29660	14.15391	0.1427	0.00119	2 1/2	2 1/2	0	54,400	2450
14.92740	14.8331	0.0937	0.000781	2 3/4	2 3/4	0	244,800	3300
12.88022	11.96689	0.9133	0.00761	2 3/8	2 3/16	3/16	40,800	3500
11.96689	11.87494	0.0920	0.000767	-	-	-	204,000	3160
9.74190	9.16159	0.5803	0.00484	1 7/8	-	-	31,460	3200

ABLATION RADIATION IN A 40 KW PLASMA JET
EXPOSURES IN 80% NITROGEN 20% OXYGEN

Initial Weight Gms.	Final Weight Gms.	Weight Loss Gms.	Weight Loss gms/sec.	Initial Length In.	Final Length In.	Length Loss In.	Q* BTU/lb.	Optical temp °F
16.3649	16.15024	0.2147	0.0429	2 13/16	2 13/16	0	3,400	3200
16.3346	14.50055	1.8340	0.0367	2 5/8	2 3/8	1/4	4,250	3200
14.4835	14.33794	0.1456	0.0291	2 1/2	2 1/2	0	5,666	3200
15.2683	14.98036	0.2879	0.00576	2 3/4	2 3/4	0	28,333	3200
12.48505	12.21800	0.2671	0.000223	2 3/8	2 3/8	0	680,000	3200
11.27018	11.2161	0.0541	0.000451	2 13/16	2 13/16	0	106,080	2200
14.29660	14.15391	0.1427	0.00119	2 1/2	2 1/2	0	54,400	2450
14.92740	14.8331	0.0937	0.000781	2 3/4	2 3/4	0	244,800	3300
12.88022	11.96689	0.9133	0.00761	2 3/8	2 3/16	3/16	40,800	3500
11.96689	11.87494	0.0920	0.000767	-	-	-	204,000	3160
9.74190	9.16159	0.5803	0.00484	1 7/8	-	-	31,460	3200

2

EVALUATION OF RIGID ELECTRICAL EMBEDMENT COMPOUNDS FOR USE FROM -55°C TO 125°C

INTRODUCTION

A rigid electronic embedment compound is required for use in computer modules. These modules are required to function at temperatures ranging from -55°C to 125°C. Economical fabrication of the modules adds the requirements that the embedment material have a low viscosity and be workable for several hours after mixing. The purpose of this test is to evaluate rigid embedment materials for module embedment applications.

OBJECT

To determine if the following materials possess the necessary physical, electrical, and handling properties for module embedment applications:

Shell Epon 828 plus curing agent Z

Shell Epon 828 plus curing agent D. (Note: When used in proportions specified herein, Epon 828 plus curing agent D meets requirements of MIL-I-16923)

Sonite HH-1200

Scotchcast CRP 236

CONCLUSIONS

Shell Epon 828 has excellent physical, electrical, and handling properties when catalyzed with Shell curing agent Z. Epon 828 resin catalyzed with curing agent D yielded equivalent electrical properties but slightly poorer physical properties; i.e., resistance to thermal shock than the curing agent Z, Epon 828 mix. In addition, the worklife of the curing agent D, Epon 828 mix had too little worklife for continuous or automated operation. Testing of Sonite HH-1200 and Scotchcast CRP 236 was discontinued when it was determined that their handling characteristics were totally unsuitable for the automated operation proposed for manufacturing the modules.

MATERIALS

Shell Epon 828 epoxide resin,

Shell Curing Agent Z, and

Shell Curing Agent D - all manufactured by Shell Chemical Corp., Houston, Texas.

Sonite HH-1200 A/B epoxide resin and curing agent manufactured by Smooth-On, Jersey City, N. J.

Scotchcast CRP 236 epoxide resin manufactured by Minnesota Mining and Manufacturing Co., St. Paul, Minn.

PROCEDURE

I. Preparation and Curing of Materials

A. Epon 828 - curing agent Z

1. 20 parts of curing agent Z were blended with 100 parts of Epon 828 resin.
2. The mixture was evacuated at 27-29 inches Hg. for 30 minutes.
3. Castings in excess of 1/8 inch minimum diameter were cured 1 hour at 180°F. Castings with less than 1/8 inch minimum dimension were cured 2 hours at 180°F.

B. Epon 828 - Curing Agent D

1. 10.5 parts of curing agent D were blended with 100 parts of Epon 828 resin.
2. All Epon 828, curing agent D specimens were cured 1 hour at 200°F.

II. Physical Properties

A. Specific Gravity

1. Cylindrical specimens 1 inch in diameter and 0.5 inch thick were cast in polyethylene molds (Cannon cap #498).
2. Specific gravity of the cured specimens was determined by the water displacement method.

B. Hardness

1. Coleman Smith hardness was determined.
2. One specimen of each material was tested at 125°C and at 25°C,

C. Thermal Shock

Resistance to thermal shock was determined.

D. Moisture Absorption

1. Weighed specimens, similar to those described in II.A., were conditioned in the humidity chamber operated per MIL-E-5272A.
2. Each 24-hour period, the specimens were removed from the chamber blotted dry on the surface, and reweighed. Total test time was 11 days.

E. Corrosion Resistance

1. 2 x 1 3/8 x 1/8 inch specimens of each material were cast on 3 x 6 inch panels of anodized aluminum, copper, silver-plated copper, and gold-plated copper.

2. The panels were exposed in the salt spray chamber for 6 days. The panels were examined for signs of corrosion at 24-hour intervals.

F. Application Time was determined.

III. Electrical Properties

A. Dielectric Strength

Dielectric strength was determined on 6 x 6 x 0.050 inch specimens of each material.

B. Dielectric Constant and Dissipation Factor

Dielectric constant and dissipation factor were determined with a Dalsen D-K analyzer.

C. Volume and Surface Resistivity

Volume resistivity and surface resistivity were determined on 6 x 6 inch specimens. The panel made from Epon 828 with curing agent Z was 0.145 inch thick and the panel made from Epon 828 with curing agent D was 0.110 inch thick.

D. Insulation Resistance

1. Two electrodes 0.25 inch long by 0.032 inch in diameter were spaced 0.3 inch apart in a 6061-T6 aluminum tube, 1 inch long x 1 inch in diameter which was embedded with one of the materials. The electrodes were formed by stripping 0.25 inch sections of insulation from the centers of 24-inch lengths of sodium-naphthalene-treated, teflon-insulated wire, MIL-W-16878-EE20.

2. The initial insulation resistance was measured at 100 V.D.C. potential.

3. The specimens were tested at three temperatures - 125°C, 40°C, and -55°C. Three specimens of each material were held at constant temperature for a total of 216 hours. The insulation resistance was determined at 24-hour intervals.

4. Three specimens of each material were conditioned in the humidity chamber per MIL-E-5272A for 216 hours. Insulation resistance of the specimens was measured each 24 hours at 160°F and 95% relative humidity.

TABLE I

PHYSICAL PROPERTIES OF EMBEDMENT MATERIALS BETWEEN -55°C AND 125°C

Material	Specific Gravity	Hardness Coleman Smith		Resistance to Thermal Shock 125°C to -55°C per MIL-I-16923	Moisture Absorption 264 hours at 95% R.H.	Corrosion Resistance (Degree of Corrosion after 6 days in 20% Salt Spray)	Appli-cation Time	Cure Cycle
		25°C	125°C					
Epon 828 with Curing Agent Z	1.204	75	75	10 cycles No failure	0.8%	Aluminum - None Cooper - Slight Gold - None Silver - very slight	8 hours	2 hours at 180°F
Epon 828 with Curing Agent Z	1.175	80	*	1 specimen 10 cycles No failure 1 specimen Failure after 4 cycles	1.4%	Aluminum - None Copper - very slight Gold - very slight Silver - None	4 hours	1 hour at 200°F

* Material too soft to measure.

TABLE II

ELECTRICAL PROPERTIES OF EMBEDMENT MATERIALS BETWEEN -55°C AND 125°C

Material	Dielectric Strength (V/MIL)	Dielectric Constant at 10 Kc	Dissipation Factor at 10 Kc	Volume Resistivity (OHM-CM)	Surface Resistivity
Epon 828 with Curing Agent Z	483	3.25	0.010	5.5×10^{14}	2.5×10^{14}
Epon 828 with Curing Agent D	525	3.1	0.006	3.6×10^{15}	2.5×10^{14}

TABLE III

VARIATION OF INSULATION RESISTANCE OF MATERIALS WITH TIME UNDER VARIOUS CONDITIONS(2)

Material	Test Condition	Specimen Number	Aging Time at Condition in Hours									
			0	24	48	72	96	120	144	168	192	216
Epon 828 with Curing Agent Z	125°C	1 Z	*	*	*	*	(3)	(3)	*	*	*	*
		2 Z	*	*	*	*			*	*	*	*
		3 Z	*	*	*	*			*	*	*	*
	40°C	4ZZ	*	*	*	(3)	(3)	*	*	*	*	*
		5 Z	*	*	*			*	*	*	*	*
		6 Z	*	*	*			*	*	*	*	*
	-55°C	7 Z	*	*	*	(3	(3)	*	*	*	*	*
		8 Z	*	*	*	(3)		*	*	*	*	*
		9 Z	*	*	*			*	*	*	*	*
	Humidity Chamber(4)	10 Z	*	*	*	*	(3)	(3)	*	*	*	*
		11 Z	*	*	*	*			*	*	*	*
		12 Z	*	*	*	*			*	*	*	*
Epon 828 plus curing agent D	125°C	1 D	24,000	40,000	13,000	13,000	(3)	(3)	19,000	25,000	26,000	28,000
		2 D	24,000	36,000	13,000	14,000			19,000	25,000	26,000	28,000
		3 D	22,000	32,000	12,000	13,000			17,000	21,000	23,000	26,000
	40°C	4 D	*	*	*	(3)	(3)	*	*	*	*	*
		5 D	*	*	*			*	*	*	*	*
		6 D	*	*	*			*	*	*	*	*
	-55°C	7 D	*	*	*	(3)	(3)	*	*	*	*	*
		8 D	*	*	*			*	*	*	*	*
		9 D	*	*	*			*	*	*	*	*
	Humidity Chamber(4)	10 D	*	*	*	*	(3)	(3)	*	*	*	*
		11 D	*	*	*	*			*	*	*	*
		12 D	*	*	*	*			*	*	*	*

(1) Asterisk (*) denotes infinite resistance
(2) All values reported in megohms
(3) No values obtained due to week end
(4) Chamber operated per MIL-E-5272A, insulation resistance measured while chamber was at 160°F and 95% relative humidity.

EVALUATION OF ELECTRONIC EMBEDMENT MATERIALS FOR USE AT 450°F

INTRODUCTION

Electronic modules embedded per standard procedure have not performed well during moisture environment qualification of certain electronic equipment. This condition necessitated the qualification of improved materials for this purpose. In addition, some growth potential with respect to temperature limitations is desired.

OBJECT

To determine if 3M EC 1663 potting compound or 3M XD-911845 experimental material has the necessary electrical and physical properties for use in module embedment applications.

CONCLUSION

Minnesota Mining and Mfg. EC 1663 embedment compound has excellent electrical and physical properties for module embedment applications. The experimental compound, XD-911845 will require further development by the manufacturer. The electrical properties of XD-911845 fell sharply as the temperature was increased.

MATERIALS

EC 1663 Silicone embedment compound.

XD-911845 experimental material.

XB 9118-13 primer for XD-911845, all manufactured by Minnesota Mining and Mfg. Company, St. Paul, Minnesota.

Churchill G-75 silicone primer manufactured by Churchill Chemical Company, Los Angeles, California.

PROCEDURE

I. GENERAL

A. Primers

1. Silicone (EC 1663) specimens were primed with G-75 primer. The primer was allowed to cure 30 minutes to 1 hour prior to application of sealant.

2. XD 911845 specimens were primed with XB 9118-13 primer. The primer was air dried 30 minutes plus 30-minute cure in circulating air oven at 180°F.

B. Cure

1. EC 1663 specimens were cured 7 days at room temperature prior to testing.

2. XD 911845 specimens were cured 3 hours at 200°F plus 7 days at room temperature.

II. PHYSICAL PROPERTIES

A. Specific Gravity

1. Cylindrical specimens 1 inch in diameter and 0.5 inch thick were cast in polyethylene molds (Cannon cap #498).

2. Specific gravity of the cured specimens was determined by the water displacement method.

B. Viscosity

Viscosity was determined on each base material and each mixed material with a Brookfield Viscometer. A number 6 spindle was used at 4 rpm.

C. Application Time

Application time was determined.

D. Water Absorption

1. Weighed specimens, similar to those described in IIA, were immersed in distilled water. Each 24-hour period the specimens were removed from the water, blotted dry on the surface, and reweighed. Total test time was 10 days.

2. Test II.D.1 was repeated except the specimens were conditioned in the humidity chamber operated per MIL-E-5272A.

E. Shear Strength

1. Preparation - One-inch lap shear specimens were prepared with the following systems:

   a. G-75 primer plus EC 1663 compound
   b. XS 9118-13 primer plus XD 911845 compound
   c. No primer, XD 911845 compound

2. Conditioning - One set of three cured lap shear specimens of each system above was conditioned in each of the following environments:

a. Controls (no conditioning)
b. Aged 100 hours at 450°F
c. Aged 100 hours in humidity chamber operated at 95% relative humidity per MIL-E-5272A.

3. Testing

Each conditioned specimen was pulled on a Baldwin Tensile Machine at a rate of 4,000 pounds/minute.

F. Primer Application Method

1. Alodined 6061-T6 aluminum panels, 2 x 6 x 0.032 inches, were primed with either G-75 or XS 9118-13 primer by each of the following methods.

   a. Brush
   b. Dip
   c. Spray

2. Each primer specimen was coated with the appropriate sealant and cured.

3. Each specimen was inspected to determine adherence of the sealant.

4. Each specimen was aged 10 days in the humidity chamber operated at 95% relative humidity per MIL-E-5272A and reinspected for adherence of sealant.

III. ELECTRICAL PROPERTIES

A. Dielectric Strength - Determined on 6 x 6 x 0.063 inch specimens.

B. Volume and Surface Resistivity - Determined on 4 x 4 x 0.125 inch specimens.

C. Dielectric Constant and Dissipation Factor - Dielectric constant and dissipation factor at 10 KC were determined with a Delsen D-K analyzer.

