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RESEARCH AND DEVELOPMENT DIVISION  
FOR THE AIR FORCE

RESEARCH AND DEVELOPMENT DIVISION

**HIGH-TEMPERATURE PROTECTIVE  
RE-ENTRY COATINGS FOR  
EXPANDABLE STRUCTURES**

**M. T. Conger**

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## FOREWORD


This report was prepared by The Goodyear Tire & Rubber Company in compliance with Contract No. AF 33(657)-11257. The contract was initiated under Project No. 7320, "Fibrous Materials for Decelerators and Structures," Task No. 732002, "Fibrous Structural Materials," and was administered by the Air Force Materials Laboratory, Research and Technology Division, Air Force Systems Command. J. J. Mattice of the Elastomers and Coatings Branch acted as project engineer.

Hot-stage microscopic studies, thermogravimetric analyses, differential thermal analyses, and adhesion studies were performed by Battelle Memorial Institute, a principal subcontractor. Battelle Memorial Institute personnel participating were M. J. Snyder and W. A. Hedden, with assistance from other specialized groups in the Battelle Laboratories of Columbus, Ohio.

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This technical documentary report has been reviewed and is approved.

  
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Elastomers and Coatings Branch  
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## ABSTRACT

This report describes the accomplishments of the second year of a two-year program, the object of which has been the development of a protective coating for use on an expandable (inflatable) re-entry structure. It is envisaged that the re-entry structure will be fabricated from a high-strength metal-fiber fabric coated with a flexible, rubber-like coating. The coating and its application to the metal fabric have been the objects of investigations under this program.

Improved apparatus and procedures were developed for testing of coated metal fabrics under simulated re-entry conditions. Coatings of three general types were tested: (1) wholly inorganic, (2) silicone rubber with fusible filler, and (3) ablative organic insulation with fusible filler.

The silicone rubber (S2077) - glass frit (AW-35 and/or 3520) coatings were found most effective and performed satisfactorily over a range of coating weights and rubber to frit ratios when heated to 2000°F in a stagnant nitrogen atmosphere. The more severe condition of heating in air required alternate coats of silicone rubber - AW-35 frit and silicone rubber - 3520 frit to a total of 6 - 8 coats. Neither coating alone could prevent excessive leakage at 2000°F in air.

**TABLE OF CONTENTS**

Section	Page
1 INTRODUCTION . . . . .	1
2 SUMMARY . . . . .	3
A. Experimental Procedures . . . . .	3
B. Inorganic Coatings . . . . .	3
C. Silicone Rubber Coatings . . . . .	3
D. Nitrile Rubber - Phenolic Resin Coatings . . . . .	5
3 TEST APPARATUS . . . . .	7
A. General . . . . .	7
B. Apparatus Design . . . . .	7
C. Apparatus Testing . . . . .	10
4 SPECIMEN PREPARATION . . . . .	13
A. General . . . . .	13
B. Preparation of Metal-Fabric Cylinders . . . . .	13
C. Inorganic Coatings . . . . .	14
D. Silicone Rubber Coatings . . . . .	14
E. Nitrile Rubber - Phenolic Resin Compositions . . . . .	14
5 TESTING COATED CYLINDERS . . . . .	17
A. General . . . . .	17
B. Procedure 1 . . . . .	17
C. Procedure 2 . . . . .	17
D. Procedure 3 . . . . .	17
E. Procedure 4 . . . . .	17
F. Procedure 5 . . . . .	17
G. Procedure 6 . . . . .	17
H. Procedure 7 . . . . .	18
I. Procedure 8 . . . . .	18
6 TEST RESULTS AND DISCUSSION . . . . .	19
A. Uncoated Specimens . . . . .	19
B. Inorganic Coatings . . . . .	19
C. Silicone Rubber Coatings . . . . .	24
D. Nitrile Rubber - Phenolic Resin Coatings . . . . .	47
7 CONCLUSIONS AND RECOMMENDATIONS . . . . .	53
<b>Appendix</b>	
I FINE WIRE, MULTIFILAMENT YARN, METAL FABRIC . . . . .	55
II MATERIALS IDENTIFICATION . . . . .	59

LIST OF ILLUSTRATIONS

Figure		Page
1	Coating of 50 Percent S2077 Solids and 50 Percent AW-35 Frit after Testing in Nitrogen (8X Magnification) . . . . .	4
2	Coating of 50 Percent S2077 Solids and 50 Percent AW-35 Frit after Testing in Air (8X Magnification) . . . . .	4
3	Test Apparatus . . . . .	8
4	Test Chamber . . . . .	8
5	Heating Rate . . . . .	11
6	Temperature Profile of Stainless Steel Tube . . . . .	11
7	Uncoated Specimen . . . . .	14
8	Inorganic Coated Specimen before Test . . . . .	15
9	Silicone Rubber Coated Specimen before Test . . . . .	15
10	Nitrile Rubber - Phenolic Resin Coated Specimen before Test . . . . .	15
11	Inorganic Coated Specimen after Test . . . . .	22
12	Inorganic Coated Specimen after 30-Minute Heat Cycle . . . . .	22
13	TGA Plots for Coating R70X48-1 . . . . .	35
14	DTA Plots for Coating R70X48-1 . . . . .	35
15	TGA Plots for S2077 Rubber . . . . .	36
16	DTA Plots for S2077 Rubber . . . . .	37
17	Pyrolysis Residue of Silicone Rubber Coating . . . . .	37
18	Exploded View of Vacuum-Heating Stage . . . . .	40
19	Coating R70X48-1 on Chromel A Wire Cloth after Heating in Air and Nitrogen Atmospheres . . . . .	41
20	Typical Nitrile Rubber - Phenolic Resin Coated Specimen after Test . . . . .	46
21	Nitrile Rubber - Phenolic Resin Coated Specimen after 30-Minute Heat Cycle . . . . .	47

LIST OF TABLES

Table		Page
I	S2077 Silicone Rubber - Glass Frit Coatings on Stainless Steel Screen (Tested in Nitrogen) . . . . .	5
II	Alternate Coatings on Nichrome V Cloth (Tested in Air) . . . . .	5
III	Nitrile Rubber Elastomeric Ablative Insulation . . . . .	6
IV	Results of Tests on Inorganic Coatings . . . . .	20
V	Effect of Saturated Salt Solutions on Relative Humidity . . . . .	23
VI	Effect of Moisture Content on Leak Temperature . . . . .	24
VII	Sodium Silicate Coatings (Specimen Ends Protected) . . . . .	25
VIII	Sodium Silicate Rubber Coatings . . . . .	25
IX	Results of Tests on Silicone Rubber Coatings . . . . .	26
X	Summary of Data on Components and Coatings . . . . .	38
XI	S2077 Silicone Rubber - Glass Frit Coatings Tested in Nitrogen . . . . .	42
XII	S2077 Silicone Rubber - Glass Frit Coatings Tested in Air . . . . .	43
XIII	Mixtures of Frits Tested in Air . . . . .	43
XIV	Dual Coatings Tested in Air . . . . .	44
XV	Alternate Coatings Tested in Air . . . . .	45
XVI	Comparison of Stainless Steel Screen Specimens with Nichrome V Specimens . . . . .	45
XVII	Composition of Coatings and Evaluation after Heating in Air or Nitrogen . . . . .	48
XVIII	Nitrile Rubber - Phenolic Resin-Glass Frit Coatings . . . . .	50

## SECTION 1

### INTRODUCTION

A two-year program, which began 15 June 1963, has been conducted by The Goodyear Tire & Rubber Company to develop a coating for a metal-fiber fabric. The coated fabric is to be used to fabricate an expandable structure capable of enduring atmospheric re-entry during which a peak skin temperature of 2000°F will be encountered. The principal structural material used in the fabrication of this structure will be the coated metal-fiber fabric composite. The coating portion of this composite and the application of the coating to the metal-fiber are the objectives of this investigation. The development of the metal fabric and the design and fabrication of re-entry structures are outside the scope of this program.

During the first year, an investigation was made of inorganic coatings, chemical vapor-deposited coatings, coatings containing char-forming ablative insulation compounds with fusible inorganic fillers, coatings containing silicone rubber with fusible inorganic filler, and newly discovered polymers (literature survey only). This work was reported in ML-TDR-64-213, dated July 1964.

This report covers the second year of the program. The major task was to optimize the composition of the best coating types to achieve a coated metal fabric composite that is sufficiently flexible and "tough" to resist damage during preflight handling and storage and that has the other characteristics required for re-entry structure use.

In order to evaluate the coatings, a new method of laboratory testing was developed. The apparatus and information relative to its operation are described. Approximately 200 specimens were evaluated using this equipment. These specimens and the test results are described in this report.

## SECTION 2

### SUMMARY

#### A. EXPERIMENTAL PROCEDURES

Experimental coatings were tested to determine if they were able to contain helium when heated to re-entry temperatures in air and/or nitrogen atmospheres. In this test the coating was the "sealer" for a 10-inch-long by 3/8-inch-diameter cylinder made from woven metal-fiber fabric. Experimental coatings were applied to the cylinders by brushing.

Each coated cylinder was pressurized to 1 psig with helium while its outside surface was exposed to circulating nitrogen or air at 0 psig. The central portion (about 1 inch) of the cylinder was heated to 2000°F. While the cylinder was heated, the loss of helium was measured. If the helium loss rate exceeded 2 cc/second/square inch (the goal set for this program), the temperature at which this occurred was recorded.

Significant differences were noted between specimens tested in nitrogen and those tested in air, as illustrated in Figures 1 and 2. In an effort to determine the cause of these differences, the behavior of the coatings and various coating components was observed both directly and indirectly, using (1) differential thermal analyses, (2) thermogravimetric analyses, (3) a "Multiple-Unit" decomposition furnace, and (4) hot-stage microscopic equipment.

#### B. INORGANIC COATINGS

A number of inorganic coatings, all of which contained substantial amounts of sodium silicate, were tested because promising results had been obtained from these coatings during the first year. However, during a simulated re-entry heating cycle, it was found that these coatings failed at about the boiling point of water.

Investigation revealed that the coatings failed because moisture evaporated from the heated portion of the specimen and condensed on the cooler portions of the specimen, causing the coating to be liquefied. Failure was due to the inability of the liquefied coating to withstand the force exerted by helium.

Further investigation revealed that coatings utilizing 2 : 1  $\text{SiO}_2 + \text{Na}_2\text{O}$  sodium silicate become too stiff when dried at 30 percent relative humidity and become fluid from high equilibrium water content when dried at 80 percent relative humidity. When in equilibrium with 50 percent relative humidity, the sodium silicate has a water content of about 30 percent and the coatings have a reasonable degree of flexibility in this condition.

No modification of the composition of sodium silicate based coatings was discovered that produced a coating that is both flexible and resistant to re-entry heat.

#### C. SILICONE RUBBER COATINGS

Coatings deposited from S2077 silicone rubber solutions containing AW-35 glass frit and/or 3520 glass frit are capable of enduring simulated re-entry tests wherein temperatures of 2000°F are reached in a nitrogen atmosphere. The coatings are effective helium diffusion barriers under these conditions at S2077/frit ratios of 0.8 to 4 and coating weights between 12 and 24 oz/sq yd. All of the specimens tested having coatings of this type were found to have maximum leakage rates well below the goal of 2 cc/second/square inch, as shown in Table 1.

Although coatings of S2077 and glass frit performed very well when tested in nitrogen, neither the frit alone nor combinations of these frits in a single coating gave the results desired when tested in air. In air, the coatings containing AW-35 frit became excessively fluid and those containing the 3520 frit developed "mud" cracks. It was observed, however, that by

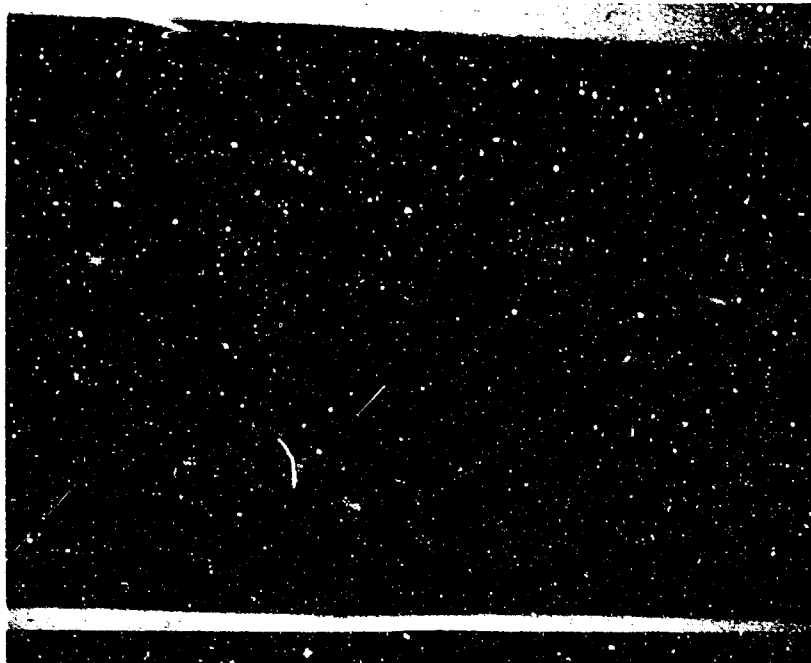


Figure 1. Coating of 50 Percent S2077 Solids and  
50 Percent AW-35 Frit after Testing in  
Nitrogen (8X Magnification)



Figure 2. Coating of 50 Percent S2077 Solids and  
50 Percent AW-35 Frit after Testing in  
Air (8X Magnification)

Table I. S2077 Silicone Rubber - Glass Frit Coatings on Stainless Steel Screen (Tested in Nitrogen)

Glass Frit	S2077-Frit Ratio <sup>a</sup>	Coating Weight (oz/yd <sup>2</sup> )	Maximum Leak Rate <sup>b</sup> (cc/sec)
AW-35	1/1	12.24	0.318
AW-35	1/1	15.64	0.119
AW-35	1/1	17.32	0.064
AW-35	1/1	21.60	0.05
AW-35	4/1	13.20	1.45
AW-35	3/1	13.84	0.60
AW-35	8/1	12.52	0.222
3520	1/1	12.24	0.318
3520	1/1	18.12	0.119
3520	1/1	22.92	0.29
3520	8/1	12.52	0.222

<sup>a</sup>Weight ratio of S2077 solids to frit.

<sup>b</sup>Specimen area is 11.8 square inches, but only about 10 percent is heated to full 2000°F.

Table II. Alternate Coatings on Nichrome V Cloth (Tested in Air)

Specimen No.	Coating Weight (oz/yd <sup>2</sup> )	First Leak Temp (°F)	Maximum Leak Rate (cc/sec)
3379-42-181	22.52	1880	0.66
3379-42-187	12.40	1660	4.28 <sup>a</sup>
3379-42-188	12.36	1810	0.41 <sup>a</sup>
3379-42-189	16.48	1940	0.51
3379-42-190	16.96	1835	0.58
3379-42-191	21.48	2000	0.37

<sup>a</sup>Specimen showed slight leak after folding; leak patched before testing.

applying multiple coatings of the two frits in separate layers, the desired results could be obtained. The results of these tests are shown in Table II.

#### D. NITRILE RUBBER - PHENOLIC RESIN COATINGS

Elastomeric ablative insulation will offer special advantages if it delays the time at which the peak skin temperature is reached and/or lowers the peak skin temperature. Such compositions can be made to have high leak temperatures, as shown by the data in Table III. The high leak temperatures were achieved by the addition of very large amounts of glass frits. Unfortunately, this causes the coatings to be very stiff and very susceptible to damage, even with careful handling.

Table III. Nitrile Rubber Elastomeric Ablative Insulation

Description	Coating Weight (oz/yd <sup>2</sup> )	Leak Temp (°F)	Maximum Leak Rate (cc/sec)
Containing 75% frit	11.6	>2000	0.13
Containing 50% frit	14.9	940	> 7
Containing 25% frit	12.1	900	> 7

## SECTION 3

### TEST APPARATUS

#### A. GENERAL

The test apparatus was designed to determine the ability of the coated fabric to perform satisfactorily as the gas envelope of an inflatable re-entry structure. The general requirement is that the coated fabric remain relatively leak-free under all thermal, mechanical, and chemical stresses of re-entry. While other properties are also important, some of the essential coated fabric requirements are as follows:

- (1) Flexibility sufficient to withstand the folding and creasing of packaging and deployment and strength sufficient to withstand the variable tension loads of re-entry.
- (2) Heat resistance sufficient to prevent failure due to the re-entry heat. For the purpose of this research program, the maximum skin temperature is assumed to increase linearly from room temperature to 2000°F in 12-1/2 minutes, remain for 5 minutes at 2000°F, followed by linear cooling to room temperature in 12-1/2 minutes.
- (3) Leak resistance under varying tensile loads from 0 to 50 percent of fabric strength. (Fabric strength is the breaking strength at 2000°F after folding of the specimen.)

The apparatus was designed so that these properties could be evaluated, and provisions were made to determine leakage during torsional stress and to attain a heating rate capable of reaching 2000°F in 2-1/2 minutes.

Consideration of the performance and test requirements led to selection of a test specimen in the form of a slender cylinder of coated metal fabric.

