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EFFECTS OF ELEVATED TEMPERATURE AND REDUCED ATMOSPHERIC PRESSURE ON ADHESIVES, POTTING COMPOUNDS AND SEALANTS

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GENERAL DYNAMICS | FORT WORTH

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Structures and
Materials

EFFECTS OF ELEVATED TEMPERATURE AND REDUCED ATMOSPHERIC PRESSURE ON ADHESIVES, POTTING COMPOUNDS AND SEALANTS

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30 December 1961

ENGINEERING DEPARTMENT

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GENERAL DYNAMICS | FORT WORTH

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EFFECTS OF ELEVATED TEMPERATURE AND REDUCED ATMOSPHERIC PRESSURE ON ADHESIVES, POTTING COMPOUNDS AND SEALANTS

J. P. Thomas
R. J. Stout

SUMMARY

Five materials which might have specialized uses on glide and boost-glide type vehicles were evaluated under space environmental conditions of up to 700°F and reduced pressure in the order of 1 x 10^{-3} mm Hg. The organic and semiorganic materials selected were among the materials believed to possess superior thermal qualities, however their behavior under reduced pressures was unknown. The materials selected for this study included, (1) a silicone rubber sealant, Dow Corning Q-2-0103, (2) a butyl rubber general purpose cement, Permalastic PX-1040, (3) an epoxy room temperature curing adhesive, Epon 931, (4) a phenylsilane-based adhesive, Dynabond 118 and (5) a Viton rubber-based cement, Fairprene 5159. An environment of 200°F and reduced pressure of approximately 1.0 x 10^{-3} mm Hg for 300 hours did not appear to significantly affect the strengths of the five materials tested. Retention of adhesion of the butyl rubber cement was also satisfactory (14 per cent loss) after 300 hours at 400°F and approximately 1.5 x 10^{-3} mm of Hg vacuum.

The silicone sealant sponged at approximately 2.5 x 10^{-3} mm Hg vacuum and 700°F. Nevertheless, it is felt that this material, with slight modifications in initial cure, may exhibit improved thermal stability. Dynabond 118 retained a portion of its strength after exposure to 700°F and approximately 2.5 x 10^{-3} Hg vacuum, but did not appear to possess adequate strength as a structural adhesive (1000 psi) under these space environmental conditions. Neither the Epon 931 nor the Viton rubber cement performed satisfactorily under the simulated space conditions of 700°F and approximately 2.5 x 10^{-3} mm Hg vacuum for 10 hours.

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INTRODUCTION

Utilization of adhesives, potting compounds and sealants will be an important factor in any space vehicle manufacturing process. They are preferred for attaching fibrous insulations, seals, nameplates, clips; sealing pressurized areas, protecting electronic equipment etc., inside and outside pressurized areas. The combined influence of reduced pressure and elevated temperature on such materials must be determined in order to aid proper selection of materials. It is known that a lack of oxygen will improve some material's performance at elevated temperatures. However, elevated temperature in conjunction with low pressure may result in degradation of a material by vaporizing plasticizers and antioxidants and degrading the basic polymer into smaller fragments. Data obtained from studies of these materials under the environments described should indicate the degree of the problem.

The Department of Defense and the National Aeronautics and Space Administration have performed and sponsored both industrial and private institutional research on the effects of temperature and vacuum on materials for space environments.

Based upon this background of the need for studies on the effects of space environment on polymeric materials, this research program was initiated at GD/FW in order to study the effects of vacuum and temperature on selected materials. Materials to be studied include adhesives, potting compounds, electrical insulation, sealants and high temperature rubberized fabric coatings which may prove useful for certain aerospace applications. A basic requirement of such materials will be that they be physically and chemically stable to space environment.[1]

OBJECTIVE

The objective of this research is to establish the behavior of potting compounds, structural adhesives and general purpose cements in a high temperature, low atmospheric pressure environment.

EXPERIMENTAL PROCEDURE

The experimental procedure is described in detail in Appendix I.