D. Insulation Resistance

1. Two electrodes 0.25 inch long x 0.032 inch in diameter were spaced 0.3 inch apart in a 6061-T6 aluminum tube, 1 inch long x 0.875 inch in diameter, which was embedded with the sealant material. The electrodes were formed by stripping 0.25 inch sections of insulation from the centers of 24-inch lengths of sodium-naphthalene treated teflon-insulated wire, MIL-W-16878-EE20.

2. The initial insulation resistance was measured at 500 V.D.C. potential.

3. Three specimens of each material were raised in 50°F increments to 450°F. The specimens were soaked at each incremental temperature for 30 minutes. The insulation resistance was measured at the end of the 30-minute soak period. The specimens were maintained at 450°F for 240 hours. Insulation resistance was measured at 450°F each 24 hours during aging.

4. Three specimens of each material were aged in humidity chamber per MIL-E-5272A for 240 hours. The insulation resistance of the specimens was measured each 24 hours in the humidity chamber at 160°F and 95% relative humidity.

RESULTS

Results are given in Tables I and II and Figures 1, 2, and 3.

TABLE I

PHYSICAL AND ELECTRICAL PROPERTIES OF EMBEDMENT

Material	Specific Gravity	Viscosity (poise)		Water Absorption		Shear Strengt	
		Base	Mixed Material	After 240 hrs. Immersion in Distilled Water	After 240 hrs. in 95% R.H. per MIL-E-5272A	Controls (as cured)	Aft Agi 240 at
EC 1663	1.48	316	351	No Wt. Gain	1.1% Wt. Loss	527 CF	267
XD 9118 -45 B/A	1.22	3540	420	1%	3%	720 CF (2) 150 AF (3)	290 130

Material	Volume Resistivity (OHM - CM)	Surface Resistivity (OHM)	Insulation Resistance (1) (MEG - OHM) (4)				Diele at 10
			As cured	200°F	300°F	450°F	
EC 1663	$6.7x10^{13}$	$1.7x10^{14}$	500,000	500,000	500,000		
XD 9118 -45 B/A	$1.6x10^{9}$	$1.48x10^{9}$	5,400	47	7	2.3	

(1) See Figures 1,2 and 3 for Aging Characteristics.
(2) Primed with XD-911813 B/A Primer
(3) Unprimed
(4) Graphic Presentation in Figure 1
AF - Adhesive Failure
CF - Cohesive Failure

1

TABLE I

AL PROPERTIES OF EMBEDMENT MATERIALS FOR 450°F APPLICATION

	Shear Strength (psi)			Application Time (hrs.)	Cure Cycle	Dielectric Strength (V/MIL)
. r 272A	Controls (as cured)	After Aging 240 hrs. at 450°F	After Aging 240 hrs. in 95% R.H. per MIL-E-5272A			
t.	527 CF	267 CF	383 CF	3	7 days at 77°F	440
	720 CF (2) 150 AF (3)	290 CF (2) 130 AF (3)	500 CF (2) 100 AF (3)	2	16 hrs. at 160°F followed by 2 hrs. at 200°F	208

(1) (4)		Dielectric Constant at 10 KC	Dissipation Factor at 10 KC	Primer Application Method
300°F	450°F			
500,000		4.3	0.009	Brush or Dip
7	2.3	10.7	0.125	Brush, Dip or Spray

2

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TABLE II

INSULATION RESISTANCE OF MATERIALS FOR 450°F APPLICATION

Material	Test Condition	Specimen Number	Test Leads	0 Hrs.	24 Hrs.	48 Hrs.	72 Hrs.	96 H
EC 1663	450°F (1)	1	A to Gnd.	100,000	100,000	100,000	100,000	100,
			B to Gnd.	100,000	100,000	100,000	100,000	100,
			A to B		500,000	500,000	500,000	500,
		2	A to Gnd.	100,000	100,000	500,000	100,000	500,
			B to Gnd.	100,000	100,000	500,000	100,000	500,
			A to B		500,000		500,000	500,
		3	A to Gnd.		100,000		500,000	500,
			B to Gnd.	500,000	100,000		500,000	500,
			A to B		500,000		500,000	500,
	Humidity Chamber (2)	4	A to Gnd.	100,000	14,000	3,000	1,300	
			B to Gnd.	50,000	14,000	4,000	1,800	1,
			A to B	50,000	20,000	8,000	3,600	2,
		5	A to Gnd.	50,000	10,000	4,000	2,200	1,
			B to Gnd.	40,000	10,000	3,200	2,000	1,
			A to B	40,000	15,000	9,000	8,000	2,
		6	A to Gnd.	50,000	12,000	8,000	5,500	4,
			B to Gnd.	30,000	12,000	9,000	9,000	4,
			A to B	50,000	20,000	20,000	40,000	12,
XD-911845 B/A	450°F (1)	7	A to Gnd.	1	1	1	1	1
			B to Gnd.	1	1	1	1	1
			A to B	2.5	8	1	1	1
		8	A to Gnd.	1	1	1	1	1
			B to Gnd.	1	1	1	1	1
			A to B	1	1	1	1	1
		9	A to Gnd.	1	1	1	1	1
			B to Gnd.	1	1	1	1	1
			A to B	2.1	3	1	1	1
	Humidity Chamber (2)	10	A to Gnd.	2,200	55	75	24	
			B to Gnd.	2,400	75	60	38	
			A to B	4,300	130	95	60	
		11	A to Gnd.	2,300	90	70	45	
			B to Gnd.	2,300	80	60	40	
			A to B	4,400	160	140	80	
		12	A to Gnd.	2,100	90	75	50	
			B to Gnd.	1,900	90	70	45	
			A to B	3,900	180	160	90	

Notes: (1) **Measurements were taken at 450°F in a forced draft oven.**
(2) **Measurements were taken at 160°F in humidity chamber operated per MIL-E-5272A, 95% Relative**
(3) **All values reported in megohms.**

TABLE II

NCE OF MATERIALS FOR 450°F APPLICATION

Hrs.	24 Hrs.	48 Hrs.	72 Hrs.	96 Hrs.	168 Hrs.	192 Hrs.	216 Hrs.	240 Hrs.
,000	100,000	100,000	100,000	100,000	500,000	500,000	500,000	500,000
,000	100,000	100,000	100,000	100,000	500,000	500,000	500,000	500,000
	500,000	500,000	500,000	500,000	500,000	500,000	500,000	500,000
,000	100,000	500,000	100,000	500,000	500,000	500,000	500,000	500,000
,000	100,000	500,000	100,000	500,000	500,000	500,000	500,000	450,000
	500,000		500,000	500,000			500,000	
	100,000		500,000	500,000	500,000	500,000	500,000	500,000
,000	100,000		500,000	500,000	500,000	500,000	500,000	500,000
	500,000		500,000	500,000			500,000	
,000	14,000	3,000	1,300	900	650	650	600	500
,000	14,000	4,000	1,800	1,300	650	650	600	400
,000	20,000	8,000	3,600	2,400	1,400	1,300	1,200	850
.000	10,000	4,000	2,200	1,500	1,000	1,000	900	600
.000	10,000	3,200	2,000	1,200	800	850	750	550
.000	15,000	9,000	3,000	2,800	2,000	2,100	2,500	1,300
000	12,000	8,000	5,500	4,000	2,400	2,500	2,300	2,000
000	12,000	9,000	9,000	4,300	1,100	1,500	1,200	2,300
000	20,000	20,000	40,000	12,000	3,600	5,500	4,000	5,000
	1	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	8	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	1	1	1	1	1	1	1	1
	3	1	1	1	1	1	1	1
200	55	75	24	20	11	10	10	6
400	75	60	38	32	17	15	13	8
300	130	95	60	50	28	25	22	15
300	90	70	45	36	18	15	13	8
300	80	60	40	35	19	16	14	9
400	160	140	80	70	35	30	26	17
100	90	75	50	40	20	17	15	10
00	90	70	45	38	19	17	14	10
00	180	160	90	75	36	32	28	18

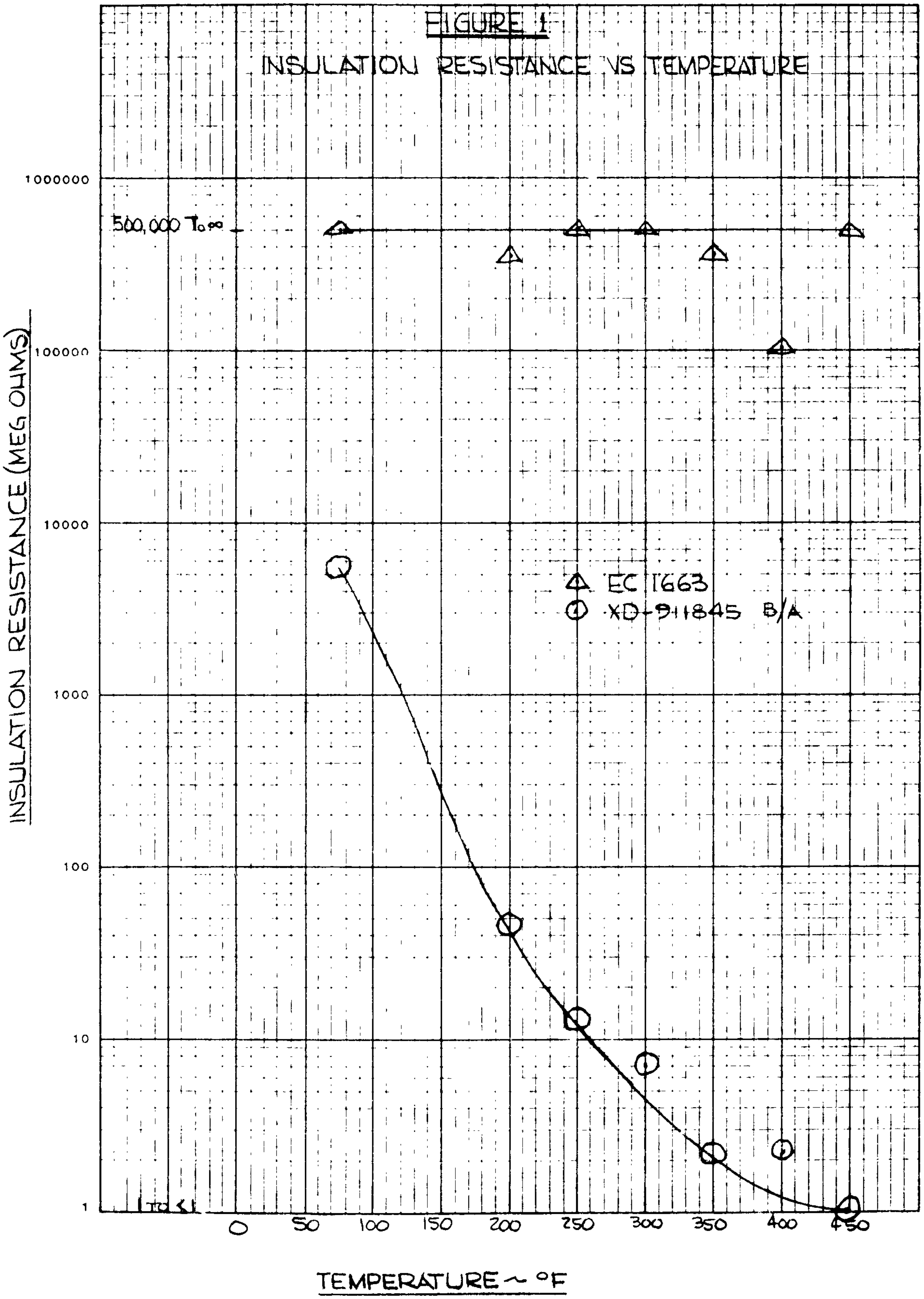
ft oven.
mber operated per MIL-E-5272A, 95% Relative Humidity