#### B. APPARATUS DESIGN

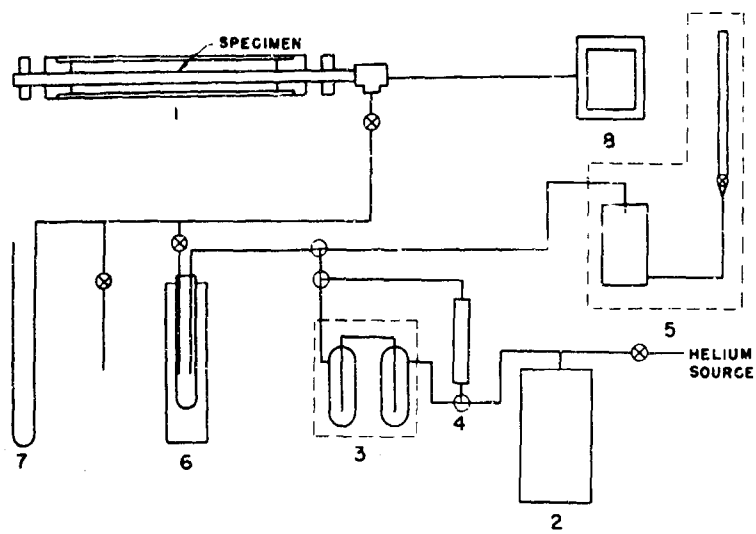
##### 1. General

The apparatus (Figures 3 and 4) comprises a test chamber, helium source, helium reservoir, flow indicator, rotameter, leakage tester, water manometer, liquid nitrogen cold trap, and temperature recorder. The heat is supplied by two oxygen-acetylene torches with No. 8 Oxweld tips. The torches are mounted in movable holders that can be continuously and simultaneously relocated with respect to the specimen chamber (for simulating the variable heat flux of re-entry).

##### 2. Test Chamber

The test chamber is the portion of the equipment that surrounds the test specimen. It is made from one-inch stainless steel pipe and has capped ends drilled 7/16 inch in diameter. A gas inlet connection is provided to allow the introduction of nitrogen gas or other selected gases to the area surrounding the test specimen. These gases escape easily from the chamber through the oversize (with respect to the diameter of the test specimen inserted in them) holes in the end caps, and the pressure of the gas in the chamber is not measurably above atmospheric pressure.

The chamber is mounted on a stand that is equipped with clamps to hold the specimen. Provisions are made for applying an axial load to the test specimen and/or for applying torsional loads during simulated re-entry heating. A bayonet-type chromel-alumel thermocouple,



- |                     |                         |
|---------------------|-------------------------|
| 1. Test Chamber     | 5. Leakage Tester       |
| 2. Helium Reservoir | 6. Cold Trap            |
| 3. Flow Indicator   | 7. Manometer            |
| 4. Rotameter        | 8. Temperature Recorder |

Figure 3. Test Apparatus



Figure 4. Test Chamber

inserted into the cylindrical specimen through one end, extends to midpoint in the length of the specimen. Helium gas enters the specimen through the same end as the thermocouple; the other end is used for application of the desired stresses. The stresses are applied manually.

### 3. Helium Reservoir

A helium reservoir is used to increase the volume of helium contained in the system to an amount large enough to prevent pressure drop in spite of leaks that may develop in the test specimen during test. To do this, approximately 1-1/3 cubic feet are used. It is pressurized (from the helium storage tank) to the test pressure before the start of each test.

### 4. Flow Indicator

The flow indicator (3, Figure 3) shows whether the inflating gas is flowing into the test specimen or away from it (volume increase by thermal expansion), even though the amount flowing is very small. The indicator consists of two glass bulbs that have interconnected tubes extending to the lower portion of each bulb. The bulbs are partially filled with water. The inflation gas flows into or out of them through an outlet in the upper portion of each bulb; it flows from one bulb to the other through the tubes in the lower end of each bulb. Depending on the direction of flow of gas (increased volume from re-entry heating or decreased volume from leaks), bubbling will be observed in one of the bulbs, indicating the direction of flow much sooner than it could be detected by pressure-measuring devices.

### 5. Rotameter

A rotameter (4, Figure 3) is used to measure the size of large leaks. It is a shielded, size No. 1 rotameter manufactured by Roger Gilmont Instruments, Inc. It is capable of measuring flow rates from 0.1 to 7 cc/second of helium at 80°F. When it is being used, the previously described flow indicator is by-passed, since bubbling in the flow indicator causes fluctuation in the rotameter readings.

### 6. Leak Tester

The size of small leaks is determined by measuring the rate at which water must be added to the system to compensate for the loss of gas from the test specimen. For this purpose, a burette (5, Figure 3) is connected to a leveling bottle by a rubber tube. The burette is mounted above the leveling bottle so that the water column in the burette maintains 1-psig pressure during testing for leak rates. By turning a three-way stopcock, the helium reservoir is turned out of the system and the pressure in the remaining portion of the system is maintained constant by moving the burette up so that the top of the water column is at the proper level. It will be noted that water flowing from the burette replaces an equal volume of gas lost by leakage from the specimen. The leakage rate may, therefore, be determined by observing the time for a measured volume of water to flow from the burette. Low leak rates may be determined with this equipment. Higher leak rates are more accurately measured with the rotameter.

### 7. Cold Trap

A cold trap (6, Figure 3) is used to prevent volatile decomposition products from the degradation of the coatings from reaching the helium flow rate devices. The one used is constructed from a vacuum bottle and a large test tube. The outlet and inlet tubes are large diameter glass tubes that extend well below the liquid nitrogen level to assure that all condensable gases are removed.

### 8. Water Manometer

A water manometer (7, Figure 3) is connected into the system near the test specimen so that its internal pressure can be determined at all times.

## 9. Temperature Recorder

A Werk III temperature recorder (8, Figure 3), having an accuracy of  $\pm 20^{\circ}\text{F}$ , is capable of continuously recording temperatures over the range from room temperature to  $2200^{\circ}\text{F}$  (using a chromel-alumel thermocouple).

## C. APPARATUS TESTING

### 1. General

To measure certain characteristics of the cylinder testing apparatus, a 3/8-inch-diameter stainless steel tube was used to simulate the coated metal-fiber fabric specimen. A thermocouple was welded to the outside of this specimen at a location adjacent to the thermocouple probe inside the tube.

### 2. Heating Rate

Several heating cycles were run to determine the capability of the equipment to heat the specimen to  $2000^{\circ}\text{F}$  in both 2.5 minutes and 12.5 minutes. It was found that such heating rates could be readily achieved. It was also found that the temperature of the external thermocouple corresponded closely with that of the internal thermocouple probe. A plot of the tubing wall temperature (at the midpoint of the length of the tube) versus time for each heating cycle is shown in Figure 5.

### 3. Temperature Profile

Figure 6 shows how the temperature of the specimen varied along its length. It will be noted that the  $2000^{\circ}\text{F}$  temperature is maintained only over a short length of the specimen and that the temperature decreases rapidly outside of this area. These data, obtained using a stainless steel tube as the test specimen, are presented to illustrate this point only. Specimens having a different coefficient of thermal conductivity and/or different dimensions would show different temperature profiles. Such temperature profiles are expected to exist on the surface of a re-entering expandable structure.

### 4. Volume Change due to Temperature Rise

During a heating cycle at constant pressure, it is necessary to bleed off some gas from the system because of the volume increase caused by thermal expansion of the gas in the specimen. An attempt was made to determine the approximate change in gas volume due to this gas expansion. This was accomplished by pressurizing the stainless steel tube to 1 psig and closing off all of the gas system except the specimen and the water manometer. During the period when the specimen was heated to  $2000^{\circ}\text{F}$ , both the increase in confined gas volume and the increase in pressure were observed. The increase in volume was found to be 5.25 cc at 1-psig pressure.

The above information was based upon the total volume increase during the heating cycle. This would cause a flow out of the specimen at 0.006 cc/sq in/sec during the 12.5-minute heating period, if it is assumed that the  $2000^{\circ}\text{F}$  temperature is maintained only over a 1-square-inch surface and that this surface is the total surface of the specimen. The actual surface area of the specimen is approximately 11.8 square inches. If this larger area is used in the calculation, the effect of gas expansion (or contraction during cooling) is smaller by one order of magnitude.

### 5. Pyrolysis Gases

During the heating cycle, all of the coatings tested in this program gave off some volatile products. While the effect of these materials upon the gas volume in the system was detectable, it was difficult to determine the quantitative effect of these "generated" gases on the total gas

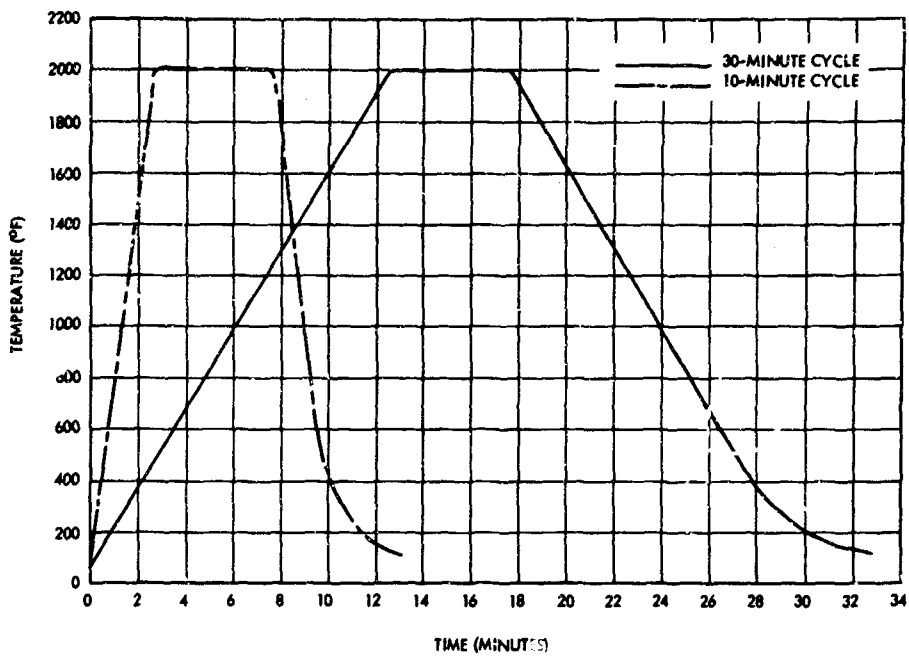


Figure 5. Heating Rate

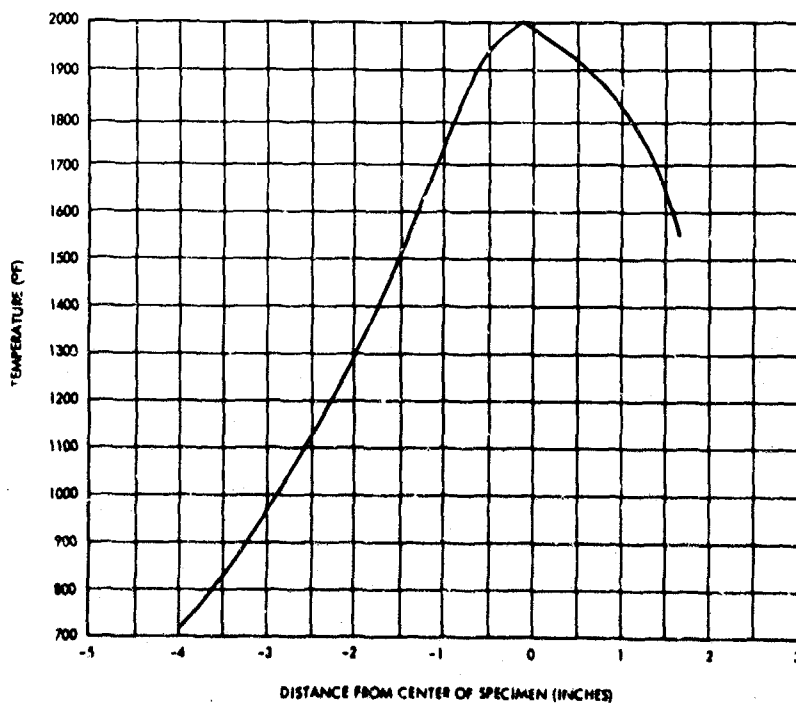


Figure 6. Temperature Profile of Stainless Steel Tube

volume change. For convenience, it was assumed that such effects on the "leak temperature" and "maximum leak rate" were too small to significantly influence the judgement of relative coating quality.

## SECTION 4

### SPECIMEN PREPARATION

#### A. GENERAL

All specimens consisted of 3/8-inch outside diameter coated metal-fabric cylinders with copper tubing inserted and silver soldered into each end. The discussion is concerned with the preparation and coating of specimens.

#### B. PREPARATION OF METAL-FABRIC CYLINDERS

Tubes were prepared with the fabric warp in the circumferential direction. The first step in their preparation was to place a precut piece of the uncoated fabric on a thick sheet of sponge rubber. A 3/8-inch brass rod was laid on the fabric. The rod was then pressed into the sponge. The resistance of the sponge to this action forced the fabric around the brass rod. The two edges of the fabric were overlapped 1/8 inch and held in this position with a metal shoe. A hand welder was used to "tack" the splice.

The tack-welded assembly was then placed in a special spot-welding jig. This jig was designed and built by Goodyear Aerospace Corporation. It consists of (1) a specimen holder attached to a Graham chain drive, (2) a Weldmatic (Model 1059B) dual-range capacitor discharge power supply (capacity is 45 watt seconds), (3) a Weldmatic (Model 1029B) heavy-duty probe hand piece adapted with a roller (one inch in diameter and 1/4-inch thick, tapered to a 1/16-inch face), and (4) a Varimatic seam weld control timer (Model No. SA3010). The combination of the spot-weld timing device and the variable chain drive can be adjusted to give the desired number of welds per inch to meet the particular welding problem. For the specimens tested in this program, the lapped seam was made using a double weld line (3/32-inch apart) with each line consisting of 25 welds per inch.

In the next operation, a 6-1/2-inch-long piece of 3/8-inch-diameter tubing was placed in each end of a 10-inch length of the welded fabric cylinder to a depth of 1/2 inch. A 1/4-inch length of 1/2-inch copper tubing was split and placed over the joint and pinched together. The split tubing, besides the cloth on the 3/8-inch copper tubing, is a heat sink during silver soldering and as such assists in making a gas-tight seal at the ends, with no holes burned in the metal fabric.

After soldering, which was accomplished using an oxygen-acetylene torch, excess soldering flux was removed with steam. The specimen was then dried. As the final cleaning step, it was placed in dichloroethylene degreasing solvent for one hour. For final drying, the specimen was placed in a drying rack and allowed to air dry at ambient conditions. Figure 7 shows a stainless steel screen specimen ready for coating.

Specimens were made using three different metal fabrics. The initial work was performed using a 200 x 200 mesh stainless steel screen. This screen was used because it was readily available and reasonably priced. A few specimens were made from a Chromel A multifilament fabric (49 filaments, 0.0007-inch-diameter, 2 x 2 twill weave, 82 warp and 102 filling threads per inch). This fabric was judged to have insufficient porosity. The third and final fabric used was made under the direction of Fabric Research Laboratories, Inc. Nichrome V (80 nickel, 20 chromium) wire, 0.0007-inch-diameter, was purchased from Driver-Harris Company. This was twisted by Fabric Research Laboratories, Inc, and the fabric was woven at Prodesco, Inc. The construction chosen for this material was a plain weave having 45 ends per inch in the warp and 45 picks per inch in the fill. Details of the weaving of the fabric are described in Appendix I.

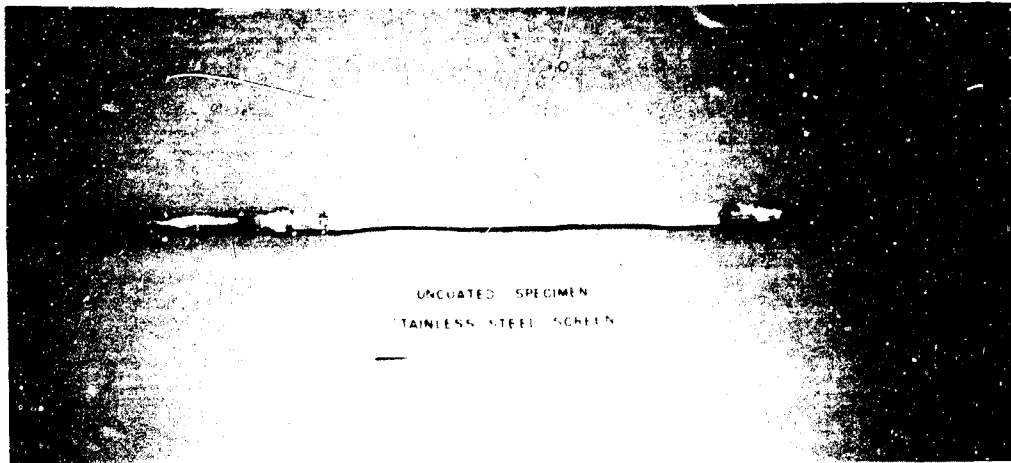


Figure 7. Uncoated Specimen

### C. INORGANIC COATINGS

For the inorganic coatings, the dry ingredients were hand-mixed. Water was added in an amount necessary to achieve a good paintable consistency. This was then brush-applied to the outside surface of a weighed metal-fabric cylinder. Care was taken to minimize the penetration of the coating into and through the fabric. Usually, after three coats, the coating appeared to be non-porous by visual inspection. This supposition was checked following drying. Additional coats were applied as necessary to stop leaks. Following drying for three days at ambient conditions, the specimens were weighed and the coating weight was calculated. Figure 8 shows a specimen coated with a 50/50 sodium silicate GD/Min-U-Sil composition.