RESULTS AND DISCUSSION

Research that has attempted to relate weight loss to vapor pressure or decomposition pressure has not been too successful since the polymeric materials degrade by breakdown to smaller fragments.[2] The molecular weight of these fragments or the equilibrium decomposition pressure of most polymers is not well established. Accordingly, calculations based on vapor or decomposition pressures are usually not useful for the organic materials of interest and it becomes necessary to turn to direct experimental studies of weight loss of polymers in vacuum. The purpose of this test was, therefore, to determine experimentally the effect of simulated atmospheric pressures and temperatures typical of the flight profiles for boost glide and glide re-entry vehicles on selected polymeric materials.

The specific materials selected for this study were:

(1) a silicone rubber sealant with an antioxidant (Dow Corning Q-2-0103)

(2) a butyl general purpose cement (Permalastic PX 1040),

(3) a room temperature curing epoxy adhesive (Epon 931),

(4) a phenylsilane based adhesive (GD/FW developed Dynabond 118)[3]

(5) Viton rubber-based cement (Fairprene 5159).

Aging of these materials at 200°F for 300 hours with or without a vacuum of 1×10^{-3} mm Hg resulted in little loss of weight or adhesion for all the materials tested as shown in Tables II, III, VI, VII, VIII, X and summarized in Table I. After 300 hours at 400°F the butyl adhesive exhibited approximately

5 percent weight loss at 1.5×10^{-3} mm Hg and over 30 percent without vacuum while adhesion was 3.0 and 0.5 lbs/inch width respectively under the same conditions.

After being subjected to the 700°F thermal environment, with and without vacuum, none of the materials retained useful strength properties. All of them were less affected in vacuo than in air but not to a degree that would permit their application in an environment of 2.5×10^{-3} mm of Hg for 10 hours at 700°F.

The Viton adhesive decomposed so badly at 700°F, it contaminated other specimens in the test chamber. This condition made it necessary to rerun the Dynabond 118 and Epon 931 adhesive specimens.

It might be unwise to conclude, based on tests described herein, that the basic polymers used in the adhesives and potting compound tested are unsuitable for use in compounding materials for use in space vehicles. Different compounding and processing techniques could possibly improve resistance of the materials to the environmental conditions. More extensive testing is needed to establish a basis for predicting the combined efforts of temperature and high vacuum on materials.

CONCLUSIONS

The effects of simulated atmospheric pressures of approximately 280,000 to 325,000 feet altitudes and temperatures up to 700°F on five different materials was determined and the following conclusions were drawn:

1. The effects of 200°F for 300 hours, either with or without 1.0×10^{-3} mm Hg vacuum, did not appear to significantly alter the strengths of the five materials tested.

2. The silicone sealant, Q-2-0103, sponged at 700°F under 2.5×10^{-3} mm Hg vacuum, but still retained some peel strength.

3. Permalastic PX-1040 general purpose cement exhibited satisfactory adhesion after 300 hours at 400°F and approximately 1×10^{-3} mm Hg vacuum.

4. Even though Dynabond 118 exhibited approximately three times the shear strength of Epon 931 after 10 hours at 700°F and 2.5×10^{-3} mm Hg vacuum, neither adhesive exhibited what might be considered a satisfactory shear strength (1000 psi).

5. The Viton Fairprene 5159 performed unsatisfactorily at 700°F both with or without reduced pressure of 2.5×10^{-3} mm Hg.

RECOMMENDATIONS

1. Since the techniques used in this report did not employ a cold wall type vacuum chamber or extremely low pressure, it is recommended that GD/FW study the effects of vacuum up to 10^{-7} mm Hg and temperature to 700°F on some of the most promising heat resistant materials in a cold wall chamber with high capacity pumping equipment.

   A cold wall chamber should be used to prevent return of evolved decomposition products to the specimen. In a cold wall chamber, evolved decomposition products would condense on the wall and remain there throughout the test.

2. It is felt that some of the materials tested in this program would perform considerably better at higher vacuum under the same thermal conditions. Tests to confirm this conjecture should be made.