2

MODEL **DATE**

FIGURE 1

INSULATION RESISTANCE VS TEMPERATURE



MODEL **DATE**

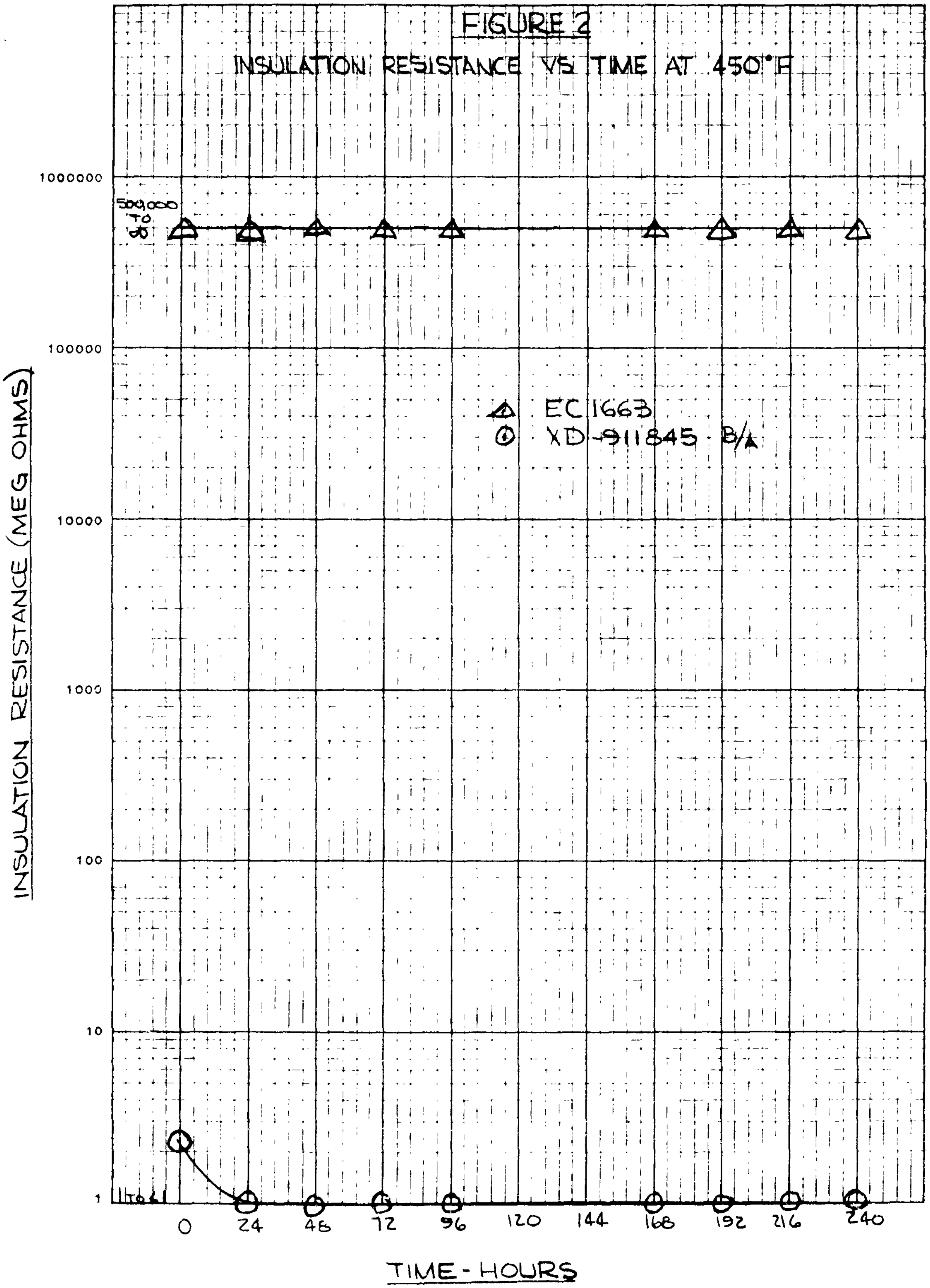


FIGURE 2

INSULATION RESISTANCE VS TIME AT 450°F

MODEL DATE

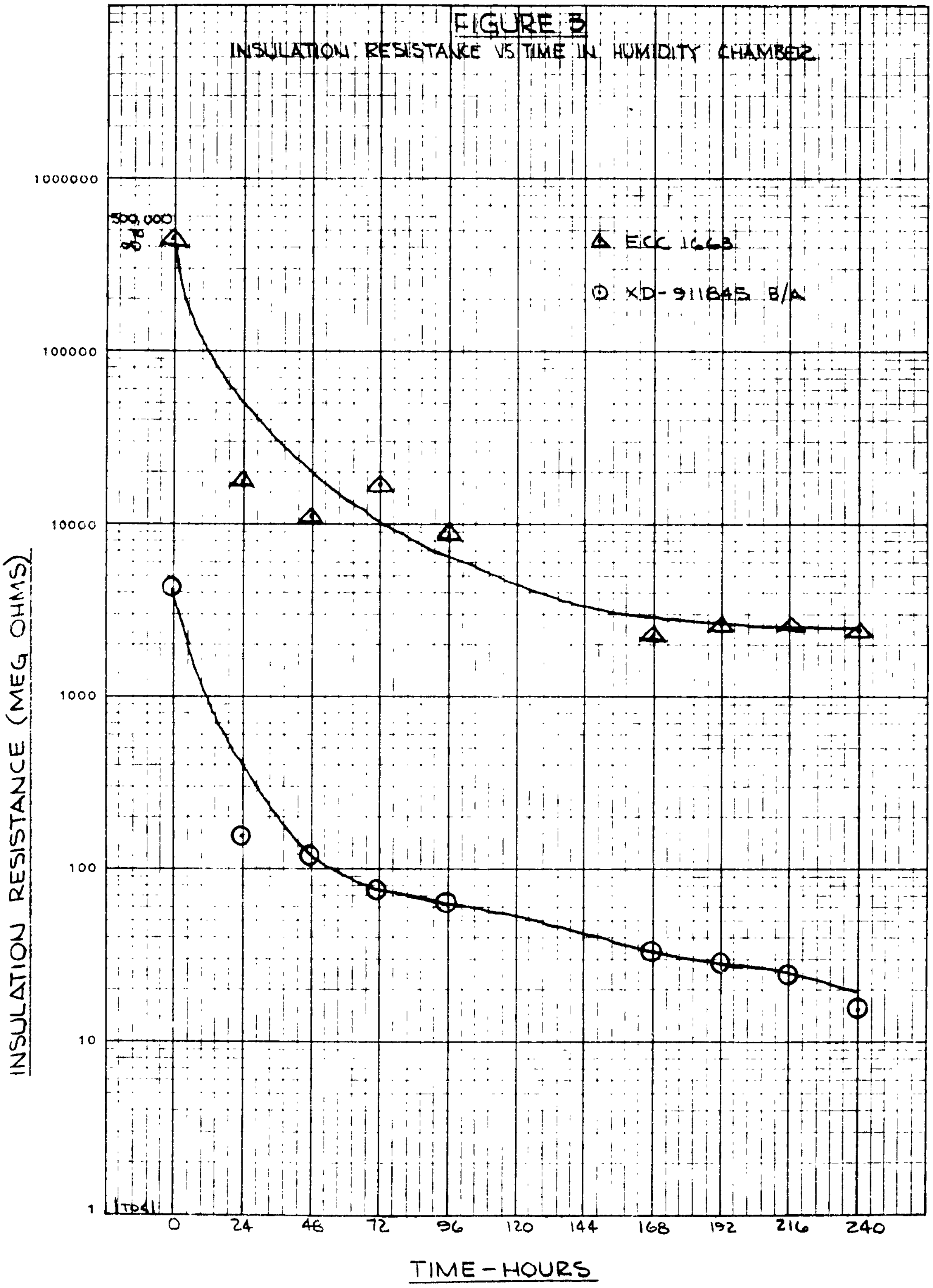


FIGURE 3

INSULATION RESISTANCE VS TIME IN HUMIDITY CHAMBER

K&E SEMI-LOGARITHMIC 359-96
KEUFFEL & ESSER CO. MADE IN U.S.A.
7 CYCLES X 60 DIVISIONS

EVALUATION OF POTTING COMPOUNDS FOR 500°F APPLICATIONS

INTRODUCTION

Increasing temperature requirements in aircraft and missiles have indicated a need for high temperature potting compounds. Original preparations of leading compound manufacturers showed good promise but left some properties to be desired. Recent advances in the state of the art have yielded compounds which should be superior to the prototype materials. The purpose of this test is to determine which materials are suitable for 500°F potting application.

OBJECT

To perform physical properties and electrical tests on potting compounds for 500°F application.

CONCLUSION

Dow Corning RTV 501 and Minnesota Mining and Mfg. EC 1663 were the most satisfactory compounds tested. General Electric RTV 60 plus T-12 catalyst meets the physical and electrical requirements, but has a short work life and is difficult to mix properly. These conditions may be alleviated by use of a new paste catalyst which became available too late to include in this test. G. E. RTV 81813 was the prototype material to RTV-60 and had unsatisfactory high temperature properties. Proseal 792 also had poor high temperature characteristics.

PROCEDURE

I. General: All tests described herein were performed on the catalyzed and/or cured sealant materials.

A. Application Time:

Application time was determined by extruding the mixed material from a Semco Gun through a Semco 254 nozzle at 90 psi pressure onto a tared container for a measured period of time. The volume of sealant per minute was computed. The application time was taken as the time at which the extrusion rate had dropped to 25 milliliters per minute.

B. Curing Rate Hardness:

The sealant material was cast into a 1 1/4 x 3 x 1/4 inch mold and cured at room temperature for 72 hours. The instantaneous Shore A hardness was determined on this specimen with a Shore A durometer.

C. Shrinkage:

The sealant was cast into a cubical mold approximately 1.0 inch on a side and open at the top. The sealant material was cured 7 days at room temperature. The volume of the cube was accurately determined by the water displacement method. The cube was placed in a circulating air oven at 500°F for 48 hours. The sealant was cooled to room temperature and volume redetermined.

D. Non-Volatile Content:

Approximately three grams of mixed sealant were weighed accurately in a tared glass container. The material was heated at 160°F for 24 hours in a circulating air oven. The material was cooled in a desiccator and reweighed.

E. Peel Strength:

One inch wide peel strength specimens were prepared:

1. Two x 2 3/8 x 0.064 inch panels of aloidined 7075-T6 aluminum, 301 stainless steel and titanium alloy were primed with G.E. SS 67 silicone primer. One half inch strips of aluminum 0.064 inch thick were attached to the panels to form a cavity 1 inch x 1 3/8 inch. This cavity was filled with sealant and a brass screen placed over the wet sealant material.

2. Approximately 1/16 inch of sealant was extruded over the screen and the whole assembly cured. Three specimens of each material and metal were reserved as controls. Three specimens of each material and metal were aged 7 days in a 3% salt water solution. Three stainless steel and three titanium panels of each material were aged 168 hours at 500°F. After aging the specimens were peeled at 90° peel on a Scott Test Machine.

F. Low Temperature Flexibility:

A film 0.064 inch thick of each sealant material was cast on 1 x 6 x 0.032 inch sheets of stainless steel. The sealant was cured 7 days at room temperature. Each specimen was aged 7 days at 500°F. At the end of this period the panels were cooled to -65°F for four hours. While at -65°F the specimens were each bent around a 2 inch radius mandrel and observed for cracking.

H. Sponging Resistance:

Hemispheres approximately 1 1/2 inch in diameter were cast of each sealant material. After curing 7 days the materials were subjected to 500°F and observed each hour for a period of four hours. Those specimens which were still intact were aged a total of 24 hours at 500°F.

II. Electrical Tests:

A. Dielectric Strength:

Dielectric Strength was determined at room temperature.

B. Dielectric Constant and Power Factor:

Dielectric constant and power factor of RTV 81813 and DC 501 were determined at 1 kilocycle. At a later date, the dielectric constant and power factor of the two remaining materials were tested with a direct reading bridge (Delsen D-K analyzer) at 10 kilocycles. The power factor was determined simultaneously.

C. Volume and Surface Resistivity:

Volume and surface resistivity were determined at room temperature.

D. Insulation Resistance:

Four electrodes, formed by stripping 3/8 inch of the Teflon insulation from four No. 20 A.W.G. wires, were imbedded in sealant contained in a 0.70 inch diameter alodined aluminum tube, 1 inch long. The sealant was cured 7 days at room temperature. Insulation resistance was measured between each lead and the shell at 500 V.D.C. potential. The specimens were raised to 500°F and insulation resistance remeasured. The specimens were aged a total of 168 hours with measurements being made every 24 hours.

E. High Potential Resistance:

Specimens prepared as in D. above were subjected to a potential of 2000 V RMS between each pin and the shell. Voltage was applied gradually at the rate of 500 volts per second.

F. Humidity Resistance:

Specimens prepared as in D. above were exposed to 5 temperature cycles of 500°F for 30 minutes followed by -65°F for 30 minutes. After these five cycles the specimens were subjected to 95% relative humidity for a period of 14 days. Insulation resistance at 500 V.D.C. was measured at room temperature after removal from the humidity chamber. The specimens were high potential tested at 750 V RMS for 5 minutes at room temperature.

TABLE I

PHYSICAL PROPERTIES OF POTTI

MATERIAL	APPLICATION TIME HRS.	SHORE A HARDNESS AFTER 72 HOURS	SHRINKAGE (VOLUME)	NON-VOLATILE CONTENT	(1) 7 DAYS IN 3% SALT SOLUTIO		
					ALUMINUM	STAINLESS STEEL	TIT
G. E. RTV 81813 + 1% L-24	2	45	8.05%	98.5%	3	6 (2)	
DC RTV 501	1.5	35	4.16%	98.1	5	5	
G.E. RTV 60 + 1% T-12	0.5	53	8.10%	98.6	6	5	
EC 1663	3	45	7.05%	98.1	7	6	
Proseal 792	2	45	8.74%	98.5	7	6	

TABLE I

ELECTRICAL PROPERTIES OF POTTING COM

MATERIAL	DIELECTRIC STRENGTH VOLTS/MIL	DIELECTRIC CONSTANT		POWER FACTOR		VOLUME RESISTIVITY OHM-CM.	SURFAC RESISI OHMS
		1 KC	10 KC	1 KC	10 KC		
G. E. RTV 81813 + 1% L-24	200	4.67		0.010		183 X 10"	570 X
D. C. RTV	200	3.22		0.006		213 X 10^{12}	27 X
G. E. RTV 60 + 1% T-12	207		4.0		0.008	66 X 10^{12}	215 X
EC 1663	205		4.3		0.009	67 X 10^{12}	172 X
Proseal 792	(1)	(1)	(1)	(1)	(1)	(1)	(1)

(1) This material not tested electrically due to sponging and/or reversion at 500°F.

TABLE I

ERTIES OF POTTING COMPOUNDS FOR 500°F APPLICATION

PEEL STRENGTH LB/IN. (3) (1) 3% SALT SOLUTION		PEEL STRENGTH LB/IN. (3) CONTROLS			AFTER AGING 168 HR AT 500° F.		LOW (3) TEMPERATURE FLEXIBILITY	REMARKS
STAINLESS STEEL	TITANIUM	ALUMINUM	STAINLESS STEEL	TITANIUM	STAINLESS STEEL	TITANIUM		
6 (2)	6	5	6	5	5	5	Passed	Material sponged at 500° F
5	-(2)	5	5	5	2	2	Passed	
5	6	5	5	5	7	7	Passed	
6	4	5	6	5	6	7	Passed	
6	5	4	4	5	4	3	Passed	Material sponged at 500° F

2

TABLE II

S OF POTTING COMPOUNDS FOR 500°F APPLICATION

E TIVITY M.	SURFACE RESISIVITY OHMS	INSULATION RESISTANCE AT 500 VDC MEGOHMS 75° F AS CURED	30 MIN. AT 500°F	168 HR. AT 500°F	HIGH POTENTIAL RESISTANCE 2000 V RMS	HUMIDITY INSULATION RESISTANCE MEGOHMS	RESISTANCE HIGH POTENTIAL RESISTANCE 750 V RMS
X 10"	570 X 10^{10}	(1)	(1)	(1)	(1)	(1)	(1)
X 10^{12}	27 X 10^{12}	①	50K	①	Passed	①	Passed
X 10^{12}	215 X 10^{12}	①	100K	①	Passed	①	Passed
X 10^{12}	172 X 10^{12}	①	①	①	Passed	①	Passed
	(1)	(1)	(1)	(1)	(1)	(1)	(1)

500°F.