### D. SILICONE RUBBER COATINGS

The coatings using silicone S2077 dispersion and a glass frit were mixed for 8 hours in a ball mill, then diluted as necessary with toluene. They were applied by brushing to the outside of a weighed cloth cylinder. After drying for 16 hours at room conditions, they were tested for leaks. If no leaks were found, they were step cured (recirculating air oven), starting at 100°F and increasing the temperature after each half hour by 25°F up to 500°F. The final cure was 16 hours at 500°F. After cure, the specimen was weighed and the coating weight was calculated. Figure 9 shows a typical specimen of this type.

### E. NITRILE RUBBER - PHENOLIC RESIN COMPOSITIONS

The nitrile rubber - phenolic resin - glass compositions were mixed on a 10-inch two-roll rubber mill. The sheets of compounded rubber were then dissolved in a 75/25 weight ratio of methylethyl ketone and ethyl alcohol to make a 50 percent solids solution, which was later diluted as necessary. The coatings were applied by brushing on the outside surface of the specimen. The coatings were dried for 72 hours at room conditions and then step cured (recirculating air oven), starting at 100°F and increasing the temperature 25°F after each half hour to 325°F. The final cure was for one hour at 325°F. Following cure, each specimen was weighed and the coating weight was calculated. Figure 10 shows a typical specimen coated with a nitrile rubber - phenolic resin glass composition.

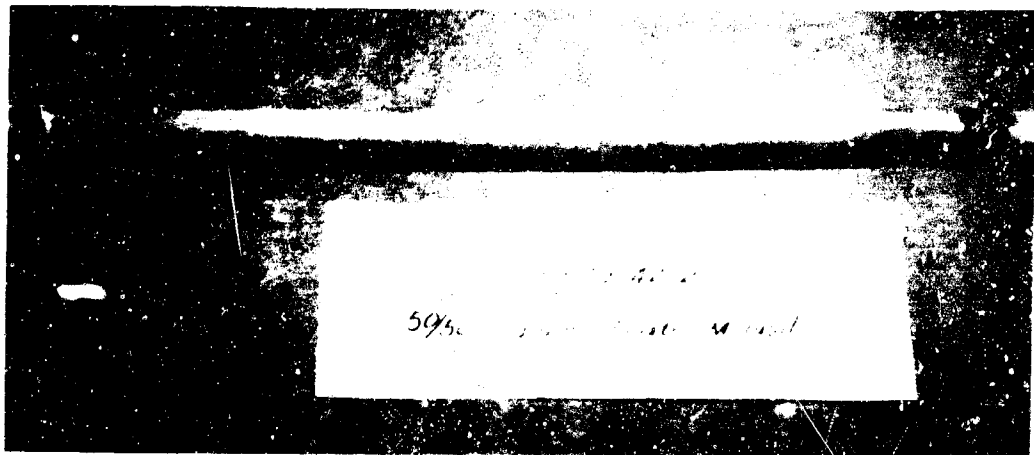


Figure 8. Inorganic Coated Specimen before Test

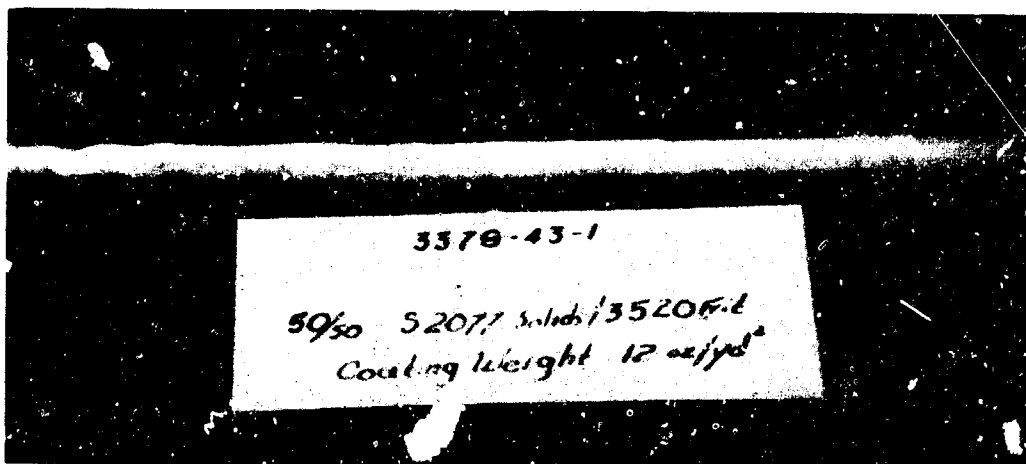


Figure 9. Silicone Rubber Coated Specimen before Test

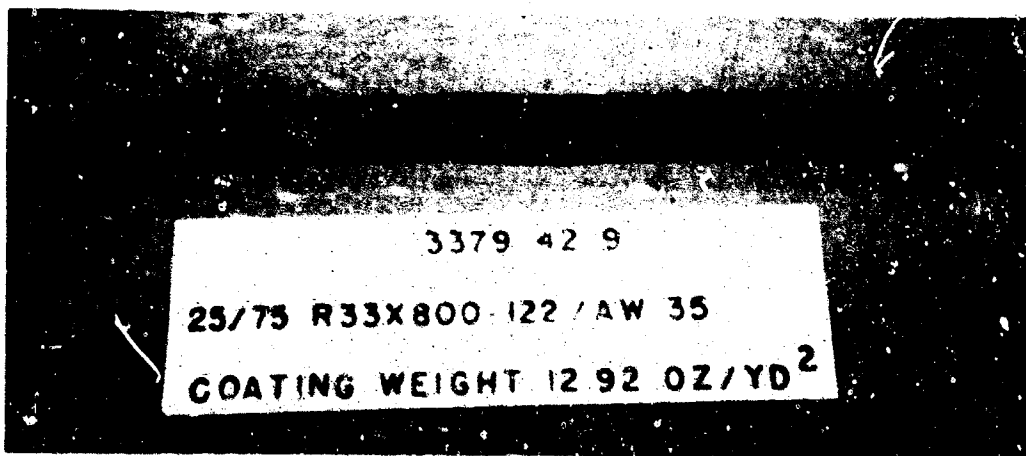


Figure 10. Nitrile Rubber - Phenolic Resin Coated Specimen before Test

SECTION 5  
TESTING COATED CYLINDERS

A. GENERAL

Eight similar methods have been used for testing the coated 3/8-inch-diameter cylinders. This section discusses the test procedures.

B. PROCEDURE 1

The test specimen was checked for leaks. If there were none, the cylinder was folded by bringing the ends together. The fold was then creased during which sufficient pressure was applied to exclude air from the fold area. The specimen was unfolded and then inflated to 1-psig helium pressure. If the pressure remained constant for 5 minutes, it was assumed that there were no gross leaks.

The specimen was then mounted in the test apparatus. Following purging with helium, the entire assembly was checked for leaks at 1-psig helium pressure. After correcting any leaks found, the cylindrical test specimen was heated to 2000°F in 12-1/2 minutes, held at 2000°F for 5 minutes, then cooled to room temperature in 12-1/2 minutes. During this time, the following steps were performed:

- (1) Nitrogen was continuously added to the space surrounding the test specimen.
- (2) An axial load of 50 percent of the breaking load of the fabric at 2000°F was applied for 30 seconds of each minute.
- (3) A 1-psig inflation pressure was maintained within the cylinder using helium.
- (4) The direction of gas flow was observed, and the temperature at which gas first flowed into the cylinder was noted on the time versus temperature chart.
- (5) The rate of flow of gas to the cylinder was measured as necessary. The time at which this was done was marked on the time versus temperature chart.

C. PROCEDURE 2

Procedure 2 was the same as procedure 1, except that the axial load was omitted.

D. PROCEDURE 3

Procedure 3 was the same as procedure 1, except that the folding of the cylinder was omitted.

E. PROCEDURE 4

Procedure 4 was the same as procedure 3, except that nitrogen was not added to the atmosphere surrounding the test specimen.

F. PROCEDURE 5

Procedure 5 was the same as procedure 3, except that air, not nitrogen, was circulated around the test cylinder.

G. PROCEDURE 6

Procedure 6 was the same as procedure 1, except that air, not nitrogen, was circulated around the test cylinder.

#### H. PROCEDURE 7

Procedure 7 was the same as procedure 1, except that the peak temperature was 2200°F instead of 2000°F.

#### I. PROCEDURE 8

Procedure 8 was the same as procedure 1, except that air, not nitrogen, was circulated around the test cylinder after the first 15 minutes of the cycle.

## SECTION 6

### TEST RESULTS AND DISCUSSION

#### A. UNCOATED SPECIMENS

Tests were performed on uncoated stainless steel specimens to determine the ultimate breaking load of the fabric at 2000°F. This was accomplished by heating the uncoated specimen in a nitrogen atmosphere in the test apparatus to 2000°F in 12-1/2 minutes, using only a slight loading. After the specimen had been at 2000°F for 5 minutes, the load was increased until the specimen broke. For this fabric, it was found that approximately 1000 grams were sufficient to cause the fabric to break. Therefore, 500 grams would be the 50-percent load used in procedure 1. However, the first specimen tested according to this procedure (3379-42-5) failed after it had been at 2000°F for two minutes. Upon examination of this specimen, the failure was found to be due to cloth failure rather than coating failure.

To determine if the cloth failure was due to interaction with the coating or to damage caused by folding, an uncoated specimen was tested in a nitrogen atmosphere after folding. The load used for this test was 500 grams. This specimen failed in tension after it had been at 2000°F for 3.25 minutes. Based on this test, it was concluded that folding of metal fabric causes considerable loss of tensile strength at 2000°F.

The breaking load at 2000°F after folding of each of the three fabrics was measured. It was thus determined that the loads to be used during testing of coated specimens should be 250 grams for stainless steel screen, 1750 grams for Chromel A cloth, and 500 grams for Nichrome V cloth. These values, besides being 50 percent of the tensile strength of the folded cylinders, are the approximate breaking strength of a 1/2-inch-wide strip of fabric following creasing.

#### B. INORGANIC COATINGS

Table IV shows the results of all tests performed on inorganic coatings. Most of the inorganic coatings evaluated contained substantial amounts of sodium silicate. In the early portion of this work it was found that, during a simulated re-entry heating cycle, the time-temperature curve showed a constant temperature at about the boiling point of water for a short time. During this time, a large leak developed. Upon inspection of these specimens after test, each showed that the coating had been converted to a hard, brittle, badly blistered material in the heated area. Figure 11 shows a typical example of this condition. When excessive leakage occurred, as it did during the testing of the specimen shown in Figure 11, the test was normally not continued. Specimen 3379-42-1, shown in Figure 12, was heated through the complete 30-minute cycle, although it leaked excessively throughout the entire cycle, starting at about the boiling point of water. The coating in the center portion of the specimen appeared to have melted and resealed; however, the coating at the ends of the specimen was badly blistered and brittle.

A study was made to determine the drying conditions necessary to obtain a flexible coating. The specimens used were strips of 200 x 200 mesh stainless steel screen that had been coated with 66.7 percent solution of Sodium Silicate GD in distilled water. Strips were placed in several closed containers above saturated solutions of salts chosen with regard to the equilibrium relative humidity obtainable. Table V shows the salts used and the relative humidity maintained by saturated solutions of these salts.

After 24 hours in the various humidity conditions, each sample was inspected. The samples exposed to 93 percent and 81 percent relative humidity absorbed moisture, and as a result, most of the sodium silicate coating had dripped into the salt solution. The specimens at 51 percent relative humidity was flexible and did not flow; however, it was somewhat tacky. The specimens at lower relative humidity were stiff and could not be creased without cracking.

Table IV. Results of Tests on Inorganic Coatings (Sheet 1)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz/yd <sup>2</sup> )
3379-42-1	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS <sup>a</sup>	18.00
3379-42-2	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	9.00
3379-42-17	R33X900-11	Alkophos CE	SS	10.24
3379-42-18	R33X900-12	75/25, Sodium Silicate GD/Min-U-Sil	SS	20.52
3379-42-19	R33X900-13	25/75, Sodium Silicate GD/Zircon	SS	27.60
3379-42-20	R33X900-14	25/75, Sodium Silicate/Feldspar	SS	26.68
3379-42-21	R33X900-15	Veegum	SS	---
3379-42-29	R33X900-16	33.3/66.7, Synar/Chromic Oxide	SS	---
3379-42-30	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	9.68
3379-42-31	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	---
3379-42-32	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	7.9
3379-42-33	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	7.4
3379-42-34	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	6.7
3379-42-25	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	6.24
3379-42-38	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	7.12
3379-42-40	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	7.6
3379-42-47	R33X900	50/50, Sodium Silicate GD/Min-U-Sil	SS	4.36
3379-42-55	R33X900-12	75/25, Sodium Silicate GD/Min-U-Sil	SS	4.76
3379-42-56	R33X900-13	25/75, Sodium Silicate GD/Zircon	SS	---
3379-42-57	R33X900-14	25/75, Sodium Silicate GD/Feldspar	SS	5.34
3379-42-58	R33X900-18	50/50, Sodium Silicate GD/Silica-Micron Flour	SS	5.28
3379-42-59	R33X900-71	Alkophos CE	SS	47.1
3379-42-69	---	Sauereisen Cement No. 18	SS	---
3379-42-123	R33X900-27	100/100/25, Sodium Silicate GD/Zircon/1547X	SS	12.90
3379-42-128	R33X900-29	100/100/75, Sodium Silicate GD/Zircon/1547X	SS	---
3379-42-130	R33X900-27	100/100/25, Sodium Silicate GD/Zircon/1547X	SS	---
3379-42-131	R33X900-30	100/100/100, Sodium Silicate GD/Zircon/1547X	SS	---
3379-42-129	R33X900-28	100/100/50, Sodium Silicate GD/Zircon/1547X	SS	---

<sup>a</sup>SS is stainless steel cloth.

Table IV. Results of Tests on Inorganic Coatings (Sheet 2)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp (°F)	Time (min)	Temp <sup>b</sup> (°F)	Time <sup>c</sup> (min)			
210	---	210	---	>7	3	
210	---	210	---	>7	3	
210	---	210	---	>7	3	
210	---	210	---	>7	3	
210	---	210	---	>7	3	
210	---	210	---	>7	3	
---	---	---	---	---	---	Brittle and flaked off cloth; no test.
---	---	---	---	---	---	Brittle and flaked off cloth; no test.
600	3.5	800	4.5	>7	3	
210	---	210	---	>7	3	
520	2.2	320	2.2	>7	3	7.5% moisture
400	2.7	400	2.7	>7	3	5% moisture
450	2.3	450	2.3	>7	3	Ends coated (R70X48-1)
520	2.9	520	2.9	>7	3	2.5% moisture
420	2.5	420	2.5	>7	3	Outside coated (RTV 511)
600	2.0	1120	6.0	>7	3	Ends coated (R70X48-1)
680	3.6	2000	18.75	>7	3	Ends coated (R70X48-1)
400	2.0	1170	6.5	>7	3	Ends coated (R70X48-1)
2000	13.3	2000	13.3	>7	3	Ends coated (R70X48-1)
400	2.0	960	5.8	>7	3	Ends coated (R70X48-1)
320	1.1	1320	7.7	>7	3	Ends coated (R70X48-1)
---	---	---	---	---	---	Leaked at room temp
---	---	---	---	---	---	Too brittle. Leaked at room temp
510	2.4	510	2.4	>7	3	
555	2.7	890	5.5	>7	3	
1085	6.6	1085	6.6	>7	3	
870	5.2	870	5.2	>7	3	
---	---	---	---	---	---	Leaked at room temp. Cracked during drying

<sup>b</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>c</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

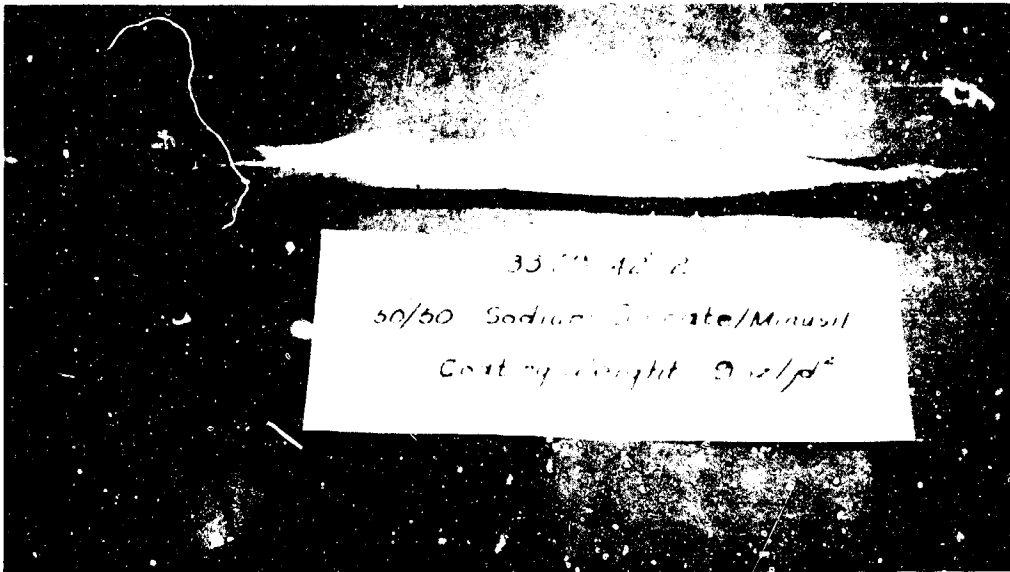


Figure 11. Inorganic Coated Specimen after Test

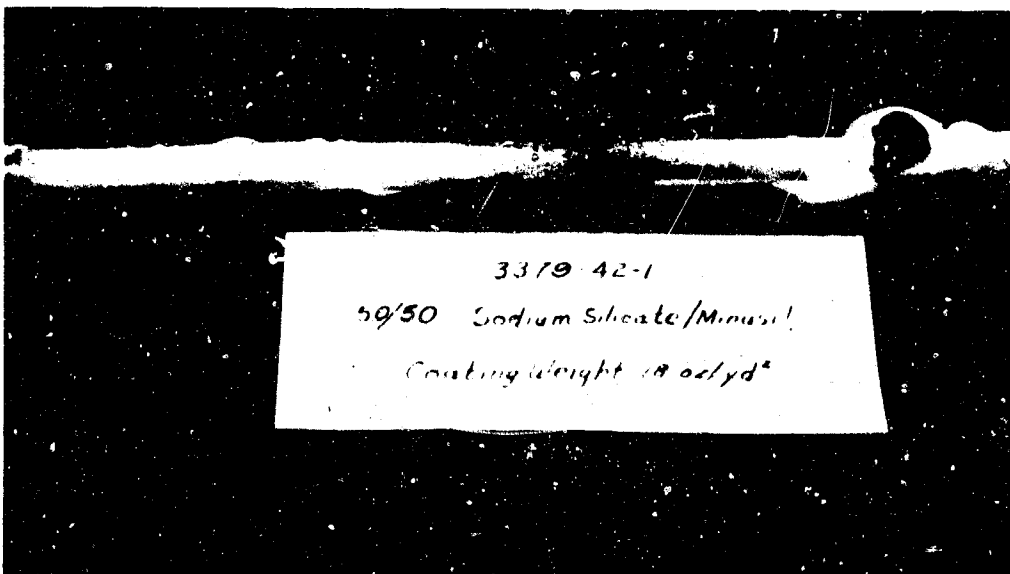


Figure 12. Inorganic Coated Specimen after 30-Minute Heat Cycle

Table V. Effect of Saturated Salt Solutions on Relative Humidity

Salt	Temp (° F)	Percent Relative Humidity
Sodium Sulfate	68	93
Ammonium Sulfate	86	81.1
Ammonium Sulfate	77	81.1
Ammonium Sulfate	68	81
Calcium Nitrate	76	51
Calcium Nitrate	65	56
Calcium Chloride	76	31
Calcium Chloride	68	32.3
Calcium Chloride	65	35
Zinc Chloride	68	10

After six days under these conditions, there appeared to be little change in the properties of these samples. The sample in the 51 percent relative humidity calcium nitrate container and the sample in the 31 percent relative humidity calcium chloride container were removed and heated in an air circulating oven at 240°F to a constant weight. A loss of 30.9 percent of the coating, presumed to be moisture, was observed for the sample conditioned in the 51 percent relative humidity calcium nitrate container, and a loss of 10.5 percent of the coating was observed for the sample conditioned in the 31 percent relative humidity calcium chloride container.

A coating consisting of an equal part mixture of sodium silicate and Min-U-Sil was dried three days at 50 percent relative humidity, then oven dried to constant weight at 250°F. The loss in weight, presumed to be water, was 12 percent of the coating weight prior to oven drying. Prior to drying, the coating was flexible. After oven drying, it was hard and brittle.

To further determine the effect of moisture content on the leak temperature of these coatings, several specimens were made using a 50/50 mixture of sodium silicate GD and Min-U-Sil. Thin coats of this material were applied to stainless steel screen specimens and, at the same time, to a film of Mylar. All were allowed to dry at 50 percent relative humidity to a constant weight. It was assumed that the same equilibrium moisture content existed in all specimens.

The moisture content of the film was determined by heating to a constant weight at 240°F. Using this moisture content as the basis, the moisture contents of the cylinders were determined following varying amounts of oven drying. These test cylinders were then tested according to procedure 3. The results of this experiment are given in Table VI.

It is interesting to note that even the "dry" specimens were found to have very wet ends when inspected following testing. When the wet ends of these specimens were coated with uncured S2077 silicone rubber and then tested, it was discovered that the specimens no longer leaked. This evidence shows that the low temperature failures during simulated re-entry testing were due to moisture that evaporated from the hottest portion of the specimen and condensed on the cooler portions of the specimen. The condensed moisture liquefied the coating and permitted excessive leakage in the wet, cool ends of the specimen.

Table VI. Effect of Moisture Content on Leak Temperature

Specimen No.	Coating Weight (oz/yd <sup>2</sup> )	Moisture Content (percent)	Leak Temp (°F)
3379-42-2	9	12	212
3379-42-32	7.9	7.5	320
3379-42-33	7.4	5	400
3379-42-35	6.24	2.5	530

To further substantiate the above theory, stainless steel screen cylinders were coated on both ends (about one third the length) with a composition of 50 percent S2077 silicone rubber solids and 50 percent AW-35 glass frit. This coating was cured for 16 hours at 500°F. Sodium silicate coatings were painted on the full length of the specimens. After drying, the specimens were tested by procedure 3, until the moisture content of the coating was about 12 percent. The data are given in Table VII.

The sodium silicate coatings, as tested, previously had leak temperatures of about 210°F. The significantly higher leak temperatures obtained when the ends of the specimens were prevented from leaking by a second coating shows that the moisture condensation theory is correct. Failure of the specimens described in Table VII occurred as a result of poor adhesion between the silicone rubber coating and the sodium silicate coating.

In an attempt to overcome this moisture problem, specimens were made using an elastomer latex in sodium silicate-zircon mixtures in anticipation that this would prove to be a means by which the water "plasticizer" could be eliminated. It was found that even excessive amounts such as 100 parts of elastomer solids resulted in specimens that were much too stiff to be folded. These specimens were tested without folding according to procedure 3. In all such specimens, excessive leakage occurred before the temperature reached 2000°F. The specimens having the least amount of latex solids had the highest leak temperature, indicating that the decomposition of the latex was a cause of the leak failure. The results are shown in Table VIII. The data shows that the addition of this latex to the soluble silicate does not make a significant improvement in the leak rate.

### C. SILICONE RUBBER COATINGS

#### 1. General

Table IX shows the compositions and results of tests performed on silicone rubber - glass frit coatings. These coatings were the most promising of the coatings tested in this program. Some of the data in Table IX are repeated in this subsection in order to illustrate important findings.

To provide knowledge about the behavior of silicone rubber coatings during re-entry heating, studies were made of the melt behavior of these coatings and coating components, using differential thermal analyses, thermogravimetric analyses, a "Multiple-Unit" decomposition furnace and hot-stage microscopic equipment.

Table VII. Sodium Silicate Coatings (Specimen Ends Protected)

Specimen No.	Coating No.	Ratio of Ingredients	Coating Weight (oz/yd <sup>2</sup> )	Leak Temp (°F)	Maximum Leak (cc/sec)
3379-42-40	R33X900	50/50, Sodium Silicate/Min-U-Sil	7.6	1120	>7
3379-42-47	R33X900	50/50, Sodium Silicate/Min-U-Sil	4.36	2000	>7
3379-42-55	R33X900-12	75/25, Sodium Silicate/Min-U-Sil	4.76	1170	>7
3379-42-56	R33X900-13	25/75, Sodium Silicate/Zircon	--	2000	>7
3379-42-57	R33X900-14	25/75, Sodium Silicate/Feldspar	5.34	960	>7
3379-42-58	R33X900-18	50/50, Sodium Silicate/Silica Micron Flour	5.28	1320	>7

Table VIII. Sodium Silicate Rubber Coatings

Specimen No.	Ratio of Ingredients	Leak Temp (°F)	Maximum Leak (cc/sec)
3379-42-130	100/100/25, Sodium Silicate GD/Zircon/1547X Solids	1085	>7
3379-42-129	100/70/50, Sodium Silicate GD/Zircon/1547X Solids	Cracked during drying.	
3379-42-128	100/100/75, Sodium Silicate GD/Zircon/1547X Solids	890	>7
3379-42-131	100/100/100, Sodium Silicate GD/Zircon/1547X Solids	870	>7

## 2. Thermogravimetric Analysis and Differential Thermal Analysis

Thermogravimetric analyses (TGA) and differential thermal analyses (DTA) were made of cured samples of R70X48-1 and its components, silicone rubber 82077, and AW-35 frit. Also, TGA's were made of 3520 frit.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 1)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz/yd <sup>2</sup> )
3379-42-3	R70X48-2	50/50, S2077 Solids/3520 Frit	SS <sup>a</sup>	11.6
3379-42-4	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.88
3379-42-5	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	9.76
3379-42-6	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	15.24
3379-42-7	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	20.45
3379-42-8	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	17.44
3379-42-22	R70X48-3	50/25/25, S2077 Solids/3520 Frit/5210 Frit	SS	11.44
3379-42-23	R70X48-4	50/50, S2077 Solids/3419 Frit	SS	17.72
3379-42-24	R70X48-5	50/50, S2077 Solids/3225 Frit	SS	14.12
3379-42-41	R70X48-1	50/50, S2077 Solids/AW-35 Frit	CA <sup>b</sup>	16.00
3379-42-42	R70X48-1	50/50, S2077 Solids/AW-35 Frit	CA	8.28
3379-42-43	R70X48-1	50/50, S2077 Solids/AW-35 Frit	CA	26.04
3379-42-44	---	S2077	SS	6.0
3379-42-45	---	S2077	SS	7.2
3379-42-48	R70X48-2	50/50, S2077 Solids/3520 Frit	CA	7.24
3379-42-49	R70X48-2	50/50, S2077 Solids/3520 Frit	CA	6.4
3379-42-50	R70X48-2	50/50, S2077 Solids/3520 Frit	CA	6.52
3379-42-51	R70X48-1	50/50, S2077 Solids/AW-35 Frit	CA	8.94
3379-42-61	R70X48-2	50/50, S2077 Solids/3520 Frit	CA	16.12
3379-42-62	R70X48-2	50/50, S2077 Solids/3520 Frit	CA	24.32
3379-42-64	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	15.14
3379-42-65	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	13.22
3379-42-66	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	14.24
3379-42-67	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.32
3379-42-68	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.16
3379-42-70	---	RTV 511	SS	19.92
3379-42-71	---	50/50, RTV 511/3520 Frit	SS	24.88
3379-42-72	---	50/50/25, RTV 511/3520 Frit/Zircon	SS	20.12
3379-42-76	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	17.32
3379-42-77	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	18.44
3379-42-78	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	15.64

<sup>a</sup>SS is stainless steel cloth.

<sup>b</sup>CA is Chromel A cloth.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 2)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp (°F)	Time (min)	Temp <sup>c</sup> (°F)	Time <sup>d</sup> (min)			
---	---	2000	---	0.13	3	
2000	17.25	2000	---	0.11	1	
2000	12.5	2000	14.5	> 7	1	Fabric failed
2000	3	2000	---	0.58	1	10-min cycle
2000	17.25	2000	---	0.1	3	
2000	14.25	2000	---	0.25	3	
780	4.3	2000	21.5	> 7	3	
1020	6.0	2000	19.5	> 7	3	
---	---	---	---	---	---	No test
---	---	2000	---	> 7	1	Leaked during cooling
0	RT	0	RT	> 7	1	Leaked during folding
---	---	2000	---	> 7	1	Leaked during cooling
1570	9.6	2000	12.5	4.85	3	
780	4.4	1205	7.2	> 7	5	
---	---	---	---	---	1	Fabric failed after 17 min at 3400-g load
0	RT	0	RT	> 7	1	Leak during folding
0	RT	0	RT	> 7	1	Leaked during folding
0	RT	0	RT	> 7	1	Leaked during folding
1900	12.0	2000	---	2.07	1	
1230	7.0	2000	---	1.54	1	
2000	14.4	2000	---	0.238	1	
2200	14.0	2200	---	0.58	1	2200°F peak temp
1230	7.1	1320	8.6	> 7	6	
1830	10.3	2200	27.3	2.56	1	2200°F peak temp
1750	10.9	2000	---	1.63	1	
1000	5.5	1000	5.5	> 7	1	
1040	6.1	1040	6.1	> 7	1	
1020	6.0	1020	6.0	> 7	1	
---	---	2000	---	0.064	1	
---	---	---	---	> 7	6	
---	---	2000	---	0.119	1	

<sup>c</sup> The temperature at which the leak rate exceeded 2 cc/sec.

<sup>d</sup> The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 3)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz/yd <sup>2</sup> )
3379-42-79	R70X48-6	66/34, S2077 Solids/AW-35 Frit	SS <sup>a</sup>	12.28
3379-42-80	R70X48-6	66/34, S2077 Solids/AW-35 Frit	SS	11.68
3379-42-81	R70X48-6	66/34, S2077 Solids/AW-35 Frit	SS	11.64
3379-42-82	R70X48-7	75/25, S2077 Solids/AW-35 Frit	SS	13.84
3379-42-83	R70X48-7	75/25, S2077 Solids/AW-35 Frit	SS	11.96
3379-42-85	R70X48-8	80/20, S2077 Solids/AW-35 Frit	SS	16.40
3379-42-63	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	12.52
3379-42-86	R70X48-8	---	SS	12.56
3379-42-87	R70X48-8	80/20, S2077 Solids/AW-35 Frit	SS	13.20
3379-42-88	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	13.60
3379-42-89	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	13.12
3379-42-92	R33X900-24	27.6/44.1/28.3, RTV 511/AW-35/Zircon	SS	11.96
3379-42-94	R33X900-23	24.6/45.9/29.5, RTV 511/AW-35/Zircon	SS	11.80
3379-42-95	R33X900-21	17.9/32.1/29.5, RTV 511/AW-35/Zircon	SS	---
3379-42-96	R33X900-23	24.6/45.9/29.5, RTV 511/AW-35/Zircon	SS	16.52
3379-42-97	R70X48-1	50/50, S2077 Solids/AW-35 Frit	8 oz/yd <sup>2</sup> SS	14.88
3379-42-101	R70X48-1	50/50, S2077 Solids/AW-35 Frit	8 oz/yd <sup>2</sup> SS	15.0
3379-42-104	R70X48-14	36.4/63.6, S2077 Solids/AW-35 Frit	SS	11.59
3379-42-105	R70X48-14	36.4/63.6, S2077 Solids/AW-35 Frit	SS	10.20
3379-42-106	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	22.96
3379-42-107	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	18.24
3379-42-108	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	18.12
3379-42-110	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.24
3379-42-111	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.76
3379-42-112	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	23.60
3379-42-113	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	12.16
3379-42-114	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	13.68
3379-42-115	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	14.96
3379-42-117	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	21.60
3379-42-118	---	S2077	SS	11.00

<sup>a</sup>SS is stainless steel cloth.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 4)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp (°F)	Time (min)	Temp <sup>b</sup> (°F)	Time <sup>c</sup> (min)			
---	---	2000	---	0.35	1	
1235	7.5	1470	9	>7	6	
2000	15.0	2000	---	0.161	1	
---	---	2000	---	0.60	1	
---	---	2000	---	0.404	1	
---	---	---	---	>7	1	
1720	---	2000	13.2	>7	6	
---	---	---	---	---	---	No test
---	---	2000	---	1.45	1	
---	---	2000	---	0.157	1	
---	---	2000	15.25	>7	8	
890	5.2	935	5.5	>7	1	One-week RT cure
1575	9.7	825	26	3.66	1	One-week RT cure
---	---	---	---	---	---	No test
955	6.7	1040	6.75	>7	1	
1360	8.6	2000	---	1721	1	
2000	14.3	2000	---	0.667	1	
1405	8.20	1680	10.3	>7	6	
2000	17.3	2000	---	0.43	1	
1145	6.8	2000	---	0.29	1	
---	8	---	8	>7	1	Damped during folding
2000	14.9	2000	---	0.119	1	
2000	13.5	2000	---	0.318	1	
---	7	---	7	>7	1	Damped during folding
1575	11.8	2000	---	0.54	1	
1260	7.6	1745	10.8	>7	6	
1700	13.0	1700	15.2	>7	6	1700°F peak temp
1300	13.7	1300	---	0.185	6	1300°F peak temp
---	---	2000	---	0.05	1	
1535	9.3	1660	10.0	>7	1	