DETERMINATION OF PHYSICAL PARAMETERS OF PROSEAL 793 CABLE MOLDING COMPOUND

INTRODUCTION

A mixture of MIL-S-8516 material and carbon black has been used in the past as a cable molding compound. However, this material has not proven satisfactory with regard to its physical properties. Coast Proseal 793 compound was submitted and showed very good characteristics when fabricated into typical parts for which the material is to be used. For specification and design purposes, it is necessary to determine some physical and handling characteristics of this material not already known.

OBJECT

To determine physical properties of Proseal 793.

CONCLUSION

Proseal 793 is suitable as a cable molding compound. The requisites of high tensile strength and good abrasion resistance were met with no apparent defects.

MATERIAL

Proseal 793 cable molding compound, manufactured by Coast Proseal Company, Los Angles, California.

PROCEDURE

GENERAL: All specimens below were prepared from material which had received 27 inch vacuum for 45 minutes.

1. Specific Gravity: Specific gravity was determined.

2. Viscosity: Viscosity was determined with a Brookfield Viscometer using a #6 spindle at 10 r.p.m.

3. Flow: The evacuated compound was placed in the Boeing flow jig and the plunger advanced. The time required for the material to flow 4 inches was determined with a stop watch and calculated as inches of flow per minute.

4. Extrusion Rate and Application Time: Extrusion rate was determined. This curve was correlated with an arbitrary value for application time to establish an index for determining application time.

5. Tack Free Time: Tack free time was determined for room temperature cure. the tack free time was also determined for 180°F cure using the same method used for room temperature. Tack free time was calculated from time of removal from vacuum chamber.

6. Curing Rate Hardness: Hardness was determined using a Shore A durometer after 24 hours and 72 hours at room temperature. In addition, hardness was determined at room temperature after aging 2, 4, and 6 hours at 180°F.

7. Abrasion Resistance: Abrasion resistance was determined using a Taber Abrader with CS15 wheels and a 1000-gram load. Two specimens were each rotated 5,000 cycles and the amount of wear measured with a micrometer.

8. Low Temperature Flexibility: Low temperature flexibility was determined.

9. Tensile Strength: A 1/8 inch thick slab of Proseal 793 was cast in a Teflon-coated mold. After curing 6 hours at 180°F, the material was cut into dumbbell specimens using ASTMD4-12-51T die D. The specimens were pulled in a Scott Tested at 2 inch/minute.

RESULTS

Test data results are given in Table I and Graph I.

FIGURE I

PHYSICAL PROPERTIES OF PROSEAL 793 CAUL

SPECIFIC GRAVITY	VISCOSITY(1) (POISE)(2)	FLOW(1) Inches per minute	ABRASION(3) RESISTANCE 5000 cycles	LOW TEMPERATURE FLEXIBILITY	TENSILE STRENGTH PSI	APPLIC TI
1.06	600-650(2)	[illegible]	No failure	Passed	2300	1.5

(1) After 45 minutes at 27 inches vacuum.

(2) Brookfield Spindle No. 6 at 10 R.P.M.

(3) Less than 1 mil wear using Taber Abrader, [illegible] wheels with 1 Kg. load.

(4) See Figure II for extrusion rate.

1

GURE I

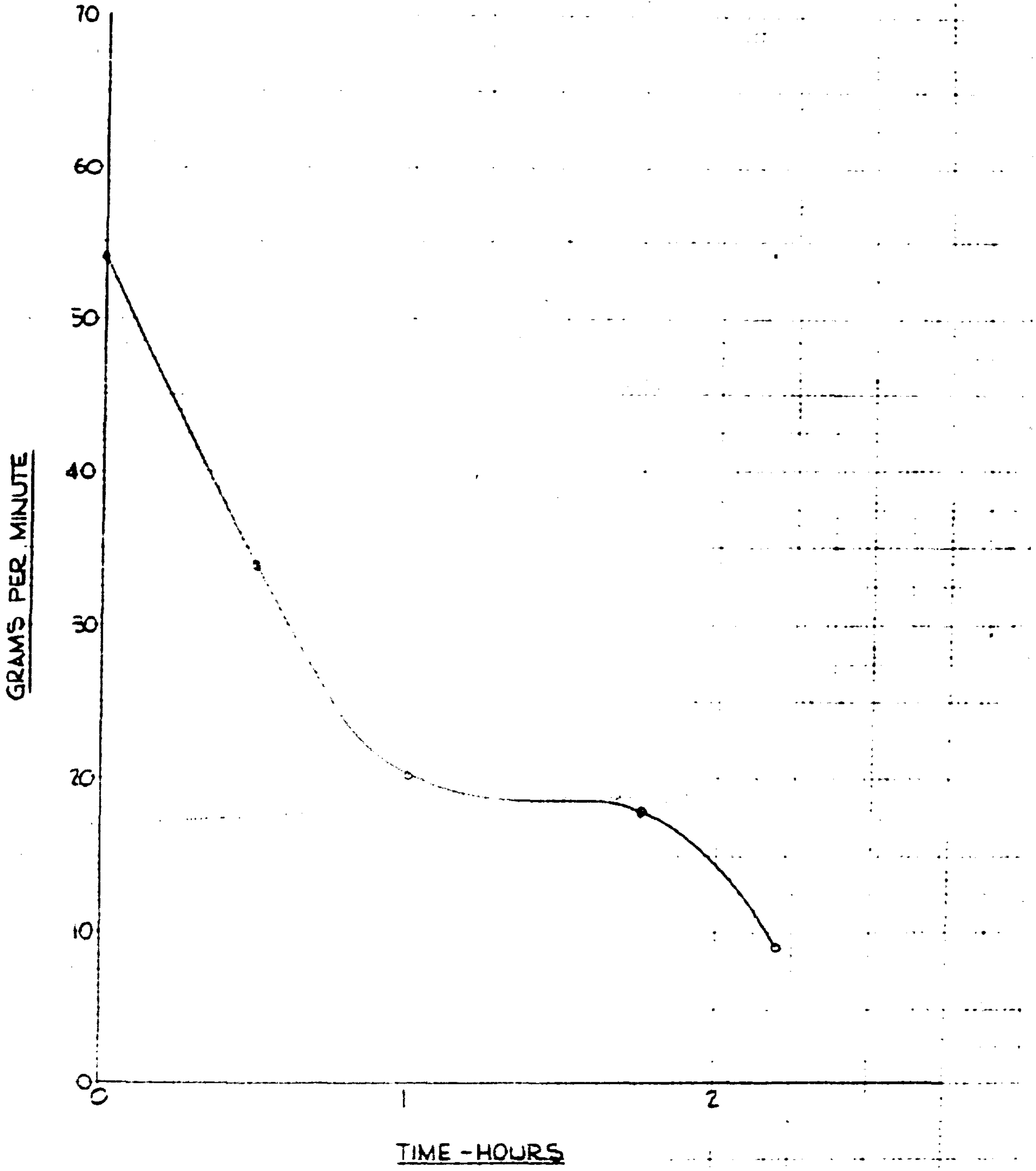
EAL 793 CABLE MOLDING COMPOUND

APPLICATION(1) TIME (4)	TACK FREE TIME		HARDNESS SHORE A					
	Room Temp.	180°F	Room Temperature		180°F			Ultimate
			24 hours	72 hours	2 hours	4 hours	6 hours	
1.5 hours	24 hours	[illegible] hours	40	70	70	77	80	85

2

FIGURE 1

PROSEAL 793 EXTRUSION RATE



EVALUATION OF MATERIALS FOR USE AS ELECTRICAL ENCAPSULATION COMPOUNDS

INTRODUCTION

Sealant (EC-1293) has been used for encapsulating electrical connections. The material specification by which EC 1293 is procured does not contain any electrical requirements. On the basis of past success using EC 1293 and a verbal agreement with the manufacturer that the formulation would meet certain electrical requirements and would not be changed, it was agreed to allow the continued use of this material pending electrical test data of EC 1293 vs Proseal 727 filled with Quartz.

The handling and application characteristics of EC 1293 were two important reasons for the original selection of this material for this application. However, the filling of Proseal 727 with quartz produces a material with comparable handling and application characteristics.

The purpose of this test is to compare the properties of EC 1293 and Proseal 727 filled with quartz for use as an electrical encapsulation compound.

OBJECT

To determine if EC 1293 and Proseal 727 plus quartz meet the specifications for electrical encapsulation by testing for:

A. Physical properties

B. Electrical properties

CONCLUSION

A. EC 1293 meets all physical requirements. The only physical test that Proseal 727 plus Quartz failed was working time.

B. Proseal 727 passed every electrical test performed.

MATERIALS

1. EC 1293/1366, manufactured by the Minnesota Mining and Manufacturing Company, Dallas, Texas.

2. Proseal 727, manufactured by Coast Pro-Seal and Manufacturing Company, Los Angeles, California.

3. Quartz, Grade P, manufactured by Charles P. Crystal, Inc., New York, New York.

PROCEDURE

1. Preparation of encapsulating material.

a. EC 1293 was prepared by mixing twelve grams of catalyst EC 1366 per one hundred grams of EC 1293.

b. Filled Proseal 727 was prepared by mixing Proseal 727 and quartz at a ratio of 1:1 by weight and adding twelve grams of catalyst 727A per one hundred grams of 727. The catalyst was added after Proseal 727 and quarts had been thoroughly mixed.

2. Testing: Each of the encapsulating materials was tested as follows:

a. Physical

(1) Working time
(2) Hardness
(3) Shrinkage
(4) Solids content
(5) Adhesion
(6) Corrosion
(7) Low-temperature flexibility

b. Electrical

(1) Resistance to arc: Not performed due to lack of equipment.

(2) Dielectric strength

(3) Dielectric constant and power factor

(4) Volume and surface resistivity

(5) Insulation resistance

(6) High-potential resistance

(7) Humidity resistance

(8) Moisture resistance

(9) Vibration resistance

(10) Flame resistance

(a) Overload of wire

(b) Open flame

RESULTS

Results of these tests are shown in Tables I and II.

TABLE I

PHYSICAL TEST OF MATERIALS FOR USE AS ELECTRICAL ENCAPSULATION COMPOUNDS

SEALANT	WORK LIFE	% SOLIDS	HARDNESS	SHRINKAGE
EC 1293	6 HRS.	99.42%	35	4.71%
727 + QUARTZ	40 MIN.	98.27%	65	6.30%

SEALANT	PEEL STRENGTH	RESISTANCE TO CORROSION	LOW TEMPERATURE FLEXIBILITY
EC 1293	20 LB COHESIVE FAILURE	NONE	PASSED
727 + QUARTZ	9 LB. SCREEN FAILURE	NONE	PASSED

TABLE II

ELECTRICAL TESTS OF MATERIA

	RESISTANCE TO ARC	DIELECTRIC STRENGTH	DIELECTRIC CONSTANT				
			1 KC 77° F	1 MC 77° F	1 KC 185°F	1 MC 185°F	1 KC 7
EC -1293							
CURE A	(1)	OVER 200 VOLTS/MIL	10.88	11.7			.031
CURE B		OVER 200 VOLTS/ MIL	10.63	11.3	10.63	11.05	.027
727 + QUARTZ							
CURE A	(1)	OVER 200 VOLTS/MIL	6.2	7.9			0.008
CURE B		OVER 200 VOLTS/MIL	6.4	8.27	6.55	8.22	0.007

	INSULATION RESISTANCE	HIGH POTENTIAL RESISTANCE	HUMIDITY RESISTANCE
EC - 1293			
CURE A	3 OK. MEG OHMS		
CURE B	3 OK. MEG OHMS	NO BREAKDOWN	NO BREAK DOWN
727 + QUARTZ			
CURE A	5 OK MEG OHMS		
CURE B	5 OK MEG OHMS	NO BREAKDOWN	NO BREAK DOWN

1

TABLE II

TESTS OF MATERIALS FOR ENCAPSULATION

	POWER FACTOR				RESISTIVITY			
					VOLUME		SURFACE	
MC 185°F	1 KC 77°F	1 MC 77°F	1 KC 185°F	1 MC 185°F	77°F	185°F	77°F	185°F
	.031	.03			66×10^{10} OHM-CM		55×10^{10} OHMS	
1.05	.027	.03	.057	[illegible]	67×10^{10} OHM-CM	4×10^{10} OHM-CM	70×10^{10} OHMS	10.4×10^{10} OHMS
	0.008	0.025			842×10^{p} OHM-CM		202×10^{10} OHM	
8.22	0.007	0.029	[illegible]	0.019	1200×10^{10} OHM-CM	10×10^{10} OHM-CM	390×10^{10} OHM	13×10^{10} OHM

HUMIDITY RESISTANCE	MOISTURE RESISTANCE	VIBRATION RESISTANC	FLAME	RESISTANCE
			OVERLOAD	OPEN FLAME
NO BREAK DOWN	2 OK MEG OHMS	(2)	NO IGNITION	NO DRIPPING
NO BREAK DOWN	2 OK MEG OHM	(2)	NO IGNITION	NO DRIPPING

(1) RESISTANCE TO ARC TEST NOT PERFORMED DUE TO LACK OF EQUIPMENT

(2) NO ADHESION FAILURE OR CAB DAMAGE; NO AIR LEAKAGE

2

THERMAL CONDUCTIVITY OF A BRAZED STAINLESS STEEL HONEYCOMB SANDWICH PANEL

INTRODUCTION

Data on thermal conductance of brazed stainless steel honeycomb sandwich panels are required. While the thermal conductivity can often be calculated by appropriate heat transfer analyses, it is believed that actual test data are necessary to determine the validity of calculated data.

The CVC fabricated brazed honeycomb sandwich panels were submitted to Southern Research Institute for thermal conductivity determinations. Because of excessive warpage of these panels at large temperature differentials, Southern Research was able only to provide thermal conductivity data at relatively low temperature differentials with their existing equipment. Since a relatively large temperature difference across the panels was desired, apparatus was fabricated at CVC which would accomplish this objective.