<sup>b</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>c</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 5)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz./yd <sup>2</sup> )
3379-42-119	---	S2077	SS <sup>a</sup>	11.00
3379-42-120	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	22.00
3379-42-124	R70X48-13	44.4/55.6, S2077 Solids/AW-35 Frit	SS	11.20
3379-42-125	R70X48-13	44.4/55.6, S2077 Solids/AW-35 Frit	SS	13.32
3379-42-126	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	SS	12.52
3379-42-127	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	SS	12.72
3379-42-132	R70X48-11	36.4/63.6, S2077 Solids/AW-35 Frit	SS	11.52
3379-42-133	R70X48-11	36.4/63.6, S2077 Solids/AW-35 Frit	SS	12.40
3379-42-135	R70X48-12	40/60, S2077 Solids/AW-35 Frit	SS	12.60
3379-42-136	R70X48-15	40/60, S2077 Solids/3520 Frit	SS	10.60
3379-42-137	R70X48-15	40/60, S2077 Solids/3520 Frit	SS	11.72
3379-42-138	21101-91-C	20/18/2, S2077 Solids/85 Frit/R-200	SS	13.04
3379-42-139	21101-91-C	20/18/2, S2077 Solids/85 Frit/R-200	SS	12.48
3379-42-140	22036-5-1	20/15/10, S2077 Solids/85 Frit/R-200	SS	12.72
3379-42-141	22036-5-1	20/15/10, S2077 Solids/85 Frit/R-200	SS	11.96
3379-42-142	22036-5-4	20/18/12, S2077 Solids/3520 Frit/R-200	SS	14.04
3379-42-143	22036-5-4	20/18/12, S2077 Solids/3520 Frit/R-200	SS	13.08
3379-42-144	22036-38-2	20/9/9/12, S2077 Solids/3520/85 Frit/R-200	SS	12.68
3379-42-145	R70X48-17	100/50/50, S2077 Solids/AW-35 Frit/3520 Frit	SS	12.84
3379-42-146	R70X48-18	100/25/75, S2077 Solids/AW-35 Frit/3520 Frit	SS	14.52
3379-42-147	R70X48-19	100/75/25, S2077 Solids/AW-35 Frit/3520 Frit	SS	13.44
3379-42-148	R70X48-20	100/75/25, S2077 Solids/85 Frit/R-200	SS	13.22
3379-42-149	22036-38-2	20/9/9/2, S2077 Solids/3520/85 Frit/R-200	SS	14.80
3379-42-150	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	7.30
	R70X48-2	50/50, S2077 Solids/3520 Frit		7.30
3379-42-151	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	7.40
	R70X48-2	50/50, S2077 Solids/3520 Frit		7.40
3379-42-152	R70X48-2	50/50, S2077 Solids/AW-35 Frit	SS	6.00
	R70X48-1	50/50, S2077 Solids/3520 Frit		6.00
3379-42-153	R70X48-2	50/50, S2077 Solids/AW-35 Frit	SS	6.06
	R70X48-1	50/50, S2077 Solids/3520 Frit		6.06
3379-42-154	R70X48-1	50/50, S2077 Solids/AW-35 Frit	SS	24.00
3379-42-155	R70X48-2	50/50, S2077 Solids/3520 Frit	SS	25.6

<sup>a</sup>SS is stainless steel cloth.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 6)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp (°F)	Time (min)	Temp <sup>b</sup> (°F)	Time <sup>c</sup> (min)			
720	3.8	1085	6.5	>7	6	
2000	13.8	2000	---	0.215	1	
2000	12.5	2000	---	0.238	1	
1680	10.4	1835	11.6	>7	6	
1450	9.0	2000	---	0.222	1	
1705	10.6	2000	12.9	>7	6	
---	---	---	---	---	---	
RT	0	1660	10.5	>7	6	Slight leak initially
2000	17.2	2000	---	0.318	1	
1145	7.1	1680	10.6	>7	6	
1280	7.7	1555	9.7	>7	6	
1340	8.4	1810	11.2	>7	6	
2000	15	2000	15.0	>7	8	
1320	8.1	1600	10.0	>7	6	
---	---	---	---	---	---	
2000	15	2000	15	>7	8	
1170	6.7	1535	9.2	>7	6	
1300	8.0	1470	9.3	>7	6	
1745	10.9	2000	14.5	>7	6	
1855	11.2	2000	13.8	>7	6	
1680	10.4	2000	12.5	>7	6	
970	5.0	1620	11.9	>7	6	
1300	8.0	1600	10.1	>7	6	
---	---	---	---	>7	6	
---	---	---	---	>7	6	
---	---	---	---	>7	6	
1640	9.7	2000	12.8	>7	6	
---	---	---	---	>7	6	
1340	7.9	1620	10.0	>7	6	

<sup>b</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>c</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 7)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz/yd <sup>2</sup> )
3379-42-164	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV <sup>a</sup>	17.84 8.96
3379-42-168	R70X48-2	50/50, S2077 Solids/3520 Frit	NV	12.76
3379-42-169	R70X48-2	50/50, S2077 Solids/3520 Frit	NV	18.92
3379-42-170	R70X48-2	50/50, S2077 Solids/3520 Frit	NV	21.38
3379-42-173	R70X48-2	50/50, S2077 Solids/3520 Frit	NV	12.36
3379-42-174	R70X48-2	50/50, S2077 Solids/3520 Frit	NV	8.68
3379-42-175	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	NV	10.04
3379-42-176	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	NV	9.44
3379-42-177	R70X48-16 R70X48-1	44.4/55.6, S2077 Solids/3520 Frit 50/50, S2077 Solids/AW-35 Frit	NV	6.60 4.92
3379-42-178	R70X48-16 R70X48-1	44.4/55.6, S2077 Solids/3520 Frit 50/50, S2077 Solids/AW-35 Frit	NV	8.72 2.68
3379-42-181	R70X48-1 R70X48-16	44.4/55.6, S2077 Solids/3520 Frit 50/50, S2077 Solids/AW-35 Frit	NV	11.26 11.26
3379-42-184	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	10.68 5.60
3379-42-187	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	6.20 6.20
3379-42-188	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	6.18 6.18
3379-42-189	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	8.24 8.24
3379-42-190	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	8.48 8.48
3379-42-191	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	NV	10.74 10.74
3379-42-203	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	SS <sup>b</sup>	10.30 10.30
3379-42-204	R70X48-1 R70X48-16	50/50, S2077 Solids/AW-35 Frit 44.4/55.6, S2077 Solids/3520 Frit	SS	10.70 10.70
3379-42-205	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	SS	18.84
3379-42-206	R70X48-16	44.4/55.6, S2077 Solids/3520 Frit	SS	19.20

<sup>a</sup>NV is Nichrome V cloth.

<sup>b</sup>SS is stainless steel cloth.

Table IX. Results of Tests on Silicone Rubber Coatings (Sheet 8)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp (°F)	Time (min)	Temp <sup>c</sup> (°F)	Time <sup>d</sup> (min)			
1470	8.8	1835	18.75	>7	6	Leaked during cooling
850	5.3	2000	12.75	>7	1	
2000	12.5	---	---	2.58	8	Difficulty in switching
---	---	---	---	---	8	Fabric failed after 13.5 min at 2150-g load.
2000	13.5	2000	---	1.99	8	
1170	6.7	1535	9.0	>7	6	
1920	11.5	2000	---	0.54	8	
1105	6.5	1555	8.5	>7	6	
1855	11.3	2000	20	3.32	8	Leaked during cooling
1060	6.0	1725	11.1	>7	6	
1880	11.7	2000	---	0.66	6	Alternate 7 coats
1810	11	2000	---	1.8	6	
1660	10.2	2000	14.0	4.28	6	Alternate 6 coats
1810	11.2	2000	---	0.41	6	Alternate 6 coats
1940	12.2	2000	---	0.51	6	Alternate 8 coats
1835	11.7	2000	---	0.58	6	Alternate 8 coats
2000	12.9	2000	---	0.41	6	Alternate 10 coats
1725	8.5	1940	12.25	>7	6	Alternate 8 coats
1745	11.0	2000	13.0	>7	6	Alternate 8 coats
2000	15.5	2000	---	0.99	8	
1425	7.4	1900	12.0	>7	6	

<sup>c</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>d</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

TGA and DTA data were obtained in atmospheres of air or nitrogen to approximately 2000°F or to the maximum temperature where meaningful data were obtained. These data are shown in Figures 13 through 16, which indicate the following:

- (1) Coating R70X48-1 shows a weight loss beginning between 300 and 400°C (see Figure 13) in both air and nitrogen. At this point, an endotherm is indicated by the DTA curve (see Figure 14). However, at about 400°C, a large exotherm is indicated for the specimen tested in air, while the specimen heated in nitrogen continues to show an endotherm. The decomposition appears to be essentially completed at about 600°C in both air and nitrogen. The total weight loss in nitrogen is slightly greater than in air.
- (2) Silicone rubber S2077 again shows a weight loss beginning between 300 and 400°C (see Figure 15). An endotherm is indicated between 300 and 400°C in both air and nitrogen (see Figure 16). At about 400°C, the specimen heated in air shows a large exotherm, while the specimen heated in nitrogen continues to show an endotherm. The decomposition is essentially completed at about 600°C. The total weight loss was somewhat less in air than in nitrogen.
- (3) Frits AW-35 and 3520 show essentially no weight change when heated in air or nitrogen, and there appears to be no exotherm or endotherm until the melting point of the frit is reached (curves not shown).

### 3. Multiple-Unit Tests

Tests indicated that specimens that have been heated to 2000°F in a nitrogen atmosphere and then switched to an air atmosphere show considerable change almost immediately. It is believed that this is due to the oxidation of some of the decomposition residue of the silicone rubber. A test was devised to determine if this is so.

The apparatus used in this test was a "Multiple-Unit" furnace (manufactured by the Hevi-Duty Electric Company) into which a 1-inch stainless steel pipe was placed. Fittings were attached to the ends of the pipe to control the flow of the selected gas within the pipe. A chromel-alumel thermocouple was inserted to about the center of the pipe. The maximum temperature range possible with this unit was 1800 to 1900°F.

Samples of S2077 silicone rubber, which had been previously cured for 16 hours at 500°F, were weighed. They were then inserted into the pipe, which had been preheated to 1800°F, while either nitrogen or air was flowing through the pipe. After 5 minutes, the samples were removed and weighed. The average weight loss for three samples tested in nitrogen was 22.8 percent; the average weight loss for three samples tested in air was 32.3 percent. All samples heated in nitrogen were dark grey; all samples heated in air were white. When air was used as the atmosphere, there was a deposit of soft, white, fluffy material on the inner surface of the pipe. This volatile material was not weighed.

A sample of S2077 silicone rubber was inserted into the pipe at 1800°F while nitrogen was flowing through the pipe. After the specimen had been at 1800°F for 5 minutes, air was turned into the pipe. The sample appeared to glow much brighter than the interior of the pipe, and the glow subsided back to the brightness of the pipe after several seconds. Several such tests were run during which any temperature change in a thermocouple that was only approximately in the location of the sample was noted. A temperature rise of as much as 200°F was observed on the thermocouple.

The above tests indicated that the differences observed between specimens tested in air and specimens tested in nitrogen can be traced to the oxidation of the decomposition residue of the silicone rubber. These products, perhaps carbon, remain in the coating when heated in nitrogen but are rapidly oxidized in air and appear to cause outgassing from the coating. In addition, the loss of these products allows the molten glass frit to wet the surrounding material, forming a blown glassy surface.