OBJECT

To determine the thermal conductivity of a brazed stainless steel honeycomb sandwich panel at various hot face temperatures up to 600°F and a constant cold face temperature of approximately 225°F.

CONCLUSIONS

The thermal conductivity of the brazed stainless steel honeycomb sandwich panel evaluated was essentially a constant value of approximately 9 to 10 Btu-in./ft.^{2}-Hr.-°F at various hot face temperatures from 350°F to 600°F and a constant cold face temperature of approximately 225°F.

The silver brazing alloy proved to be the primary mode of heat transfer. This was due to the fact that the silver brazing alloy bridged across the panel by capillary action, during the brazing cycle, in the node areas of the honeycomb core.

The thermal conductivity of any silver brazed honeycomb sandwich panel will depend to a large extent upon the amount of silver in the node areas of the honeycomb core.

MATERIALS

Brazed Honeycomb Panel --

The panel was a 12" x 12" x 0.532" brazed, PH15-7Mo stainless steel honeycomb with nominal 0.016" PH15-7Mo stainless steel faces. The honeycomb foil was nominal 0.0015" thickness having 3/16" cell spacing. The brazing foil was a 92.5% silver, 7.3% copper, and 0.2% lithium alloy 0.002" thick prior to brazing. The finish on the faces was that which resulted from the "as rolled" condition and the brazing cycle heat treatment.

The thermal conductance of the brazed stainless steel honeycomb sandwich panel was determined using a vapor calorimeter apparatus designed and fabricated at CVC. The vapor calorimeter type apparatus was chosen for this work because it was believed that inherent warping of the panel at large temperature differentials would not affect the test results. Warpage can seriously affect the test results when apparatus employing rigid heating and cooling plates is used. In the vapor calorimeter apparatus, the heat flux through a test section (sandwich panel) of the calorimeter is determined from the weight of condensate collected in a given period of time, and the latent heat of vaporization of water.

In the vapor calorimeter apparatus (Figure 1) a brazed stainless steel sandwich panel was welded to and comprised the bottom section of a calorimeter which was vented to the atmosphere through two air-cooled reflux condensers. A center or metering calorimeter of known cross-sectional area was symmetrically affixed to the center area of the panel resulting in equivalent guard areas on each of the four sides of the metering calorimeter. The metering calorimeter was fitted with a condenser tube leading to the outside where the tube was cooled with ice water and the distillate collected in a volumetric flask.

The entire bottom face of the honeycomb panel was radiantly heated using two banks of eight GE T-3 quartz lamps fitted with gold-plated reflectors. These lamps were placed approximately 14 inches below the specimen surface to minimize gradient effects across the specimen face. The hot face temperature was controlled by means of a Research, Inc., ignition controller (Figure 2).

Specimen surface temperatures were monitored using chromel-alumel thermocouple wire. The ends of the thermocouple wire were spread and spotwelded to the specimen faces to insure good contact and to minimize film effects. The thermocouple leads on the hot face surface were coated with sauereisen cement in an attempt to reduce excessively high readings due to radiant heat. The leads on the cold face surface were coated with a thin epoxy coating. Various areas on both sides of the panel were monitored.(Figure 1).

Before initiating a test run, water is placed in both the guard and metering calorimeter areas of the vapor calorimeter apparatus. When this water reaches steady-state conditions, at its boiling point, a very high degree of distillation efficiency and a very low heat loss in the metering calorimeter should be achieved. This is due to the fact that the water in the guard area and its vapor phase and the water in the calorimeter and its vapor phase would all be at approximately the same temperature.

After assembling the apparatus radiant heat was applied to the brazed honeycomb panel section until the entire apparatus had come to equilibrium. The desired hot face temperature was pre-set and maintained by the ignition controller. Steady-state conditions were determined by constant temperature readings on both the hot and cold faces of the brazed panel and by a steady distillation rate of the water from the metering calorimeter. In all determinations, at least 50 ml of water was distilled prior to beginning the timed test run.

During the test, all thermocouples were read at approximately 5-minute intervals to ascertain that steady-state conditions existed.

The distillate was collected for an accurately timed interval of 30 to 60 minutes. Upon completion of the test run, the distillate was allowed to come to room temperature and the volume accurately determined.

Knowing the hot and cold face temperature, volume of distillate, time, heat of vaporization of boiling water, area of the metering calorimeter, and thickness of the panel, the thermal conductance of the panel was calculated at each temperature condition. See Table I for results.

RESULTS

Table I shows the difference in thermal conductivity values that were obtained using separate thermocouples and recorders to measure the hot face temperatures. One of the hot face thermocouples led to the ignitron temperature controller, which actually controlled the temperature of the hot face, and the other led to a multichannel recorder. These recorders were calibrated against each other and with both a millivoltmeter and a calibrated thermometer over the effective temperature range. It is likely that the difference in hot face temperature readings was due to the inherent difficulties which exist in measuring surface temperatures. Another possible contributing factor for this discrepancy is that the temperature gradient across the hot face may have been greater than expected.

The thermocouples on the cold face of the panel were coated with a thin epoxy film. In looking at this coating in retrospect, it was realized that the film would prevent free cooling of the thermocouples by the water. Figure 3 was used to correct the cold face thermocouple readings.

It is interesting to note that the thermal conductivity values, employing the vapor calorimeter apparatus, showed an increase with a corresponding decrease in hot face temperature and temperature drop across the specimens. By contrast, similar brazed panels, tested by Southern Research Institute employing an ASTM C177 guarded hot plate apparatus, showed an increase in conductivity with a corresponding increase in hot face temperature and temperature drop across the specimen. In addition, values obtained by the vapor calorimeter method proved to be much higher than those obtained with the guarded hot plate apparatus. However, different brazed honeycomb sandwich panels were used in these two tests.

The discrepancies mentioned above led to a metallographic examination of another brazed honeycomb sandwich panel in an attempt to explain the heat transfer characteristics of this type of construction.

Visual examination of this panel showed that a solid column of silver brazing alloy extended completely across the panel along the nodes of the honeycomb cells. A portion of this panel was sectioned parallel to the faces at the midpoint of the thickness of the specimen and mounted for metallographic study. This cross-section had an appearance as shown in Figure 4. The cross-sectional area of the silver brazing alloy in various node areas of this section is shown in Table II.

The cross-sectional area of the silver brazing alloy in the specimen examined proved to constitute approximately 21% of the total metallic cross-sectional area of the core. Therefore, the stainless steel honeycomb cell walls accounted for 1.91%, the brazing alloy 0.51%, and the air spaces 97.58% of the total cross-sectional area of the core.

Since the silver brazing alloy conducts heat at a rate of approximately 20 times that of the PH15-7Mo steel and approximately 10,000 times that of air, a significant amount of heat would be transferred across the silver brazing alloy in the node areas.

The following calculations show that the brazing alloy, at 21% of the total metallic cross-sectional area of the core, would conduct approximately 74% of the total heat across the panel at a hot face temperature of 600°F and a cold face temperature of 230°F:

1. Heat Flow by Conduction:

$$Q = \frac{KA\ (T_1 - T_2)}{t}$$

Q = rate of heat flow, Btu per hour
K = thermal conductivity, Btu-in./ft.^{2}-hr-°F
T_1 = hot face temperature
T_2 = cold face temperature
t = thickness
A = area

a. $$Q(\text{silver at } 415°F) = \frac{2590 \times 0.023 \times 0.21 \times 370°F}{0.5} = 9250 \text{ Btu}$$

b. $$Q(\text{PH15-7Mo foil-}415°F) = \frac{124 \times 0.023 \times 0.79 \times 370°F}{0.5} = 1670 \text{ Btu}$$

2. Heat flow by radiation and conduction in the cellular air spaces of the honeycomb core:

a. Radiation:

$$Q\ 1,2 = A \cdot \sigma\ (T_1{}^4 - T_2{}^4)\ \frac{1}{\frac{1}{\epsilon_1} + \frac{1}{\epsilon_2} - 1}$$

Q = rate of heat flow, Btu per hour
σ = constant (0.173×10^{-8})
T_1 = temperature hot face in °F
T_2 = temperature cold face in °F
ϵ_1 and ϵ_2 = emissivity of inside surfaces of hot and cold faces (assumed to be 0.1 as these surfaces were highly reflective)

$$Q \text{ (air at } 415°\text{F)} = (1)(0.173 \times 10^{-8})(1060^4 - 690^4)\frac{1}{\frac{1}{.1} + \frac{1}{.1} - 1}$$

$$= 89 \text{ Btu}$$

b. Convection:

$$Q = (h)(HR)(Ft^2)(T_1 - T_2)$$

$$h = Ck \left[\frac{a\,(T_1 - T_2)}{L}\right]^{1/4}$$

$$\text{where} \quad a = \frac{2g}{T_1 - T_2}\left[\frac{\rho^2\, C_p}{\mu\, k}\right]$$

Q = rate of heat flow, Btu per hour
h = heat transfer coefficient, Btu/HR-Ft^2-°F
g = gravity in ft/hr^2
C = natural convection constant
k = conductivity of air at 415°F, Btu/Hr-Ft-°F

T_1 = hot face temperature in °R
T_2 = cold face temperature in °R
L = characteristic length of core, ft.
ρ = density in lb/ft^3
μ = dynamic viscosity lb/hr.-ft.
C_p = specific heat in Btu/lb.-°F

(1) Solving for a :

$$a = \frac{(2)(32.17)(3600^2)}{1060 + 690}\left[\frac{(0.045^{2}*)(0.245)}{(0.064)\ (0.022)}\right]$$

$$= 16.7 \times 10^4$$

(2) Solving for h :

$$h(\text{air at } 415°\text{F}) = (0.7)(0.022)\left[\frac{16.7 \times 10\ \ (1060-690)}{0.0113}\right]^{1/4}$$

$$= 4.16$$

*Value at 415°F for air at atmospheric pressure. The atmosphere and the pressure inside the cells is unknown.

(3) Solving for Q :

$$Q = (h)(HR)(Ft^2)(T_1 - T_2)$$

$$Q = (4.16)(1)(1)(370^\circ) = 74\%$$

Then, percent of heat transfer by silver conduction

$$= \frac{9250}{9250 + 1670 + 89 + 1540} = 74\%$$

The unexpectedly large amount of silver found in the nodes gave rise to examination of a longitudinal cross-section of a brazed specimen. This cross-section showed that the silver brazing alloy filleted at the butt-joints of the honeycomb foil - brazing alloy interface as shown in Figure 5. The average height of the fillets was approximately 0.02" and presented cross-sectional areas as shown in Table III. It is believed that the filleting action of the silver brazing alloy will tend to increase the thermal conductance of a brazed panel by offering little resistance to heat flow along the effective height of the fillet. However, this effect was not included in the heat transfer calculations.

The conductivity of silver decreases with an increase in temperature and the conductivity of steel increases with an increase in temperature as shown in Figure 6. Since the silver is the better conductor and the steel is of greater volume, calculations show that the thermal conductivity of brazed alloy panels is essentially a constant value at various temperature drops with various amounts of silver in the cross-sectional area of the honeycomb core (Figure 7).

It is believed that the amount of silver in the node areas of the honeycomb will vary appreciably from panel to panel and, perhaps, even in the same panel. Since the thermal conductivity of a honeycomb panel will depend, to a large extent, upon the cross-sectional area of the silver at the midpoint of the thickness of the honeycomb, the thermal conductivity may vary appreciably from panel to panel. Figure 8 was prepared to show the change in thermal conductivity of brazed panels that would occur with a change of silver brazing alloy.

TABLE I

DATA FOR THERMAL CONDUCTIVITY OF A BRAZED SANDWICH PANEL AS DETERMINED EMPLOYING THE WATER CALORIMETER THERMAL CONDUCTIVITY APPARATUS

Run No.	Btu/Sq.Ft./Hr.	Hot Face Temperature, °F	Cold Face Temperature, °F	Thermal Conductivity, $\frac{\text{Btu-In.}}{\text{Ft}^2\text{-Hr-°F}}$
1	6060	600^a 575^b	226* 226*	8,80 9.25
2	4730	500^a 455^b	225* 225*	9.35 10.90
3	6400	600^a 570^b	226* 226*	9.25 9.90
4	6170	600^a 570^b	226* 226*	8.95 9.55
5	4550	500^a 480^b	225* 225*	8.98 9.15
6	2440	350^a 340^b	221* 221*	10.40 10.90
7	3270	400^a 385^b	223* 223*	9.82 10.72
8	2550	350^a 337^b	221* 221*	10.52 11.70

(a) **Ignitron Temperature Controller**
(b) Multichannel Recorder
* Corrected cold face temperatures (see Figure 3)

TABLE II

CROSS-SECTIONAL AREA OF SILVER BRAZING ALLOY IN HONEYCOMB CORE NODES AT MIDPOINT OF THICKNESS OF PANEL

Node Areas	Silver Triangular Joints	Area of Sivler in Triangles In.^{2}	Average Area of Silver in Triangles of Each Node Examined, In.^{2}
1	A	8.4×10^{-5}	11.7×10^{-5}
	B	15.0×10^{-5}	
2	A	9.6×10^{-5}	9.9×10^{-5}
	B	10.2×10^{-5}	
3	A	6.5×10^{-5}	5.8×10^{-5}
	B	4.8×10^{-5}	
4	A	7.2×10^{-5}	6.3×10^{-5}
	B	5.4×10^{-5}	
5	A	2.9×10^{-5}	7.0×10^{-5}
	B	11.1×10^{-5}	
6	A	12.8×10^{-5}	10.0×10^{-5}
	B	7.2×10^{-5}	
7	A	10.7×10^{-5}	9.0×10^{-5}
	B	7.2×10^{-5}	
8	A	13.2×10^{-5}	10.8×10^{-5}
	B	8.4×10^{-5}	
9	A	2.4×10^{-5}	8.4×10^{-5}
	B	14.4×10^{-5}	
10	A	10.4×10^{-5}	12.0×10^{-5}
	B	13.6×10^{-5}	
11	A	11.8×10^{-5}	12.4×10^{-5}
	B	13.0×10^{-5}	
12	A	7.2×10^{-5}	5.1×10^{-5}
	B	3.0×10^{-5}	
13	A	14.3×10^{-5}	10.7×10^{-5}
	B	7.0×10^{-5}	
14	A	17.7×10^{-5}	10.9×10^{-5}
	B	4.0×10^{-5}	
15	A	13.0×10^{-5}	12.2×10^{-5}
	B	11.4×10^{5}	

Average area of silver triangles in all nodes examined = 9.5×10^{-5}
A and B denote adjacent nodes. Section taken from approximately the middle of the core.