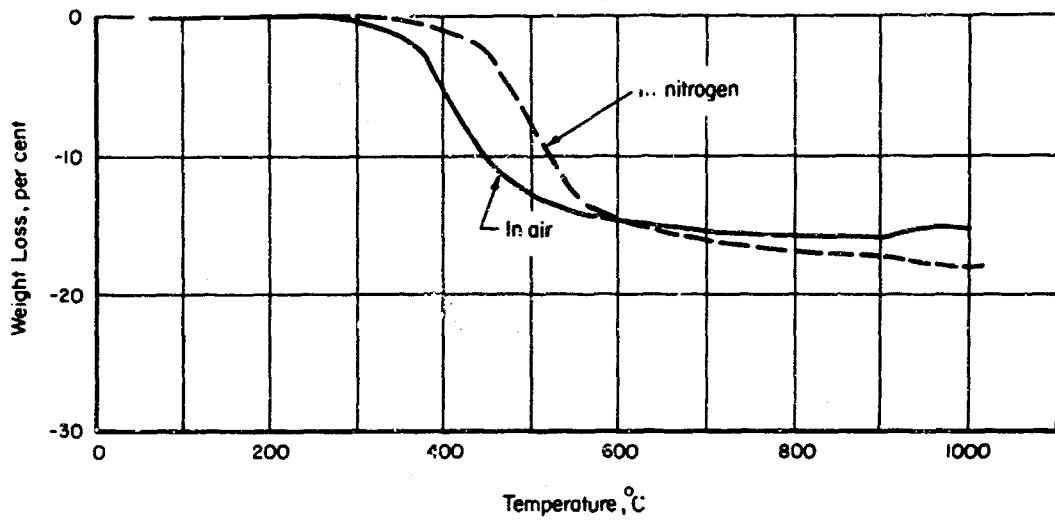


Figure 13. TGA Plots for Coating R70X48-1

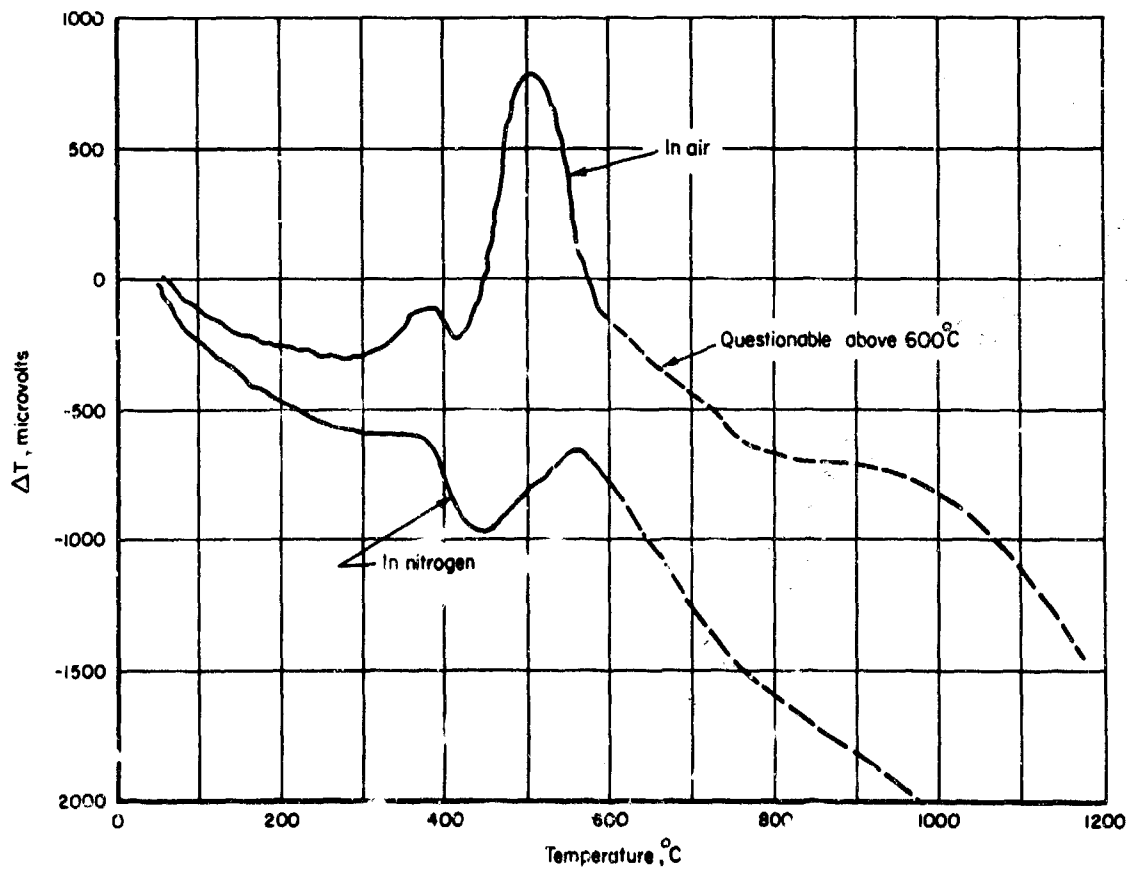


Figure 14. DTA Plots for Coating R70X48-1

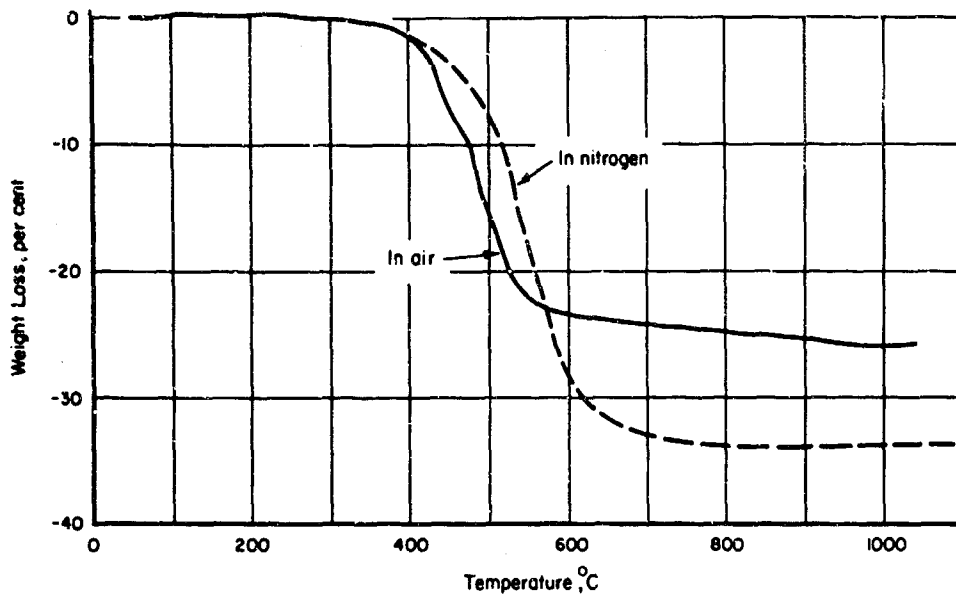


Figure 15. TGA Plots for S2077 Rubber

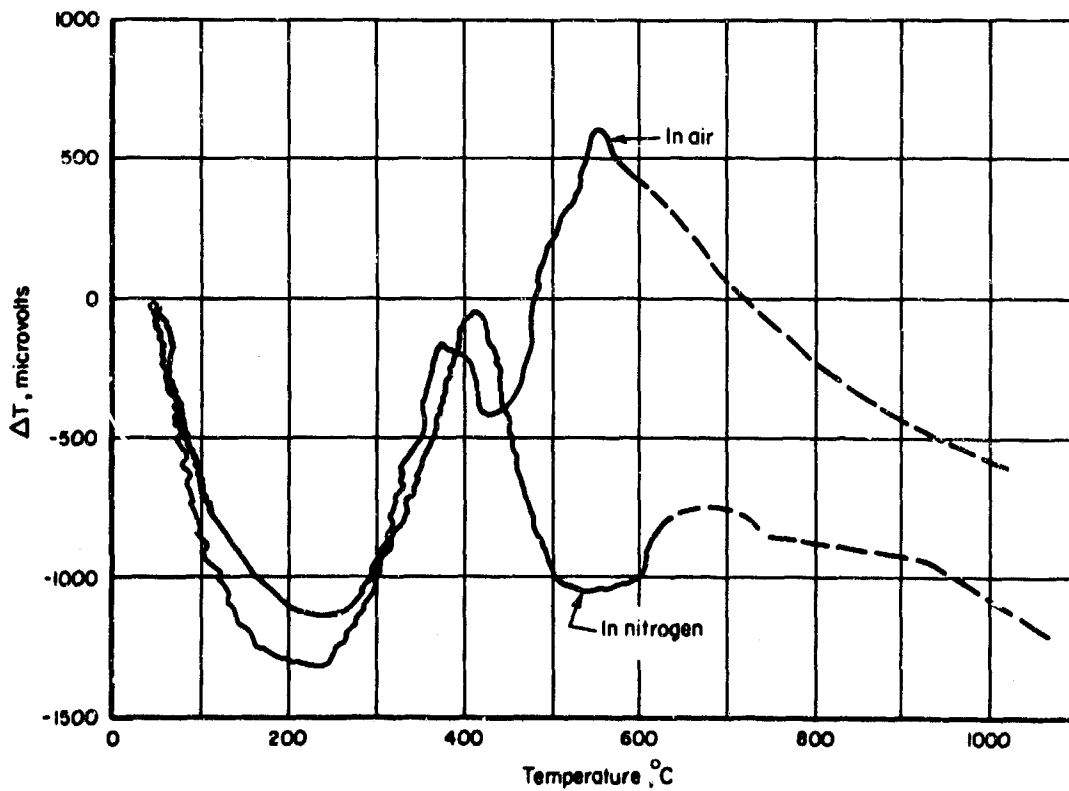


Figure 16. DTA Plots for S2077 Rubber

There appear to be discrepancies between weight loss data as determined in the "Multiple-Unit" furnace and the TGA data for S2077 silicone rubber. The TGA data show that the S2077 tested in nitrogen gives a greater weight loss than a similar sample tested in air. In the "Multiple-Unit" furnace test, the reverse was indicated. The TGA was run in such a manner that the condensable volatiles were weighed; in the "Multiple-Unit" furnace test, only the hard char was weighed.

To provide additional knowledge about this type of coating, samples of silicone rubber composition R70X48-1 (50/50, S2077 solids/AW-35 frit), cured and uncured, were decomposed in a furnace at 1000°F for 1 hour. Two samples of uncured material lost 17.42 and 17.30 percent weight; a sample of cured material lost only 12.86 percent weight. In all three instances, the residue was a hard, dark grey, ceramic-like material. It should be noted that the weight loss from the cured sample was about the same as would be expected for a flexible sodium silicate composition. Figure 17 shows the residue from these samples.

#### 4. Hot-Stage Microscopy

To observe the characteristics of coatings while being heated in an air or nitrogen atmosphere, examinations were made using a hot-stage microscope. The heating stage used was an E. Leitz model designed for observation of specimens in air, vacuum, or inert atmospheres (see Figure 18). The heating stage was equipped with a platinum heater strip (not shown in Figure 18) with a thermocouple welded to its underside for temperature measurement.

A sample was placed on the top side of the platinum heater strip. Microscopic observations were made at 56X and at 80X while the sample was being heated from room temperature to 2000°F. The samples were heated slowly and, in general, reached 2000°F in 10 to 12 minutes. They were held at 2000°F for 5 minutes, then cooled rather rapidly. During this time, an atmosphere of either air or nitrogen was maintained around the sample.

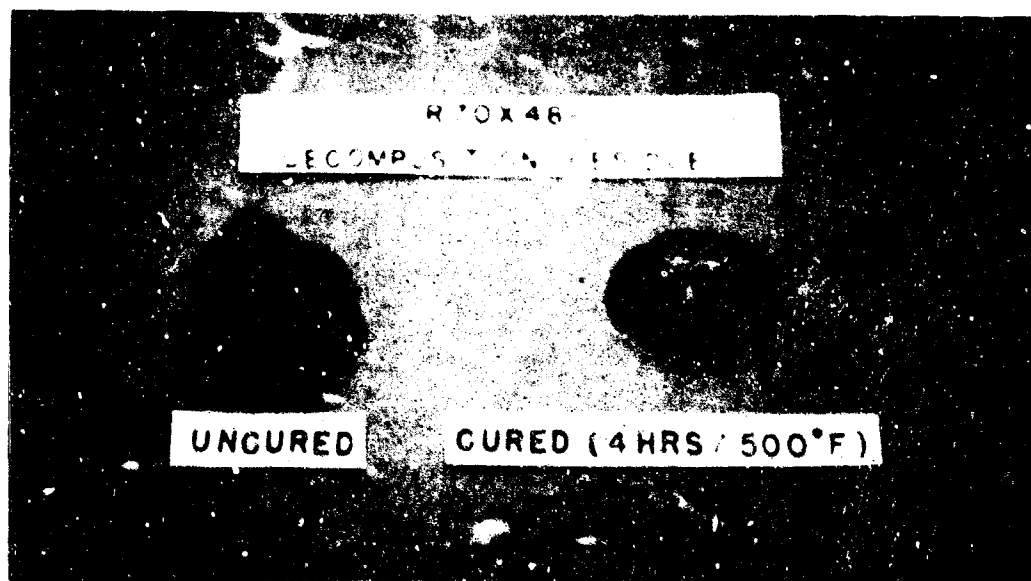


Figure 17. Pyrolysis Residue of Silicone Rubber Coating

Table X. Summary of Data on Components and Coatings (Sheet 1)

Component or Coating*	S2077 Rubber		Frit 85		Frit AW-35		R70X48-1 on SS Wire		R70X48-1		R70X48-1 no Wire		21101-91A		21101-91E		21101-98A	
	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>
Event Observed With Hot-Stage Microscope	Temperature (°F) at which observed event occurred:																	
Brown spots appear.	400-500	500-600	---	---	---	---	---	---	---	---	---	---	700	600-700	---	800	500	600
Spots darken.	600-700	600-800	---	---	---	---	---	---	820	800-1020	600-700	800	900-1000	800-900	---	---	800	---
Molten phase appears.	---	---	1100	1180	---	1180	1380	870-1000	1200	1400	1000	1440	1200	1080	---	800	---	1220
Active melting and out-gassing.	---	---	1200-1500	1240-1300	---	1280-1350	1550	1540-2000	1380-1500	1580-2000	1300-1500	1500	1300-1500	1300	---	1500	1400-1700	1400-1540
Cracks form.	1200-1300	1550	---	---	---	---	---	---	---	---	---	---	1200	---	---	---	1220	---
Cracks extend or enlarge.	1400-1500	1700-2000	---	---	---	---	---	---	---	---	---	---	1300-1480	---	---	---	---	---
Vapor or smudge forms.	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---
Molten phase wets matrix or spreads.	---	---	1700	1380-1700	1400-1500	1400-1600	1650-1800	---	1780	---	1430-1500	---	1800	2000	1650-1780	---	1540	1850
Outgassing stops.	---	---	---	---	---	---	1900	2000	---	2000	1900	1900	2000	2000	1900	2000	2000	2000
Molten phase absorbed in matrix or solidifies.	---	---	1900-2000	---	2000	2000	---	---	---	---	1600-1700	---	---	2000	<sup>b</sup>	2000	2000	2000
Pits or holes form.	---	---	---	---	---	---	1860	---	---	---	---	---	2000	---	2000	---	---	---
Permanent blisters form.	---	---	---	---	---	---	2000	---	1950-2140	---	1800-2000	---	---	2000	---	---	---	---
TGA/DTA Decomposition - temperature range	750-730	750-1380	None	None	None	None	860-1020	750-1100	750-840	770-950	---	---	---	---	---	---	---	---
Adhesive Evaluation	Poor	Poor	---	---	---	---	---	---	Poor	Poor	---	---	Poor	Poor	Good	Poor	Fair	Poor

\*Applied on Chrome/ A wire cloth unless other specified. <sup>b</sup> Remains molten.

Observations were made of several coatings prepared with various pigment and frit combinations in S2077 silicone rubber dispersion. Included were coatings containing No. 85 frit or No. 3520 frit and pigment No. R-200<sup>1</sup> in various combinations. A summary of the results is given in Table X.

Substantial differences were observed in the melting characteristics of R70X48-1, depending upon whether it was heated in air or nitrogen atmosphere. When this coating was heated in air, small droplets of molten glass formed at temperatures in the range of 1300 to 1500°F. As heating continued, the molten spots of glass appeared to wet the surrounding non-molten areas. Then, at about 1800 to 2000°F, the molten material changed from a bright to a dull luster and appeared to solidify, perhaps because of saturation of the melt with oxide residue or because of absorption of the melt by residue. During the 5-minute hold period at 2000°F, blisters developed, resulting in an uneven surface.

When this coating was heated in nitrogen, isolated fusion appeared to start at a slightly higher temperature, about 1400°F, and to persist over a greater temperature range. In contrast to the wetting and spreading behavior in air, the droplets did not spread readily to surrounding areas, but remained as distinct droplets on the surface. Changing the atmosphere from nitrogen to air at 2000°F resulted in the immediate formation of blisters similar to those reported for this coating in air. Figure 19 shows this coating after heating in air and after heating in nitrogen.

<sup>1</sup>Ingredients of AW-35 frit were purchased from Harshaw Chemical Company.

Table X. Summary of Data on Components and Coatings (Sheet 2)

21101-98D		22036-25-1		22036-25-2		22036-25-3		22036-1-3		22036-5-4		22036-38-1		22036-38-2		22036-38-3		22036-38-3 on SS Wire		
Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	Air	N <sub>2</sub>	
Temperature (°F) at which observed event occurred:																				
---	600-700	---	---	650	---	---	680-700	---	---	---	---	700	700	800	800	---	---	---	---	
---	---	700	700	---	---	800	---	---	---	---	---	---	---	---	---	---	---	---	---	
1380	1300	---	---	---	1300	1400	1400	800	---	1250	---	1300	1300	1240	1440	1100	1300	1280	1200	
1500-1680	1370-1620	1580-1800	1580-1780	1400	1400	1400-1680	1480-1800	1480	1380	1350	1400	1440	400-1800	1350	1450-2000	1500	1400	1400	1300-1500	
2000	2000	1800	---	1400	---	2000	---	1480	---	---	---	---	---	---	---	---	---	1400	1980	
---	---	1700	---	1800	---	---	---	1480-1800	---	---	---	---	---	---	---	---	---	---	1500	---
---	1600	---	---	---	---	---	---	---	---	---	---	---	2000	---	---	---	---	---	---	1800
1680-1900	2000	1800	1880	1500	1500	---	e	1500	1650-1950	1400	1800-1900	1340	1700	1350	1800-2000	1440-1800	1500-1700	1500	1600	
1600	2000	2000	2000	2000	2000	1700	---	2000	---	1600	---	2000	1800	1700	2200	---	---	2000	2000	
1800-2000	1800-2000	1950	---	---	2000	1680	---	b	b	1600	2000	1700-2000	2000	1800	2200	2000	2000	1900	1800	
---	2000	---	---	---	---	---	---	---	---	2000	---	---	2000	---	---	---	---	---	---	
---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	---	
Good	Poor	Good	Poor	Good	Poor	Good	Poor	Fair	Poor	Good	Good	Good	Poor	Good	Poor	---	Fair	---	---	

<sup>c</sup> Molten phase does not spread. <sup>d</sup> Cracks developed with fast cooling.

Observation of variations of coatings revealed the following:

- (1) Increasing the pigment content alone did not notably change the initial melting point. However, the pigment appeared to cause the molten portion to be less fluid and to promote devitrification at temperatures near 2000°F. Coatings with high pigment content exhibited lesser differences in air and nitrogen than did those coatings with low pigment content.
- (2) A substantial difference appeared when no pigment was included. Pigment-free coatings shrank considerably as the frit melted, and many cracks developed, particularly when heated in air.
- (3) Increasing the total frit content resulted in larger molten areas in the coating. This was especially noticeable in coatings heated in air. In nitrogen, the molten glass areas remained as distinct small centers at the lower temperatures. At higher temperatures (near 2000°F), the molten glass appeared to be absorbed into small areas surrounding it, but remained separated from adjacent molten spots by areas that did not appear to melt.

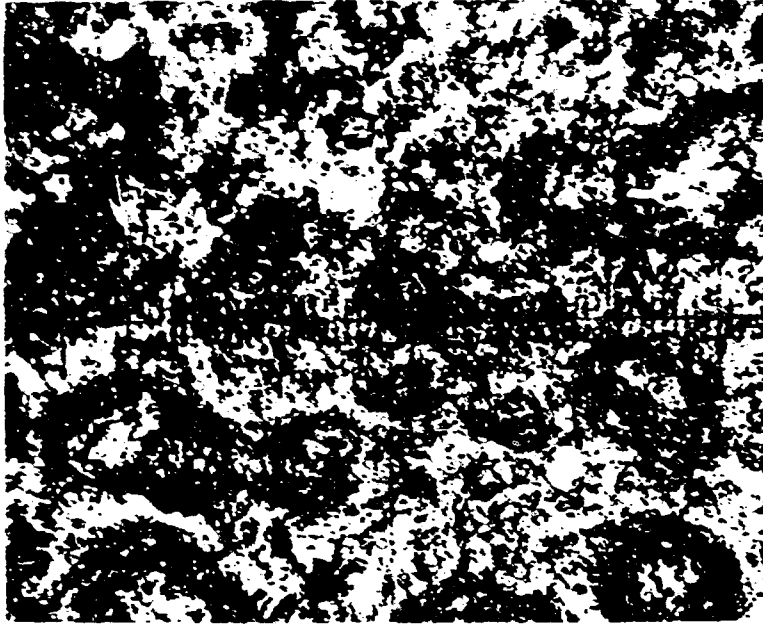
### 5. Cylinder Tests

Coatings consisting of mixtures of S2077 silicone rubber with AW-35 frit or 3520 frit were tested in both nitrogen and air atmospheres. All of the mixtures tested in nitrogen gave maximum leakage rates well below 2 cc/second throughout the entire re-entry heating cycle. This is illustrated by the data in Table XI. All specimens listed were tested according to



NOTE: MODIFIED HEATER STRIP WITH THERMOCOUPLE ATTACHED WAS SUBSTITUTED FOR HEATING ELEMENT SHOWN.

Figure 18. Exploded View of Vacuum-Heating Stage



A. HEATED IN AIR



B. HEATED IN NITROGEN

Figure 19. Coating R70X48-1 on Chromel A Wire Cloth after Heating in Air and Nitrogen Atmospheres

Table XI. S2077 Silicone Rubber - Glass Frit Coatings Tested in Nitrogen

Specimen No.	Ratio of Coating Ingredients	Coating Weight (oz. yd <sup>2</sup> )	Maximum Leak Rate (cc sec)
3379-42-68	50/50, S2077 Solids, 3520 Frit	12.16	1.63
3379-42-76	50/50, S2077 Solids/AW-35 Frit	17.32	0.064
3379-42-78	50/50, S2077 Solids/AW-35 Frit	15.64	0.119
3379-42-79	66/34, S2077 Solids/AW-35 Frit	12.28	0.35
3379-42-81	66/34, S2077 Solids/AW-35 Frit	11.64	0.161
3379-42-82	75/25, S2077 Solids/AW-35 Frit	13.84	0.60
3379-42-83	75/25, S2077 Solids, AW-35 Frit	11.96	0.404
3379-42-88	50/50, S2077 Solids, AW-35 Frit	13.80	0.155
3379-42-105	36.4/63.6, S2077 Solids, 3520 Frit	22.96	0.290
3379-42-106	50/50, S2077 Solids, 3520 Frit	22.96	0.290
3379-42-108	50/50, S2077 Solids/3520 Frit	18.14	0.119
3379-42-110	50/50, S2077 Solids/3520 Frit	12.24	0.334
3379-42-112	50/50, S2077 Solids, 3520 Frit	23.60	0.540
3379-42-117	50/50, S2077 Solids, AW-35 Frit	21.60	0.050
3379-42-124	44.4/55.6, S2077 Solids/AW-35 Frit	11.20	0.238
3379-42-126	44.4/55.6, S2077 Solids, 3520 Frit	12.52	0.222
3379-42-135	40/60, S2077 Solids, AW-35 Frit	12.60	0.318

procedure 1 and were on stainless steel screen. It can be seen that these coatings are effective helium barriers over a wide range of coating weights and over a wide range of rubber-frit ratios.