TABLE III

LONGITUDINAL CROSS-SECTIONAL AREA OF SILVER BRAZING ALLOY FILLET AFTER BRAZING

Node Area	Silver Triangular Joints	Area of Silver in Triangles In.^{2}	Average Area of Silver In Triangles of Each Node Examined In.^{2}
1	A	1.4×10^{-4}	1.5×10^{-4}
	B	1.6×10^{-4}	
2	A	1.4×10^{-4}	1.1×10^{-4}
	B	0.8×10^{-4}	
3	A	2.0×10^{-4}	1.5×10^{-4}
	B	1.0×10^{-4}	
4	A	1.6×10^{-4}	1.3×10^{-4}
	B	1.0×10^{-4}	
5	A	2.2×10^{-4}	1.6×10^{-4}
	B	0.9×10^{-4}	

Average area of silver triangles in all nodes examined (bottom side)
$= 1.4 \times 10^{-4}$

1	A	1.8×10^{-4}	0.7×10^{-4}
	B	0.6×10^{-4}	
2	A	0.4×10^{-4}	0.7×10^{-4}
	B	1.1×10^{-4}	
3	A	1.0×10^{-4}	0.8×10^{-4}
	B	0.5×10^{-4}	
4	A	0.5×10^{-4}	0.6×10^{-4}
	B	0.7×10^{-4}	
5	A	0.9×10^{-4}	1.0×10^{-4}
	B	1.0×10^{-4}	

Average area of silver triangles in all nodes examined (top side)
$= 0.7 \times 10^{4}$

A and B denote adjacent nodes
Average height of triangular fillet = 0.020"

FIGURE 1

CVC CALORIMETER TYPE THERMAL CONDUCTIVITY APPARATUS

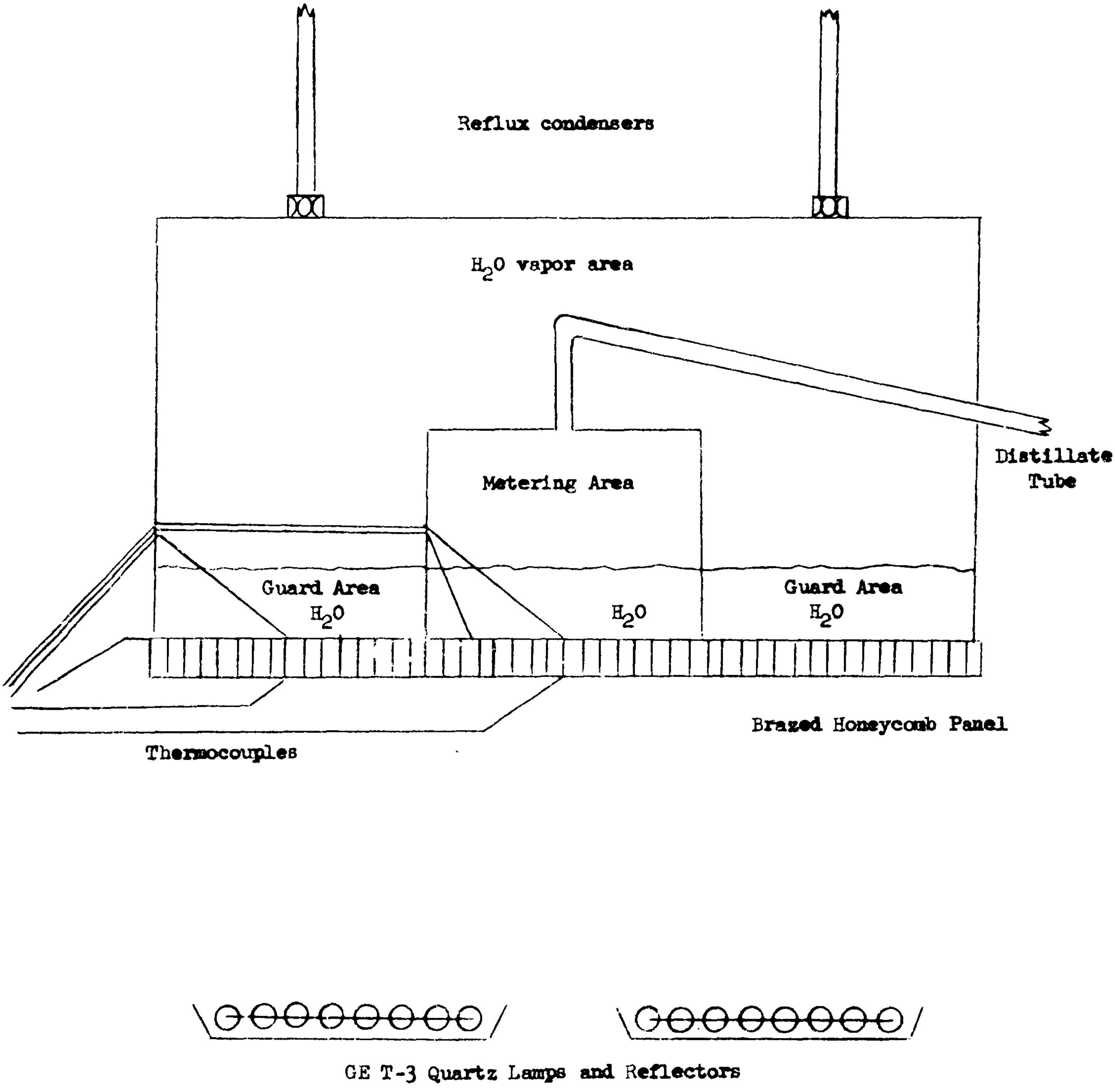
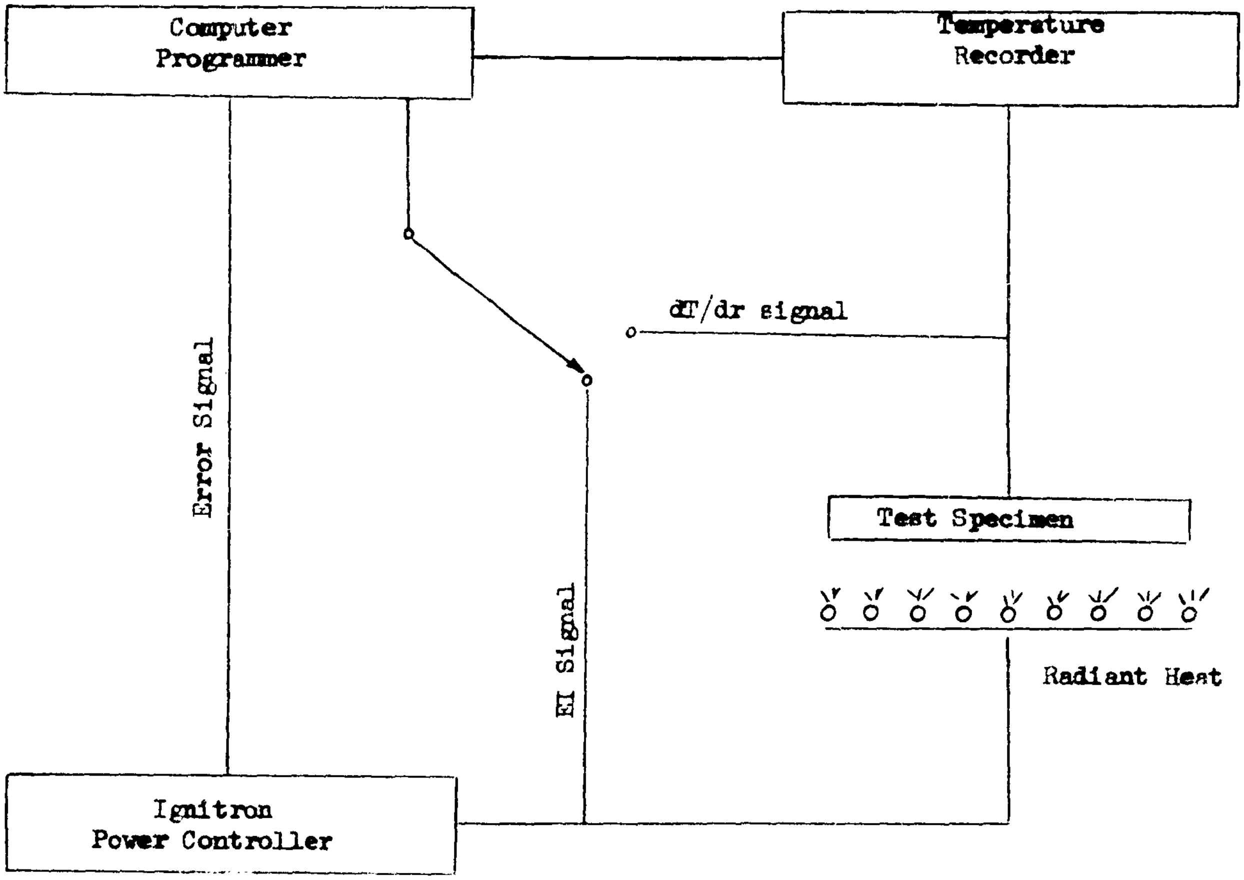


FIGURE 2

SCHEMATIC OF POWER CONTROL SYSTEM FOR RADIANTLY HEATING BRAZED HONEYCOMB PANEL



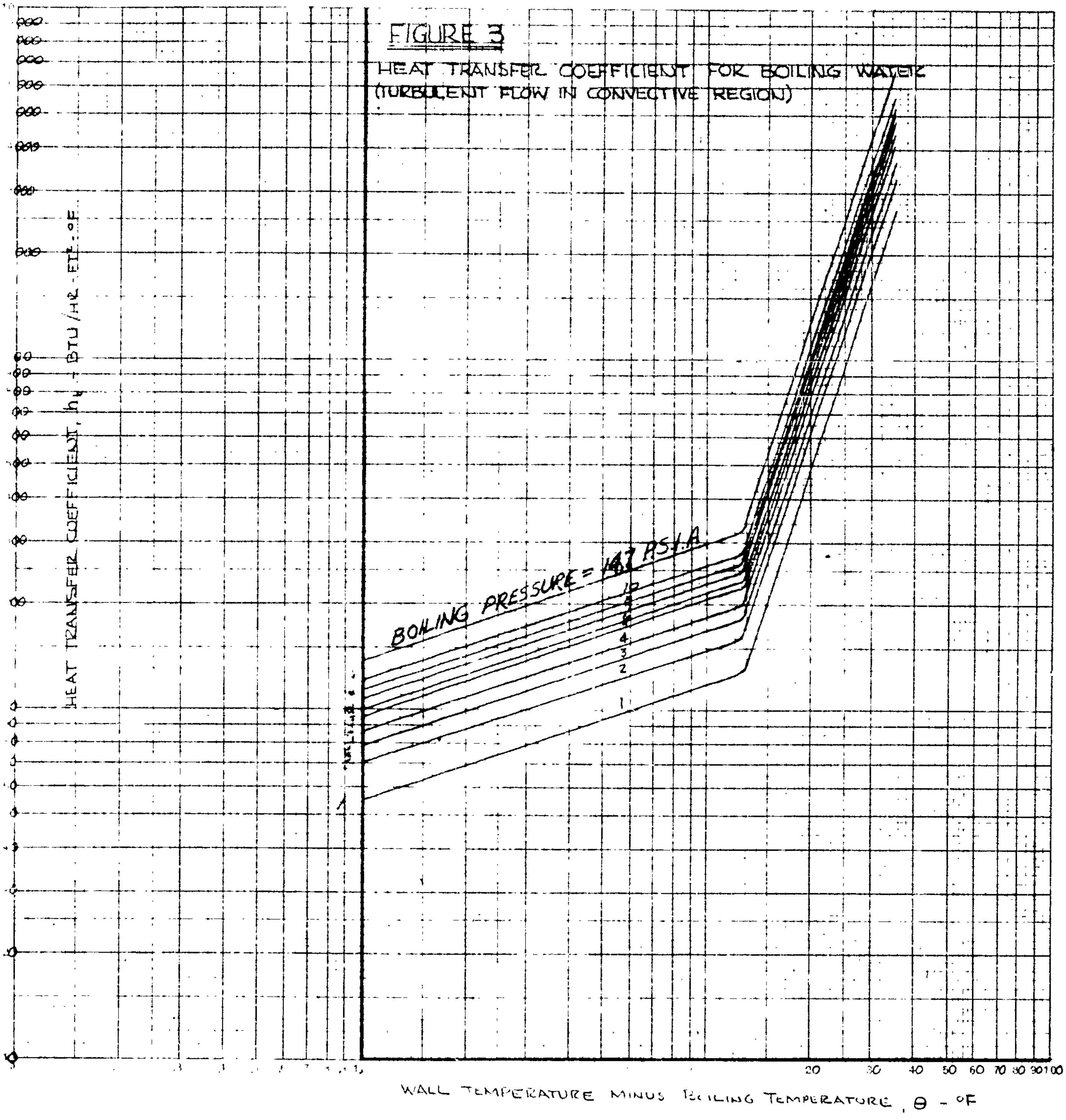
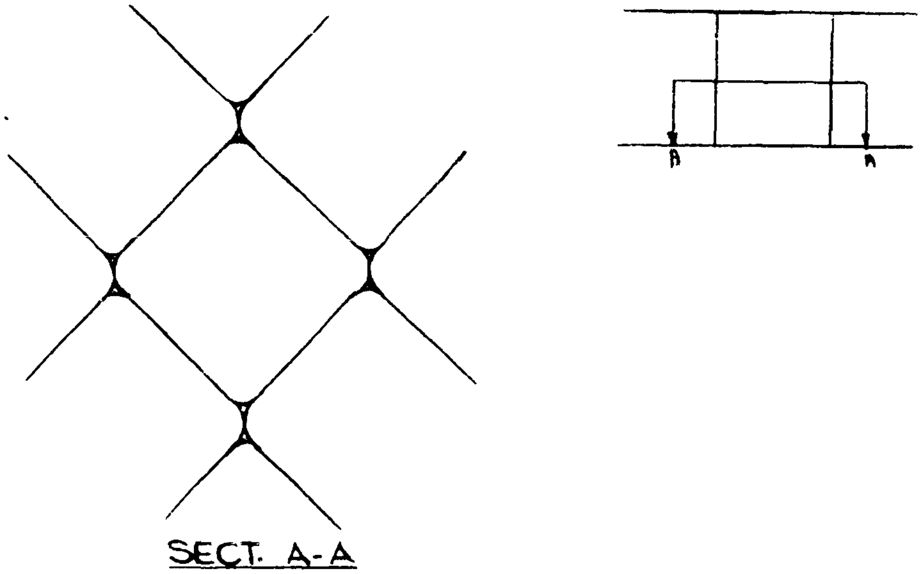