While these coatings perform very well when tested in a nitrogen atmosphere, none of the specimens tested in air has successfully withstood the entire 30-minute heating cycle. Microscopic examination of coatings after testing has shown that the coatings become more fluid when tested in air than when tested in nitrogen. The fluidity of the coating is reduced as the amount of glass frit is reduced, but they failed by cracking. Microscopic examination of coatings of 50/50 S2077 solids and 3520 frit, which had been tested in air at 2000°F, showed much less flow than the coatings containing a corresponding ratio of AW-35 frit, but they also failed by cracking. These coatings are illustrated by the data in Table XII. All of these specimens were tested according to procedure 6.

Based upon the study of the behavior of coatings during re-entry heating, it was felt that combinations of glass frits might give coatings capable of enduring a re-entry heating cycle in the presence of air. However, all of these coatings gave maximum leak rates greater than 7 cc/second. This is shown in Table XIII. All specimens were stainless steel screen.

The above data show that these frits alone or combinations of these frits in a single coating gave coatings having insufficient resistance to re-entry heating in the presence of air. However, it was felt that some combination of the properties of these coatings might give sufficient resistance if applied in separate coatings. The first attempt was made to use combinations in which one coating was applied as a top coating for another. The results of tests

Table XII. S2077 Silicone Rubber - Glass Frit Coatings Tested in Air

Specimen No.	Ratio of Coating Ingredients	Coating Weight (oz./yd <sup>2</sup> )	Leak Temp <sup>a</sup> (°F)	Leak Time <sup>b</sup> (min)
3379-42-63	50, 50, S2077 Solids/ AW-35 Frit	12.52	2000	15.2
3379-42-66	50, 50, S2077 Solids/ 3520 Frit	14.24	1320	8.6
3379-42-77	50, 50, S2077 Solids/ AW-35 Frit	18.44	2000	14.1
3379-42-80	66, 34, S2077 Solids/ AW-35 Frit	11.68	1470	9.0
3379-42-104	36.4/63.6, S2077 Solids/ 3520 Frit	11.59	1680	10.3
3379-42-113	50/50, S2077 Solids/ 3520 Frit	12.16	1745	10.8
3379-42-125	44.4/55.6, S2077 Solids/ AW-35 Frit	13.32	1835	11.6
3379-42-127	44.4/55.6, S2077 Solids/ 3520 Frit	12.72	2000	12.9
3379-42-133	36.4/63.6, S2077 Solids/ AW-35 Frit	12.40	1660	10.5
3379-42-136	40, 60, S2077 Solids/ 3520 Frit	10.6	1680	10.6
3379-42-137	40/60, S2077 Solids/ 3520 Frit	11.72	1555	9.7

<sup>a</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>b</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

Table XIII. Mixtures of Frits Tested in Air

Specimen No.	Ratio of Coating Ingredients	Coating Weight (oz./yd <sup>2</sup> )	Leak Temp <sup>a</sup> (°F)	Leak Time <sup>b</sup> (min)
3379-42-144	100/45/45/60, S2077 Solids/ 3520 Frit/85 Frit/R-200	12.68	1470	9.3
3379-42-145	100/50/50, S2077 Solids/ AW-35 Frit/3520 Frit	12.84	2000	14.5
3379-42-146	100/25/75, S2077 Solids/ AW-35 Frit/3520 Frit	14.52	2000	13.8
3379-42-147	100/75/25, S2077 Solids/ AW-35 Frit/3520 Frit	13.44	2000	12.5
3379-42-149	100/45/45/12, S2077 Solids/ 3520 Frit/85 Frit/R-200	14.80	1600	10.1

<sup>a</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>b</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

Table XIV. Dual Coatings Tested in Air

Specimen No.	Base Coat	Weight (oz. yd <sup>2</sup> )	Top Coat	Weight (oz. yd <sup>2</sup> )	Leak Temp <sup>c</sup> (°F)	Leak Time <sup>d</sup> (min)	Maximum Leak Rate (cc/sec)
3379-42-164	R70X48-1 <sup>a</sup>	17.84	R70X48-16 <sup>b</sup>	8.96	1835	18.75	>7
3379-42-178	R70X48-16	8.72	R70X48-1	2.68	1725	11.1	>7
3379-42-184	R70X48-1	10.68	R70X48-16	5.60	--	--	1.8

<sup>a</sup>R70X48-1 is a 50/50 mixture of S2077 silicone rubber solids and AW-35 frit.

<sup>b</sup>R70X48-16 is a 44.4/55.6 mixture of S2077 silicone rubber solids and 3520 frit.

<sup>c</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>d</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

performed on specimens prepared in this manner, using Nichrome V cloth, are given in Table XIV. The data for specimen 3379-42-184 show that the multiple-coat method has some possibilities.

A second method of multiple coating was investigated. In this method, coatings of R70X48-1 and R70X48-16 were alternately applied to Nichrome V cloth. The results of tests performed on specimens made in this way are given in Table XV, which shows that this type of coating is capable of meeting the goals of this program when tested in air.

## 6. Comparison of Metal Fabrics

During the early portion of this program, only the stainless steel screen was available; consequently, most of the data was obtained using coated stainless steel screen specimens. A small quantity of Chromel A cloth was furnished by the Air Force. This was an extremely low porosity cloth made from 0.0007-inch-diameter Chromel A, using 49 filaments. The yarn was a 2 x 2 twill weave having 82 warp and 102 filling ends per inch.

Late in the program a Nichrome V cloth was purchased. The manufacture and description of this cloth are given in detail in Appendix I.

The results of tests performed on specimens made from the Chromel A cloth showed that there was little or no adhesion between the cloth and the coating after simulated re-entry heating. Examination of the specimens after testing revealed that the coating had failed to penetrate the tightly woven cloth. It was felt that a less tightly woven cloth would be of value in increasing the coating penetration.

Since the Nichrome V cloth was purchased late in the program, it was not studied in combination with all candidate coatings. However, this cloth can be compared with the stainless steel screen on the basis of results from specimens coated with similar coatings. This is done in Table XVI. The data indicates that the coating performance on stainless steel screen is about the same as that on Nichrome V cloth. However, the alternate coating did not give an acceptable leak rate on the stainless steel cloth when tested in air, but was quite good on the Nichrome V cloth.

## 7. Adhesion Studies

Coated metal cloth samples were tested for adhesion to Chromel A cloth. The samples were supported on a porcelain boat in a Vycor glass tube in an atmosphere of static air or flowing nitrogen. The sample and containing tube were heated in an electric furnace from

Table XV. Alternate Coatings Tested in Air

Specimen No.	Number of Coats	Coating Weight (oz/yd <sup>2</sup> )	Maximum Leak Rate (cc/sec)
3379-42-181	7	22.52	0.66
3379-42-187 <sup>a</sup>	6	12.40	4.28
3379-42-188 <sup>a</sup>	6	12.36	0.41
3379-42-189	8	16.48	0.51
3379-42-190	8	16.96	0.58
3379-42-191	10	21.48	0.37

<sup>a</sup> Slight leak after folding; patched before heating.

Table XVI. Comparison of Stainless Steel Screen Specimens with Nichrome V Specimens

Cloth Type	Coating No.	Coating Weight (oz/yd <sup>2</sup> )	Leak Temp <sup>a</sup> (°F)	Leak Time <sup>b</sup> (min)	Maximum Leak Rate (cc/sec)	Procedure
Stainless Steel	R70X48-1	13.12	2000	15.25	>7	8
Nichrome V	R70X48-1	13.9	2000	19.25	>7	8
Stainless Steel	R70X48-1	12.52	2000	15.2	>7	6
Nichrome V	R70X48-1	14.48				6
Stainless Steel	R70X48-16	18.84	--	--	0.99	8
Nichrome V	R70X48-16	10.04	--	--	0.54	8
Stainless Steel	R70X48-16	12.16	1745	10.8	>7	6
Nichrome V	R70X48-16	9.44	1555	8.5	>7	6
Stainless Steel	8 alternate coats	21.40	2000	13	>7	6
Nichrome V	7 alternate coats	22.52	--	--	0.66	6

<sup>a</sup> The temperature at which the leak rate exceeded 2 cc/sec.

<sup>b</sup> The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

room temperature to 2000°F in 12-1/2 minutes, held for 5 minutes at 2000°F, and cooled to about 100°F in 12 to 13 minutes.

After heating, the adhesion and flexibility of the coatings were evaluated by inspection and by bending the sample over mandrels of different diameters. A sample first was bent over a 3/8-inch-diameter mandrel, then over successively smaller mandrels until the coating spalled or the cloth broke.

Coatings were prepared by ball milling silicone rubber dispersion S2077 with frit pigment mixtures and diluting with toluene to obtain a paintable consistency. These were then applied by brushing on strips of Chromel A cloth that had been cleaned by treatment in an electrolytic alkaline cleaner bath. An initial coat, which penetrated the wire mesh, was air dried, then two coats were applied on one surface of the cloth. The air-dried coatings were cured by heating 16 hours at 500°F.

The results of these studies, given in Table XVII, reveal the following:

- (1) The R-200 pigment contributes to adhesion and flexibility of the coating.
- (2) Increasing the pigment content of coating R70X48-1 improved its adhesion and flexibility.

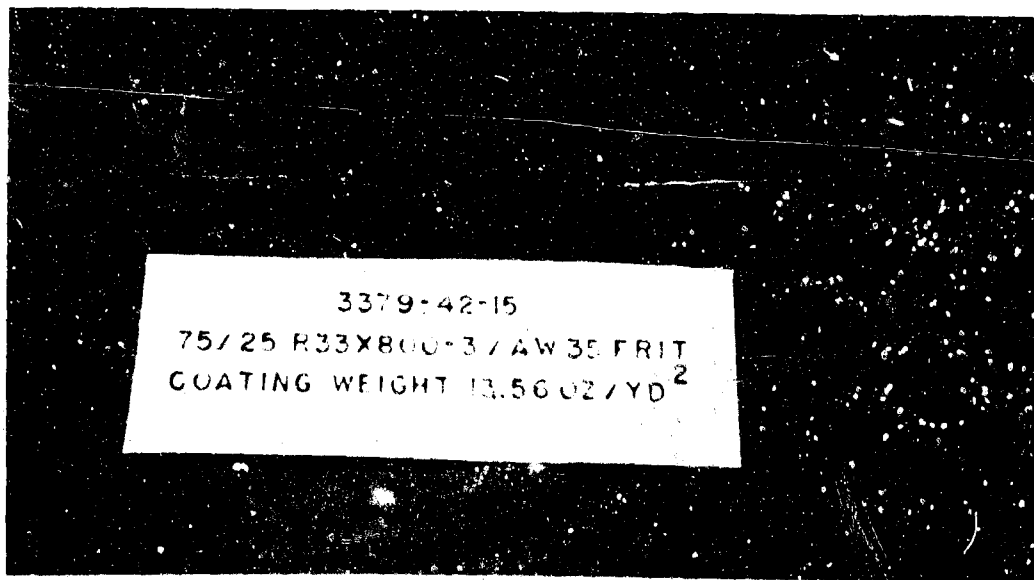


Figure 20. Typical Nitrile Rubber - Phenolic Resin Coated Specimen after Test

- (3) Coatings heated in nitrogen had poor adhesion compared to those heated in air.
- (4) Coatings containing 3520 frit and R-200 pigment had better adhesion than coatings containing 85 frit and R-200 pigment.

#### D. NITRILE RUBBER - PHENOLIC RESIN COATINGS

Several test specimens were coated with nitrile rubber - phenolic resin - glass frit compositions. All except one of these specimens failed before reaching 2000°F, as shown by the data in Table XVIII. All such coatings showed considerable blowing and surface cracking. Figure 20 shows a typical specimen after testing.

Specimen 3379-42-14 went through most of the cycle without leaking excessively. In this test, nitrogen was not turned into the chamber. After 27 minutes, this specimen showed excessive leakage. This was near the end of the cooling cycle. The pressure in the system had dropped somewhat and was being brought back to 1 psig when failure occurred. Figure 21 shows this specimen.

All of these specimens were too stiff to withstand the crease test without leaking. As a result, none of the specimens tested had been folded.

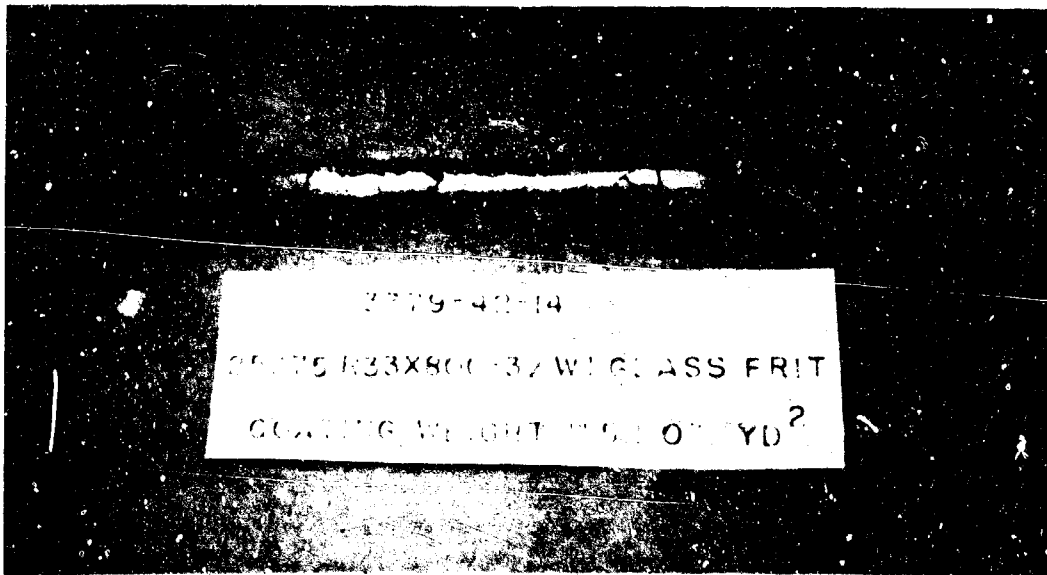


Figure 21. Nitrile Rubber - Phenolic Resin Coated Specimen after 30-Minute Heat Cycle

Table XVII. Composition of Coatings and Evaluation  
after Heating in Air or Nitrogen (Sheet 1)

Coating No.	Variation in Pigment Content						Percent of Pigment in Filler	Ratio of S2077 Solids to Filler	Approx Coating Weight (oz/yd <sup>2</sup> )
	Composition of Filler, Parts by Weight per 100 Parts S2077 Rubber (40% solids)					---			
	Frit			Pigment					
	AW-35	85	3520	R-200 <sup>a</sup>	20-2 <sup>b</sup>				
21101-93 (CS-105)	40	---	---	---	---	---	1/1	c	
21101-91A	---	40	---	---	---	---	1/1	c	
21101-91B	---	38	---	2	---	10	1/1	c	
21101-91C	---	36	---	4	---	20	1/1	c	
21101-91D	---	30	---	10	---	25	1/1	c	
22036-1-1	---	28	---	12	---	30	1/1	c	
21101-91E	---	24	---	16	---	40	1/1	c	
21101-98A	---	---	40	---	---	0	1/1	c	
21101-98B	---	---	36	4	---	10	1/1	c	
21101-98C	---	---	32	8	---	20	1/1	c	
21101-98D	---	---	24	16	---	40	1/1	19	
22036-25-1	---	---	36	---	4	10	1/1	12	
22036-25-2	---	---	32	---	8	20	1/1	12	
22036-25-3	---	---	24	---	16	40	1/1	12	
22036-1-1	---	28	---	12	---	30	1/1	c	
22036-1-2	---	35	---	15	---	30	1/1.25	c	
22036-1-3	---	42	---	18	---	30	1/1.5	c	
22036-1-4	---	56	---	24	---	30	1/2.0	c	
21101-91E	---	24	---	16	---	40	1/1	c	
22036-5-1	---	30	---	20	---	40	1/1.25	10	
22036-5-2	---	36	---	24	---	40	1/1.5	15	
22036-5-3	---	48	---	32	---	40	1/2.0	14	

<sup>a</sup>Harshaw Chemical Co. <sup>b</sup>BMI experimental pigment, 22036-20-2. <sup>c</sup>Est coating wt = 10 - 12 oz/yd<sup>2</sup>

Table XVII. Composition of Coatings and Evaluation  
after Heating in Air or Nitrogen (Sheet 2)

Appearance and Spalling Characteristics after Heating			
2000°F in Air		2000°F in Nitrogen	
Radius of Bend That Caused Spalling (in.)	Remarks	Radius of Bend That Caused Spalling (in.)	Remarks
---	Very brittle, wire fractured easily.	---	Poor adherence, spalled easily.
0.062	Shrinkage cracks in coating.	---	No test.
0.062	Very brittle, wire fractured.	---	Poor adherence, spalled easily.
> 0.187	Same as above.	---	Same as above.
< 0.017	Good adherence; flexible, could be folded.	---	Same as above.
---	Very brittle, wire fractured.	0.125	Poor adherence.
< 0.017	Good adherence; flexible, tight fold possible.	> 0.187	Same as above.
> 0.187	Very brittle, wire fractured.	> 0.187	Poor adherence, spalled easily.
< 0.017	Good adherence, did not spall.	> 0.187	Poor adherence, spalled with slight bend.
< 0.017	Same as above.	0.082	Fair adherence.
> 0.187	Brittle, wire fractured.	---	Poor adherence, spalled with slight bend.
0.062	Good adherence, cracked but did not spall.	0.093	Poor adherence, spalled.
0.095	Fine cracks in coating, did not spall.	---	Very poor adherence, flaked off easily.
0.062	Shrinkage cracks enlarged, but coating did not spall.	0.167	Fair adherence, spalled with fold.
---	Very brittle, wire fractured.	0.125	Poor adherence.
0.062	Fine cracks in coating.	0.125	Poor adherence.
0.082	Coating cracked; did not spall.	0.125	Partly spalled.
---	Very brittle, wire fractured.	0.017	Fine cracks but good adherence.
< 0.017	Good adherence.	> 0.187	Fair adherence.
< 0.017	Coating cracked but did not spall; good adherence.	0.125	Fair adherence, some spalling.
0.125	Brittle, wire fractured.	0.125	Shrinkage cracks, some spalling.
---	Very brittle, blisters in coating.	< 0.017	Fine cracks but good adherence; did not spall.