FIGURE 3

HEAT TRANSFER COEFFICIENT FOR BOILING WATER (TURBULENT FLOW IN CONVECTIVE REGION)

FIGURE 4

APPEARANCE OF MIDSECTION OF BRAZED HONEYCOMB CORE MATERIAL



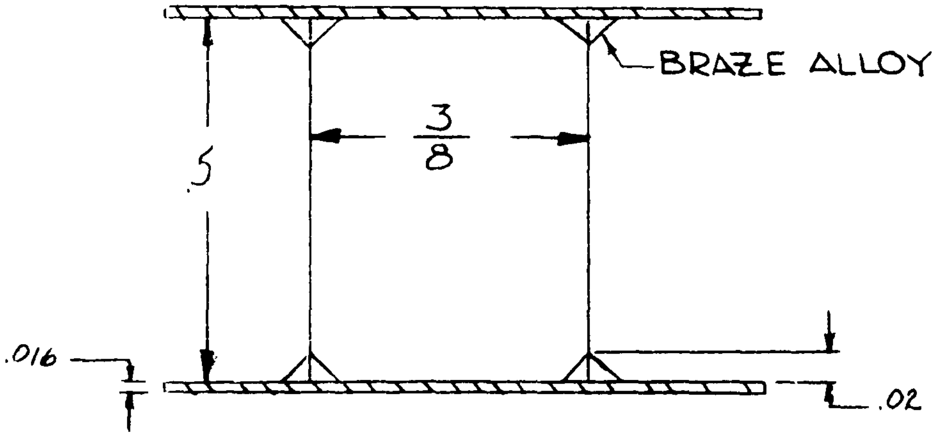


FIGURE 5

APPEARANCE OF LONGITUDINAL CROSS SECTION OF BRAZED HONEYCOMB SANDWICH PANEL

FIGURE 6

APPROXIMATE THERMAL CONDUCTIVITY OF SILVER BRAZING ALLOY AND PH 15-7Mo STAINLESS STEEL

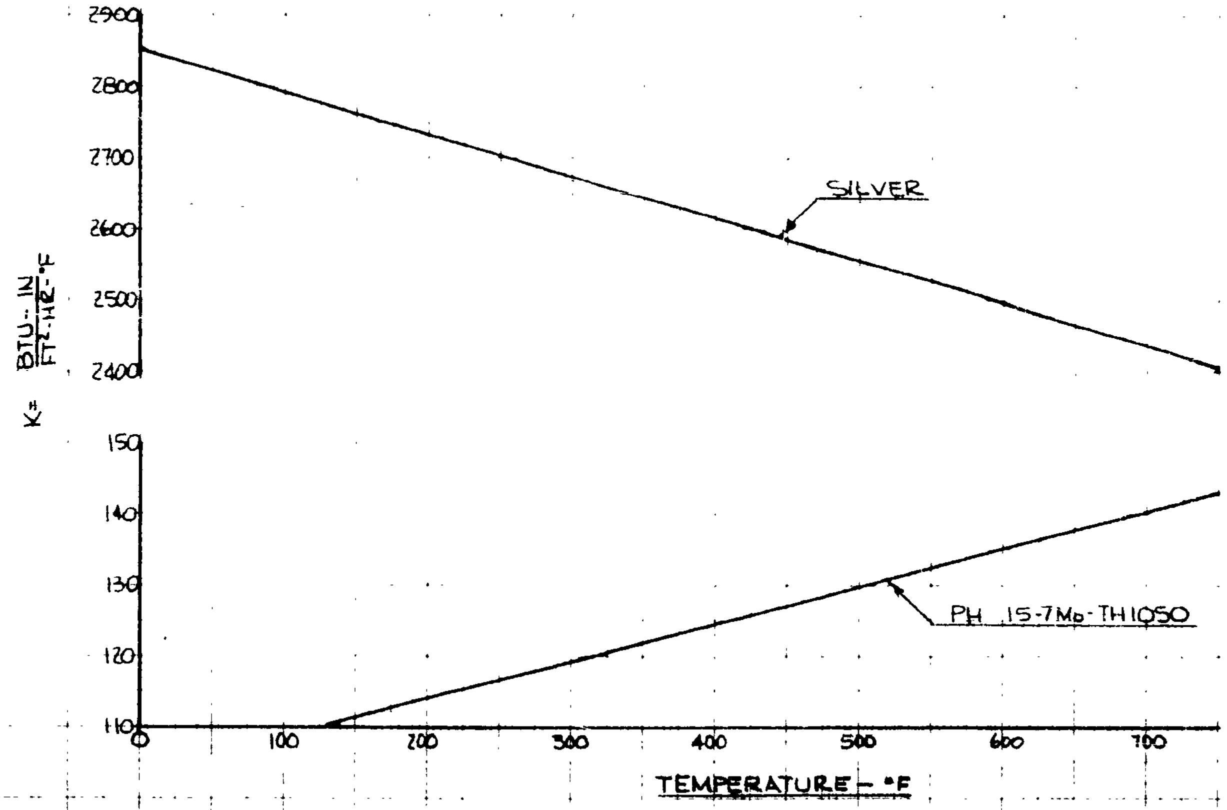


FIGURE 7

VARIATION IN CONDUCTIVITY WITH TEMPERATURE DROP
ACROSS BRAZED HONEYCOMB PANEL WITH VARIOUS AMTS
OF SILVER ALLOY IN THE X-SECTIONAL AREA OF THE NODES

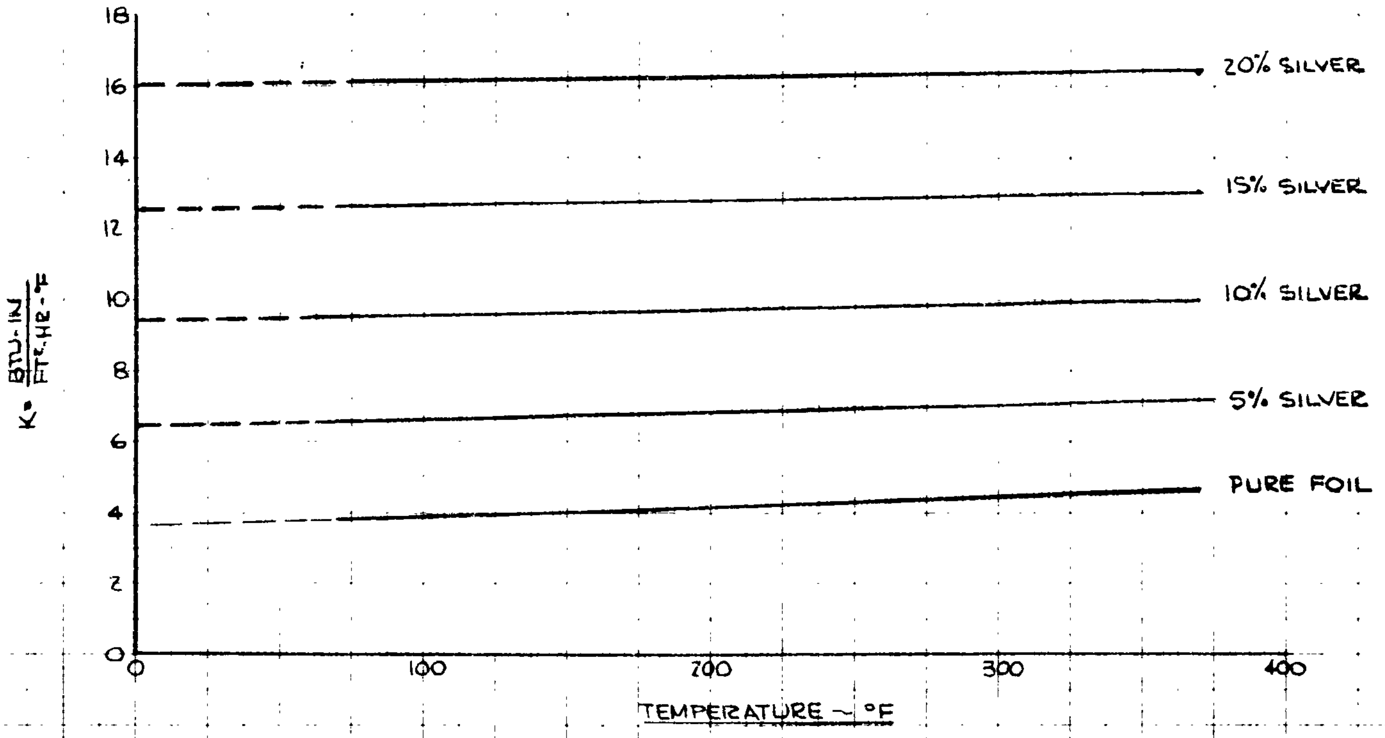
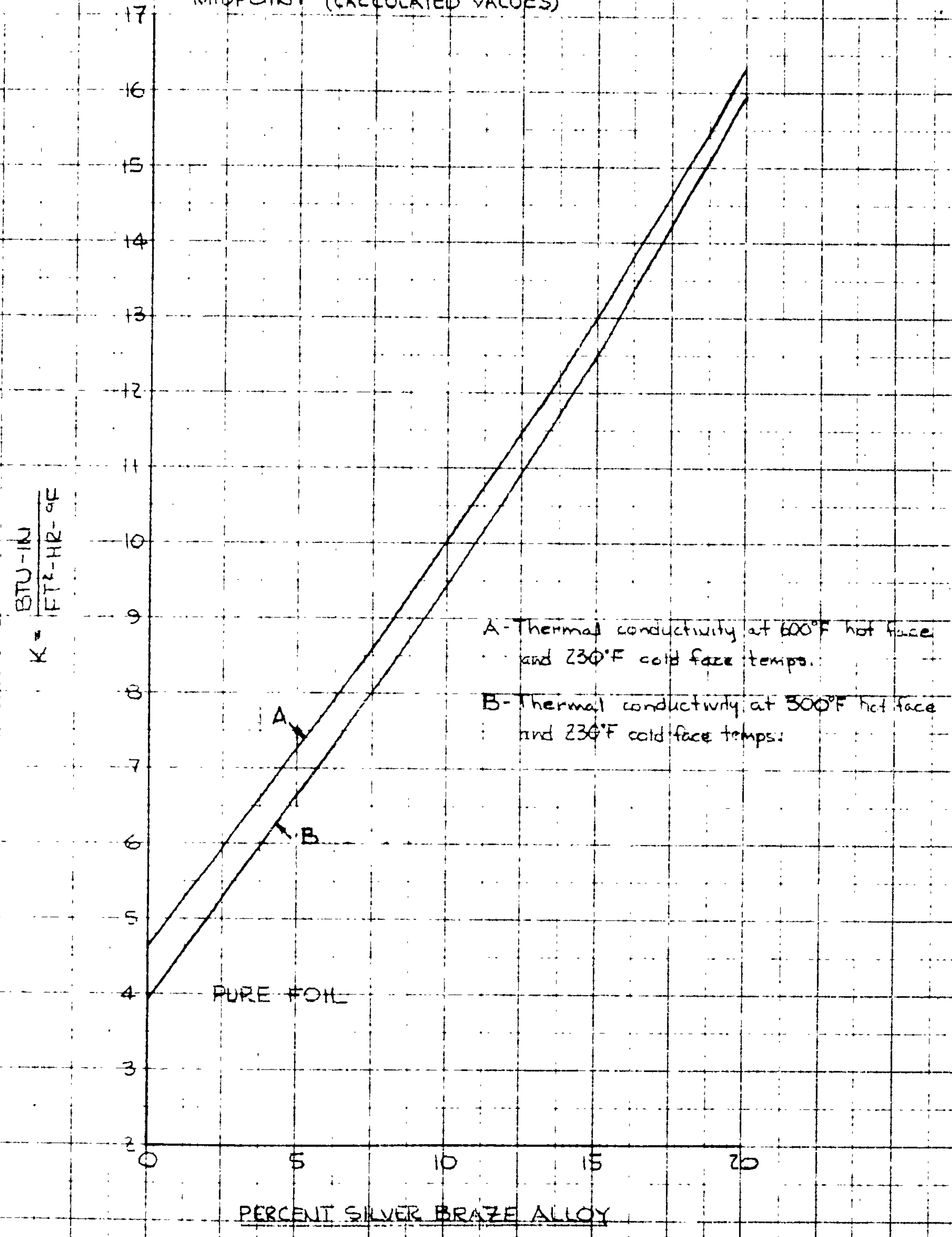


FIGURE 8

EFFECT OF THERMAL CONDUCTANCE WITH CHANGE OF SILVER BRAZING ALLOY IN THE CROSS-SECTIONAL AREA OF THE HONEYCOMB NODES AT THICKNESS MIDPOINT (CALCULATED VALUES)



A - Thermal conductivity at 600°F hot face and 230°F cold face temps.

B - Thermal conductivity at 300°F hot face and 230°F cold face temps.

CORROSION AND MATERIAL EVALUATION OF ALLOY STEEL SAMPLES SUPPLIED BY VANADIUM-ALLOYS STEEL COMPANY AND LATROBE STEEL COMPANY AS ALTERNATE MATERIALS FOR HYDRAULIC VALVES

INTRODUCTION

In an effort to alleviate the problems associated with manufacturing power control servo-valves, two steel companies submitted samples of tool steels for evaluation as possible replacements for the 440C steel alloy now commonly employed. The steels submitted were MGR punch and die steel, Heat No. 3761D; Olympic FM die steel, Heat No. 14908; Select B die steel, Heat No. 18088; Jet Forge tool steel, Heat No. 30181; Vasco 7152 tool steel, Heat No. 28852; and CK special tool steel, Heat No. 29660.