Table XVII. Composition of Coatings and Evaluation after Heating in Air or Nitrogen (Sheet 3)

Coating No.	Variation in Pigment Content					Percent of Pigment in Filler	Ratio of S2077 Solids to Filler	Approx Coating Weight (oz/yd <sup>2</sup> )
	Composition of Filler, Parts by Weight per 100 Parts S2077 Rubber (40% solids)							
	Frit			Pigment				
	AW-35	85	3520	R-200 <sup>a</sup>	20-2 <sup>b</sup>			
21101-98D	---	---	24	16	---	40	1/1	19
22036-5-4	---	---	36	24	---	40	1/1.5	20
22036-5-5	---	---	48	32	---	40	1/2.0	16
22036-38-1	---	9	27	24	---	40	1/1.5	8
22036-38-2	---	18	18	24	---	40	1/1.5	12
22036-38-3	---	29	9	24	---	40	1/1.5	8

<sup>a</sup>Harshaw Chemical Co. <sup>b</sup>BMI experimental pigment, 22036-20-2.

Table XVIII. Nitrile Rubber - Phenolic Resin Glass Frit Coatings (Sheet 1)

Specimen No.	Coating No.	Ratio of Coating Ingredients	Cloth Type	Coating Weight (oz/yd <sup>2</sup> )
3379-42-9	R33X900-1	25/75, M707/AW-35 Frit	SS <sup>a</sup>	---
3379-42-10	R33X900-2	25/75, R33X800-3/AW-35 Frit	SS	17.90
3379-42-11	R33X900-4	50/50, R33X800-3/AW-35 Frit	SS	14.92
3379-42-12	R33X900-4	50/50, R33X800-3/AW-35 Frit	SS	14.92
3379-42-13	R33X900-5	70/25, R33X800-3/AW-35 Frit	SS	12.08
3379-42-14	R33X900-6	25/75, R33X800-3/AW-35 Frit	SS	11.52
3379-42-15	R33X900-5	75/25, R33X800-3/AW-35 Frit	SS	13.56
3379-42-16	R33X900-3	25/75, M707/W1 Glass Flake	SS	---
3379-42-25	R33X900-7	25/75, R33X800-3/4-4 Frit	SS	6.20
3379-42-26	R33X900-8	25/75, M707/4-4 Frit	SS	7.20
3379-42-27	R33X900-9	50/50, R33X800-3/4-4 Frit	SS	7.6
3379-42-28	R33X900-10	50/50, M707/4-4	SS	10.4

<sup>a</sup>SS is stainless steel cloth.

Table XVII. Composition of Coatings and Evaluation after Heating in Air or Nitrogen (Sheet 4)

Appearance and Spalling Characteristics after Heating			
2000°F in Air		2000°F in Nitrogen	
Radius of Bend That Caused Spalling (in.)	Remarks	Radius of Bend That Caused Spalling (in.)	Remarks
> 0.187	Very brittle, wire fractured.	---	Poor adherence, spalled with slight bend.
> 0.187	Very brittle, wire fractured.	0.062	Cracked but did not spall.
> 0.187	Very brittle, wire fractured.	0.062	Cracked but did not spall.
0.017	Good adherence, fine craze.	0.125	Poor adherence, spalled easily.
0.167	Good adherence, rigid, wire fractured.	0.167	Poor adherence, fine cracks, spalled at cut edges.
---	No test.	0.093	Fair adherence, some spalling.

Table XVIII. Nitrile Rubber - Phenolic Resin Glass Frit Coatings (Sheet 2)

First Leak		Leak Failure		Final Leak Rate (cc/sec)	Procedure No.	Remarks
Temp <sup>b</sup> (°F)	Time <sup>c</sup> (min)	Temp (°F)	Time (min)			
---	---	---	---	---	---	Leaked at room temperature.
1375	---	1470	---	> 7	3	
940	5	940	5	> 7	3	
940	5	940	5	> 7	3	
900	4.7	900	4.7	> 7	3	
925	5.75	2000	27	> 7	3	
---	---	---	---	---	---	Blown after cure; no test.
---	---	---	---	---	---	Leaked at room temperature.
940	10.5	1120	12	> 7	3	
---	---	---	---	---	---	Leaked at room temperature.
200	---	200	---	> 7	3	
960	5.6	1280	7.9	> 7	3	

<sup>b</sup>The temperature at which the leak rate exceeded 2 cc/sec.

<sup>c</sup>The time from the beginning of the heating cycle until the leak rate exceeded 2 cc/sec.

## SECTION 7

### CONCLUSIONS AND RECOMMENDATIONS

The multilayer coatings composed of S2077 silicone rubber - AW-35 frit and S2077 silicone rubber - 3520 frit show sufficient promise for satisfactory performance during 3000°F atmospheric re-entry to justify further investigation on a larger scale. The data presented in this report indicate that the performance of the coating during such a program will depend on the following:

- (1) The variation of skin temperature at any instant of time during re-entry as well as peak skin temperature and other time-temperature factors.
- (2) The amount of oxygen in the atmosphere surrounding the structure during re-entry.
- (3) The weave of the cloth to which the coating is applied and the other characteristics of the cloth such as (a) those that would affect the ability of the coating to wet cloth during application and during the molten phase that occurs during re-entry, (b) resistance to chemical degradation by oxygen and the molten coating at the peak skin temperature, (c) resistance to flexural fatigue, (d) creasing, and (e) tensile strength and creep at and near the peak skin temperature.
- (4) Treatment of the structure before re-entry (for example, simulated packaging involving folding and creasing).

Although some data obtained during the course of this project indicated that cyclic loads and erosion due to high-velocity gas impingement did not affect the performance of the coating during re-entry, it is recommended that phenomenon such as will produce variable stress (direction and magnitude) and surface erosion be included in future programs.

Even though it may not be practical to subject a large re-entry structure to the re-entry environment, it is recommended that a full-scale vehicle be built and subjected to such tests as are practical (for example, change in shape and dimensions as a function of inflation pressure and externally applied loads). Probably of equal importance is that building such a vehicle will provide the opportunity to discover, and possibly solve, problems associated with the fabrication of a structure from these particular materials.

## APPENDIX I

### FINE WIRE, MULTIFILAMENT YARN, METAL FABRIC

Report Prepared by  
Fabric Research Laboratories, Inc.  
1000 Providence Highway  
Dedham, Massachusetts

#### WIRE

Fabric Research Laboratories, Inc., received 12.224 pounds of 0.0007-inch diameter Nichrome V (80 nickel - 20 chromium) wire from the Driver-Harris Company. The wire was bare, bright, and fully annealed. It was received on 1P spools (3-inch over-all length, 1-1/2 inch barrel OD, 0.635-inch barrel ID, 2-1/2 inch flange OD) with 10 ends on each spool. An open wind (about 20 wraps/inch) and an oil lubricant were used by Driver-Harris in spooling the wire.

Some difficulty has always been experienced in removing fine wire from the spools on which it is supplied.<sup>1, 2, 3</sup> However, this 12 pounds of wire was of the highest quality received to date. Difficulty was encountered in removing the wire from only about 12 spools. FRL® personnel were able to clear up all but one of these spools within about five minutes under a microscope. Only a small quantity of wire was left on the one spool that could not be cleared up, and it was not needed.

The wire on some of the first few spools was a gold color while the wire on the rest of the spools was silver in color. Wire of both colors was tensile tested on an Instron. No differences were observed in either strength or elongation to rupture. The gold color probably resulted from air leaking into the annealing tubes, causing oxidation of the wire.

#### YARN

The 0.7-mil Nichrome V wire was twisted and plied into a 100-filament yarn (10/10/1, 5 S/3 Z tpi<sup>4</sup>). The 10 ends of wire on each spool were twisted 5 S<sup>5</sup> turns per inch and 10 ends of this twisted yarn were plied 3 Z<sup>4</sup> turns per inch. The yarn was balanced, i.e., it exhibited little tendency to twist on itself when relaxed.

The twisting was done on a textile ring-twister. The 10 ends of wire were removed from the spools on which they were supplied by dereeling.<sup>3</sup>

In the plying operation, the twisted yarn (10 ends of wire twisted together 5 S tpi) was removed from the bobbins, onto which they had been twisted, by pulling off over the end in conjunction with a "wisker disk<sup>6</sup>". A wisker disk consists of radially oriented nylon monofilaments glued between paper disks. It is held on the end of the bobbin over which the yarn is plied. The nylon monofilaments provide the correct amount of back tension on the yarn, minimize wire contact with the bobbin flange, and trap the wire (between filaments) upon rapid

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<sup>1</sup> Refer to ML-TDR-64-102, by M. J. Coplan, W. D. Freeston, Jr., and M. M. Platt.

<sup>2</sup> Refer to ASD-TDR-62-542, Part II (Phase I - Weaving of Stranded Metal Yarns), by W. D. Freeston, Jr., and J. W. Gardella.

<sup>3</sup> Refer to AFML-TR-63-342, by W. D. Freeston, Jr., R. E. Sebring, and A. K. Swanson.

<sup>4</sup> Turns per inch.

<sup>5</sup> S and Z denote twist direction.

<sup>6</sup> Manufactured by Azonic Products, Inc, Palos Heights, Illinois.

take-up decelerations, thereby preventing the wire from sluffing off the end of the spool. The back tension can be varied appropriately with wire diameter and number of filaments per yarn by taking a wisker disk with a large number of filaments and cutting out every third or fourth filament until a suitable tension is achieved.

The wire was twisted and plied at a spindle speed of 5400 rpm. The feed roll speed during the twisting operation was 87 ft/min, and during the plying, 145 ft/min. One pound of plied yarn was put on each bobbin. The machine running time for each doff of twisted yarn was, therefore, 150 minutes and for the plied yarn, 90 minutes.

Nylon travelers were used.<sup>1</sup> An E14C was used for the twisting and an E131D for the plying. The travelers were replaced at the end of each doff.

A wax coating was put on the yarn during the plying operation by running the yarn over a felt pad saturated with polyethylene glycol 300 condensed with stearic acid and in solvent.<sup>2</sup> The purpose of the coating is to facilitate weaving. It tends to improve the abrasion resistance of the yarn and hold the filaments in a tighter bundle.

Large diameter Alsimag<sup>3</sup> and/or Heanium<sup>4</sup> guides were used throughout the twisting, plying, warping, and weaving of the wire yarn.

Eleven packages of yarn were made, one pound of yarn on each package. Yarn from six of the packages was tensile tested. The rupture loads ranged from 3.95 to 4.24 pounds, and the rupture elongation from 8.6 to 10.9 percent. A 10-inch gauge length and 0.5 in./min jaw speed were used.

## FABRIC

The fabric was woven at Prodesco, Inc, Perkasio, Pennsylvania in accordance with procedures developed by FRL<sup>®</sup> under programs sponsored by the Air Force Materials Laboratory.<sup>5 6</sup>

The metal yarn was woven into a plain weave fabric on a standard 40-inch wide silk loom.<sup>6</sup> A 45-dent, oval-wire reed with a 55 percent airspace was used, and the fabric was woven at approximately 110 picks per minute.

A 9-1/2 yard warp, made by winding the yarn on a reel with a circumference of 9-1/2 yards was used. Each wrap of yarn was laid alongside the next, 45 ends to the inch, for 38 inches. The yarns were clamped between metal strips at two adjacent locations on the reel periphery. The yarn between the two sets of clamps was then taped and cut. One set of clamps was kept fastened to the reel; the other was released and the free end of the warp taped to a warp beam. The warp was then wound on the beam, a layer of paper between each wrap, and the beam placed in the loom. The warp was drawn through the heddles and reed, grouped in bunches of approximately 90 ends and tied to a cloth leader.<sup>6</sup>

The yarn wound on the reel in making the warp was dereeled off the bobbins on which it had been wound in the twisting operation. It was pulled through a trough containing polyethylene glycol 300 condensed with stearic acid and in solvent, thereby recoating the yarn.

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<sup>1</sup> Refer to AFML-TR-63-342, by W.D. Freeston, Jr., R.E. Sebring, and A.K. Swanson.

<sup>2</sup> Manufactured by Specialty Products Co, Jersey City, New Jersey.

<sup>3</sup> American Lava Corp.

<sup>4</sup> Heany Industrial Ceramic Corp.

<sup>5</sup> Refer to ML-TDR-64-102, by M.J. Coplan, W.D. Freeston, Jr., and M.M. Platt.

<sup>6</sup> Refer to ASD-TDR-62-542, Part II (Phase I - Weaving of Stranded Metal Yarns), by W.D. Freeston, Jr., and J.W. Gardella.

The filling yarn for the fabric was wound on conventional quills by dereeling the yarn off the twister bobbins. A standard shuttle was used. The filling yarn was not resized.

The fabric was held out during weaving by using two ends of the metal yarn as catch cords and No. 2 Draper crow pickers.

The width of the fabric off the loom was about 27-3/8 inches. Twenty feet of fabric were obtained. The number of picks and ends per inch was measured at several locations in the fabric. The number ranged from 45 to 46. The permeability of the fabric could not be measured because FRL's Frazier Permeometer was in use on another program. However, the fabric did not appear to be as porous as anticipated. The yarns flattened considerably during weaving.

The brown streak in the fabric results from the gold-colored wire, as previously discussed.

APPENDIX II  
MATERIALS IDENTIFICATION

Material	Description	Supplier
Alkophos CE	Colloidal liquid aluminum phosphate	Monsanto Research Corp
AW-35 Frit	Low-temperature glass frit (6.27% Cu, 7.92% , 1.35% Boron, 54.51% Pb, 5.0% Si, 1.09% Al)	Harshaw Chemical Co
Chromic Oxide	Chromium oxide	Minnesota Mining & Mfg Co
Feldspar	Clinchfield feldspar No. 202	International Minerals
Min-U-Sil	5 $\mu$ silica powder	Pennsylvania Glass Sand Corp
M707	Classified (description in classified final report, NOrd 16640 Subcontract No. 35)	The Goodyear Tire & Rubber Co
RTV 511 Silicone	Room-temperature vulcanizing silicone rubber compound	General Electric Co
R-200 Pigment	Copper chromite black pigment	Harshaw Chemical Co
R33X800-3	100 Chemigun N5, 120 Durez 12687, 5 zinc oxide, 3 Me-Tuads, 2 stearic acid	The Goodyear Tire & Rubber Co
Sodium Silicate GD	Dry powder (27.5% Na <sub>2</sub> O, 55.0% SiO <sub>2</sub> , 17.5% H <sub>2</sub> O)	Philadelphia Quartz Co
Synar	Polymeric silicic acid	Pennsalt Chemical
S2077 Silicone	Silicone rubber dispersion	Dow Corning Corp
W1 Glass Flake	2-1/2 $\mu$ flake high lead glass	Owens Corning Fiberglass Corp
Zircon	Milled unsized zircon powder	Titanium Alloy Mfg Co
1547X Latex	Nitrile rubber latex	The Goodyear Tire & Rubber Co
3225 Glass Frit	325-mesh glass frit	Ferro Corp
3419 Glass Frit	200-mesh glass frit	Ferro Corp
3520 Glass Frit	200-mesh glass frit	Ferro Corp
4-4 Glass Frit	PbO (80%), B <sub>2</sub> O <sub>3</sub> (10%), Al <sub>2</sub> O <sub>3</sub> (5%), SiO <sub>2</sub> (5%)	Battelle Memorial Institute
5210 Glass Frit	Glass frit	Ferro Corp
85 Frit	Low-temperature glass frit	Harshaw Chemical Co

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13. ABSTRACT This report describes the accomplishments of the second year of a two-year program, the object of which has been the development of a protective coating for use on an expandable (inflatable) re-entry structure. It is envisaged that the re-entry structure will be fabricated from a high strength metal fiber fabric, coated with a flexible, rubber-like coating. The coating and its application to the metal fabric have been the objects of investigations under this program. Improved apparatus and procedures were developed for testing of coated metal fabrics under simulated re-entry conditions. Coatings of three general types were tested; the general types were (a) wholly inorganic, (b) silicone rubber with fusible filler, and (c) ablative organic insulation with fusible filler. The silicone rubber (S2077)-glass frit (AW35 and/or 3520) coatings were found most effective and performed satisfactorily over a range of coating weights and rubber to frit ratios when heated to 2000°F in a stagnant nitrogen atmosphere. The more severe condition of heating in air required alternate coats of silicone rubber-AW35 frit and silicone rubber-3520 frit to a total of 6-8 coats. Neither coating alone could prevent excessive leakage at 2000°F in air.		

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Protective Coating Re-entry Coating Silicate Coating High Temperature Coating Frit-Filled Coating Coated Metal Fabric						

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