OBJECT

1. To determine corrosion resistance compared with 440C when tested according to military specifications MIL-E-5272A, Amendment 1.

2. To determine microstructure for indication of effect of heat treatment on mechanical properties.

CONCLUSIONS

1. The Jet Forge and Vasco 7152 steels exhibited corrosion properties comparable to AISI 440C. The corrosion properties of the other steels are not equivalent to those of 440C.

2. The precipitation of carbides in the grain boundaries of the two steels after heat treatment is not acceptable because of a reduction in the resistance of the material to intergranular corrosion.

MATERIAL AND SPECIMENS

Material	Heat Number
MGR punch and die steel	3761D
Olympic FM die steel	14908
Select B die steel	18088
Jet Forge tool steel	30181
Vasco 7152 Tool steel	28852
CK Special tool steel	29660

PROCEDURES

1. Three-inch lengths of the sample material were rough machined and heat treated to the 300,000 psi range (Rockwell 58-60) using procedures presented in Table I.

2. The heat treated samples were ground to a surface finish of RMS 32.

3. The samples were placed in a humidity chamber for 240 hours at a humidity of 95% and temperatures as set forth by paragraph 4.4.1 of military specification MIL-E-5272A, Amendment 1.

RESULTS

Table I presents the heat treatment used on the corrosion specimens. The composition of the tool steels submitted for evaluation is presented in Table II.

The samples of Vasco 7152 and Jet Forge steels appeared to have better corrosion resistance than any of the steels tested, and equal to that exhibited by AISI 440C. However, the microstructure of the steels after heat treatment is unsatisfactory because of the migration of carbides to the grain boundaries. Although the corrosion properties of the other steels were not equal to those of 440C, two of them did exhibit good microstructure after heat treatment. Both of these steels have a uniform martinsitic microstructure with well-distributed carbides and no evidence of grain boundary outlining.

TABLE I

HEAT TREATMENT USED ON TOOL STEEL CORROSION SPECIMENS

	MGR	OLYMPIC FM	SELECT B FM	JET FORGE	CK SPECIAL	VASCO 7152
Preheat	1250°F	1200-50°F	1200-1400°F	1550°F	1550°F	1550°F
Austenitize	1825-50°F	1825-50°F	1725-1750°F	1925°F	1850-75°F	1925-50°F
Time at Temp. 1 inch	1 hour	1 hour	1 hour	3/4 hour	3/4 hour	3/4 hour
Quench	Air	Air	Air	Air	Air	Air
Tempering Temperature	400°F	500°F	500°F	1000°F	350°F	925°F
Tempering Time	5 + 5 hr.	5 + 5 hr.	5 + 5 hr.	5 + 5 hr.	6 + 6 hr.	6 + 6 hr.

TABLE II

COMPOSITION OF TOOL STEELS SUBMITTED FOR EVALUATION

1	MGR	0.55C	5.00Cr	1.20 Mo	1.20 W	0.30 Mn	0.95 Si
2	Olympic FM	1.50C	12.00Cr	0.75 Mo	1.00 V	0.30 Mn	0.30 Si
3	Select B	1.00C	5.25Cr	1.10 Mo	0.25 V	0.70 Mn	0.30 Si
4	Vasco 7152	1.70C	17.50Cr				
5	Jet Forge	0.45C	8.00Cr				
6	CK Special	0.85C	10.75Cr				

ZIRCONIA MELTING POINT DETERMINATION

INTRODUCTION

One of the principal requirements of a material for nose cone application is a high melting point. The purpose of this evaluation is to determine the melting point of a series of zirconia and zircon materials.

Twelve zirconia compounds and two zircon compounds were submitted to the Structures Materials Laboratory for evaluation in the 40 KW Plasma Arc Facility.

OBJECT

To determine the melting point of several zirconia and zircon materials.

CONCLUSION

The melting point of fourteen materials was determined and relative emissivity values were calculated at the melting point. The melting point range of the zirconia materials, was 4580 to 4800°F.

MATERIALS

The zirconia materials listed in Table I were purchased from Zirconium Corporation of America and the Astroceram A and B materials were received from Instrumentation Associates.

PROCEDURE

1. All specimens were tested in duplicate except the 8% yttria, 4% lime and the Astroceram A and B cements.

2. The 0.5 inch diameter x 1.0 inch long specimens were mounted perpendicular to the 40 KW plasma flame and held in position with a zirconia brick.

3. The temperature was measured using both the radiation and optical pyrometers. The pyrometers were mounted as a single unit to allow the instruments to be focused on approximately the same area.

4. The torch was moved in at intervals of 0.06 inches when the temperature approached the melting point.

5. The melting point was defined as the flow of molten material at the sample surface as observed through an optical pyrometer.

6. The relative emissivity was determined by:

   (a) Measuring the temperature difference between the optical and radiation pyrometers.

   (b) Reading the emissivity off the parametric graph which was derived from the emissivity correction data for each of the instruments.

RESULTS

1. The results of the proceeding test are tabulated in Table I.

TABLE I

MELTING POINT DATA OF ZIRCONIA AND ZIRCON MATERIALS

SPECIMEN NUMBER	SAMPLE DESCRIPTION	MELTING POINT - °F		EMISSIVITY
		OPTICAL	RADIATION	
1	Zirconia +3% CaO - Coarse Molded	4720	4450	0.74
2	Zirconia +3% CaO - Coarse Molded	4720	4400	0.70
3	Zirconia +4% CaO - Coarse Molded	4580	4190	0.64
4	Zirconia +4% CaO - Coarse Molded	4640	4400	0.77
5	Zirconia +4% CaO - Coarse Extruded	4660	4400	0.79
6	Zirconia +4% CaO - Coarse Extruded	4640	4300	0.69
7	Zirconia +4% CaO - Fine Molded	4660	4225	0.62
8	Zirconia +4% CaO - Fine Molded	4680	4475	0.80
9	Zirconia +5% CaO - Coarse Molded	4660	4450	0.80
10	Zirconia +5% CaO - Coarse Molded	4640	4400	0.78
11	Zirconia +4% CaO +3% CeO_2 - Coarse Molded	4680	4430	0.76
12	Zirconia +4% CaO +3% CeO_2 - Coarse Molded	4680	4350	0.69
13	Zirconia +4% CaO - Cement Molded	4680	4350	0.66
14	Zirconia +3% Y_2O_3 - Coarse Molded	4760	4400	0.67
15	Zirconia +3% Y_2O_3 - Coarse Molded	4760	4425	0.69
16	Zirconia +8% Y_2O_3 - Coarse Extruded	4740	*	-
17	Zirconia +8% Y_2O_3 - Coarse Extruded	4780	*	-
18	Zirconia +12% Y_2O_3 - Coarse Molded	4800	4325	0.60
19	Zirconia +12% Y_2O_3 - Coarse Molded	4800	4475	0.70
20	Zirconia +8% Y_2O_3+3% CeO - Coarse Molded	4760	*	-
21	Zirconia +8% Y_2O_3 +3% CeO - Coarse Molded	4780	*	-
22	Zirconia +8% Y_2O_3 - Cement Molded	4780	4600	0.82
23	Astroceram A - Cement	3820	3600	0.74
24	Astroceram B - Cement	3840	3625	0.75

* Nozzle of torch blanked off radiation pyrometer

PHYSICAL PROPERTY TESTS ON SINTERED ZIRCONIA

INTRODUCTION

To better understand the behavior of sintered zirconia at elevated temperatures, the following properties need be obtained: apparent porosity, impervious volume, and bulk density. This data should be made available so that a correlation can be made with thermal response and physical strength data.

Ultimately, parameters established on test data and physical property measurements would enable design of thermal insulation systems.

OBJECT

To determine the physical properties of zircoa sintered zirconia.

CONCLUSIONS

The apparent porosity, impervious volume, and bulk density were successfully determined for 20 zirconia materials.

MATERIALS

All materials measured were obtained from Zircoa Corporation of America, Solon, Ohio. See Table I for tabulation of materials examined in this survey.

PROCEDURE

The procedures followed in this test are as outlined in reference (a). A condensed outline of this procedure is as follows:

A. Dry Weight, $\underline{D}$. Weight all specimen initially.

B. Impregnation with water. Boil weighed specimens in distilled water for 5 hours. Allow to cool and soak for an additional 24 hours.

C. Suspend Weight, $\underline{S}$. Weight specimens immersed in water to obtain saturated displacement weight.

D. Saturated Weight, $\underline{W}$. Blot specimens lightly with a damp cloth to remove excess water from surface and weigh.

E. Calculations were:

1. Exterior Volume, $\underline{V}$ cu. cm.

$$V = W - S$$

2. Volume of open pores and impervious portion

Volume of open pores, cu. cm. $= W - D$
Volume of impervious portion, cu. cm. $= D - S$

3. Apparent Porosity, P .

$$P = \frac{W - D}{V} \times 100 = \%$$

4. Water Absorption, A .

$$A = \frac{W - D}{D} \times 100 = \%$$

5. Apparent Specific Gravity, T .

$$T = \frac{D}{D - S} \text{ gm/cu. cm.}$$

6. Bulk Density, B .

$$B = \frac{D}{V} \text{ gms/cu. cm.}$$

Conversion to lbs./cu. in. = 0.036 x gm/cu. cm.

RESULTS

1. Twenty of the 26 designated materials were measured by the ASTM procedure.
2. All physical property data are tabulated in Table I.

TABLE I

PHYSICAL PROPERTY MEA

Specimen No.	Description		Texture	Consistency	Dry Weight D (gm)	Immersed Weight S (gm)	Saturated Weight W (gm)	Ex Vo V
	Stabilization	Fabrication						
1 a	5% CaO	Molded	Coarse	Homogeneous	15.05	12.32	15.84	3
b	4% CaO	Molded	Coarse	Homogeneous	17.06	14.08	17.74	3
c	4% CaO	Extruded	Fine	Homogeneous	12.21	9.89	12.22	2
d	4% CaO	Extruded	Coarse	Homogeneous	17.78	14.67	18.53	3
e	3% CaO	Molded	Coarse	Heterogeneous	16.49	13.59	17.13	3
f	12% Y_2O_3	Molded	Coarse	Homogeneous	14.64	12.27	15.71	3
h	8% Y_2O_3	Extruded	Coarse	Homogeneous	5.83	4.84	6.21	1
i	3% Y_2O_3	Molded	Coarse	Heterogeneous	15.80	13.09	16.53	3
k	8% Y_2O_3	Cement			33.93	28.12	37.27	9.
l	4% CaO	Cement			32.26	26.53	34.53	8.
m	8% Y_2O_3	Extruded	Fine	Homogeneous	5.80	4.66	5.81	1.
2 a	5% CaO	Molded	Fine	Homogeneous	18.30	14.64	18.32	3.
b	5% CaO	Extruded	Fine	Homogeneous	5.43	4.40	5.44	1.
d	4% CaO	Extruded	Coarse	Homogeneous	10.78	8.90	11.26	2.
e	4$ CaO	Molded	Fine	Homogeneous	21.24	17.48	21.26	3.
f	3% CaO	Extruded	Coarse	Homogeneous	7.35	6.13	7.59	1.
g	12% Y_2O_3	Extruded	Fine	Homogeneous	7.29	6.05	7.54	1.
i	3% Y_2O_3	Extruded	Coarse	Heterogeneous	6.73	5.58	7.07	1.
j	4% CaO, 3% CaO_2	Molded	Coarse	Homogeneous	11.59	9.66	12.45	2.
m	8% Y_2O_3 4% CaO_2	Molded	Coarse	Homogeneous	17.09	14.21	17.93	3.

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TABLE I
PROPERTY MEASUREMENTS & DATA

sed t a)	Saturated Weight W (gm)	Exterior Volume V = W-S	Volume of Open Pores = W-D	Volume of Impervious Portions = D-S	Apparent Porosity P = $\frac{W-D}{V}$ x 100	Void Volume A = $\frac{W-D}{D}$ x 100	Apparent Specific Gravity T = $\frac{D}{D-S}$	Bulk Density B = $\frac{D}{V}$ $\frac{gm}{cm^3}$	lbs./in.^{3}
	15.84	3.52	0.79	2.73	22.40	5.25	5.51	4.28	.154
	17.74	3.66	0.68	2.98	18.58	3.99	5.72	4.66	.168
	12.22	2.33	0.01	2.32	0.43	0.08	5.26	5.24	.188
	18.53	3.86	0.75	3.11	19.43	4.22	5.72	4.61	.166
	17.13	3.54	0.64	2.90	18.08	3.88	5.69	4.66	.168
	15.71	3.44	1.07	2.37	31.2	7.30	6.19	4.28	.154
	6.21	1.37	0.38	0.99	27.74	6.52	5.89	4.26	.154
	16.53	3.44	0.73	2.71	21.22	4.62	5.83	4.59	.165
	37.27	9.15	3.34	5.81	36.50	9.84	5.84	3.71	.134
	34.53	8.00	2.27	5.73	28.38	7.04	5.63	4.03	.145
	5.81	1.15	0.01	1.14	0.87	0.17	5.09	5.04	.182
	18.32	3.68	0.02	3.66	0.54	0.11	5.00	4.97	.179
	5.44	1.04	0.01	1.03	0.96	0.18	5.27	5.22	.188
	11.26	2.36	0.48	1.88	20.33	4.45	5.73	4.57	.165
	21.26	3.78	0.02	3.76	0.53	0.09	5.65	5.62	.203
	7.59	1.46	0.24	1.22	16.41	3.27	5.92	5.02	.181
	7.54	1.49	0.30	1.24	20.13	4.96	5.88	4.89	.176
	7.07	1.49	0.34	1.15	22.82	6.09	5.85	4.52	.162
	12.45	2.77	0.86	1.93	31.0	7.42	6.00	4.18	.150
	17.93	3.72	0.84	2.88	22.58	4.92	5.93	4.59	.167