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1. Mattice, James J. The Vacuum - Thermal Stability of Organic Coating Materials, WADD Tech. Report 60-126, Aug. (1960)

2. Jaffe, L. D. Effects of Space Environment Upon Plastics and Elastomers, NASA Tech. Report No. 32 - 176, Nov. (1961)

3. Warren, J. M. Investigation of Inorganic-Organic High Temperature Adhesive Materials, General Dynamics/Fort Worth, Technical Report ERR-FW-060 (1961)

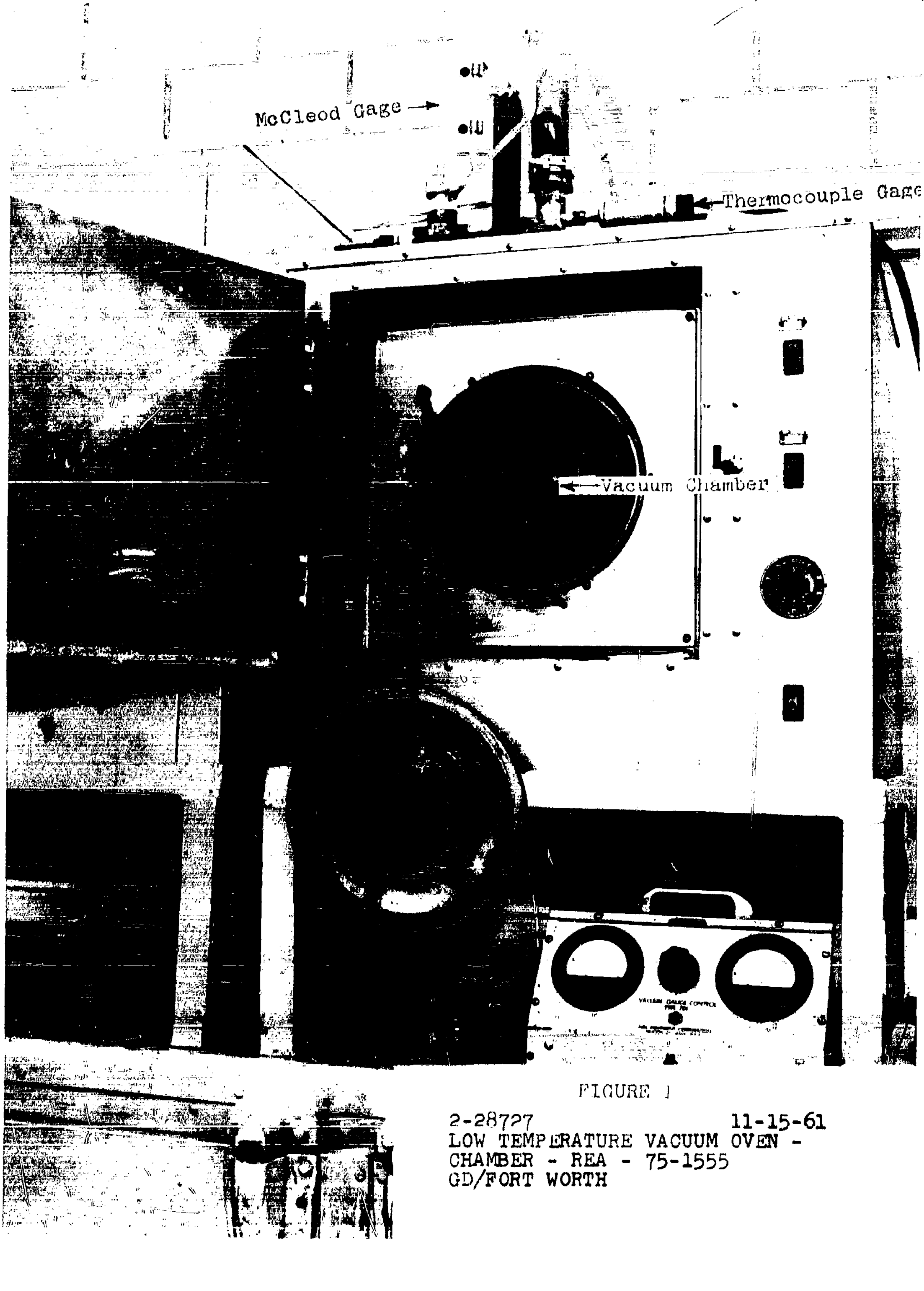

FIGURE 1

2-28727 11-15-61
LOW TEMPERATURE VACUUM OVEN - CHAMBER - REA - 75-1555
GD/FORT WORTH

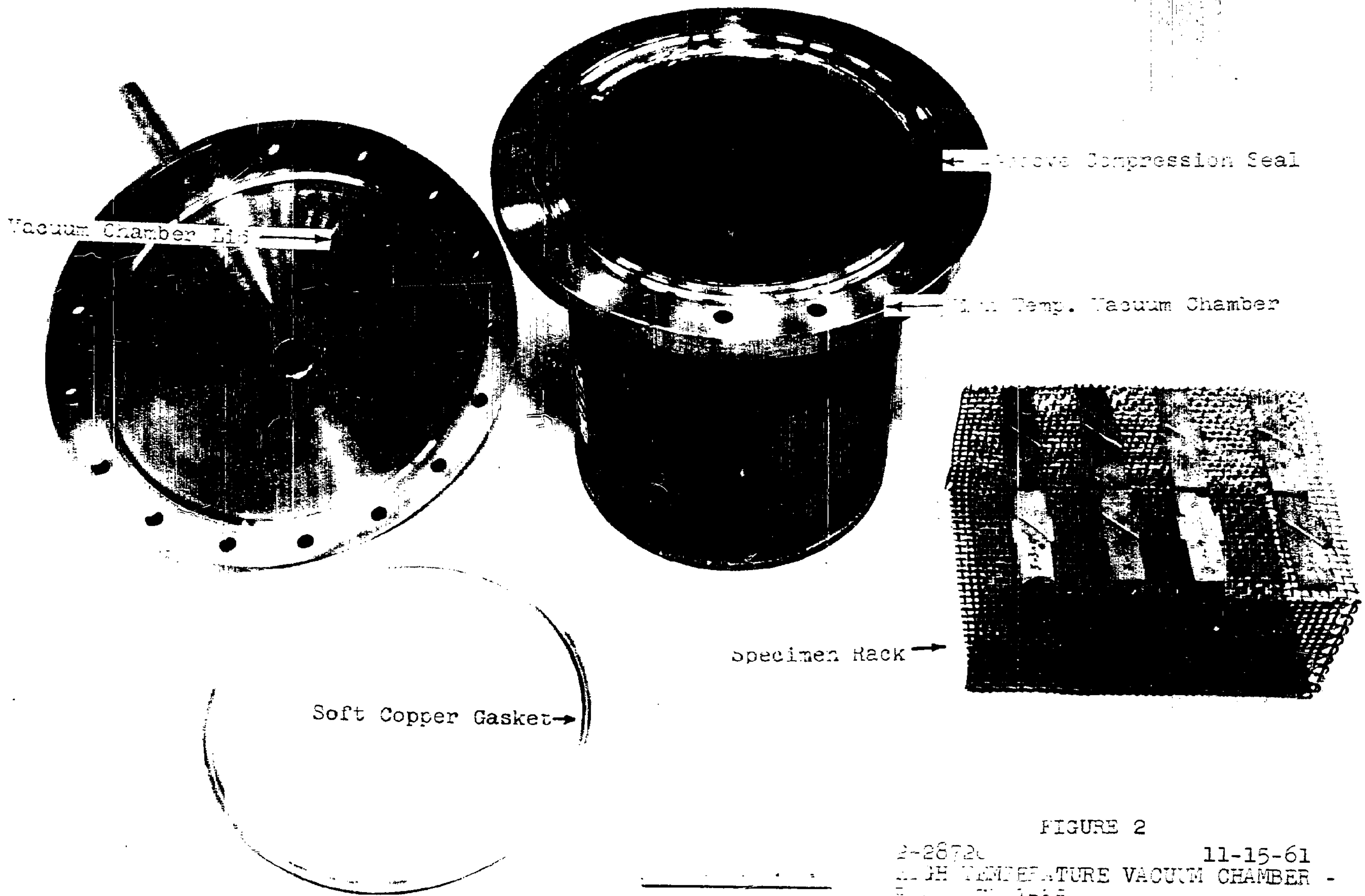

FIGURE 2

11-15-61

TURE VACUUM CHAMBER -

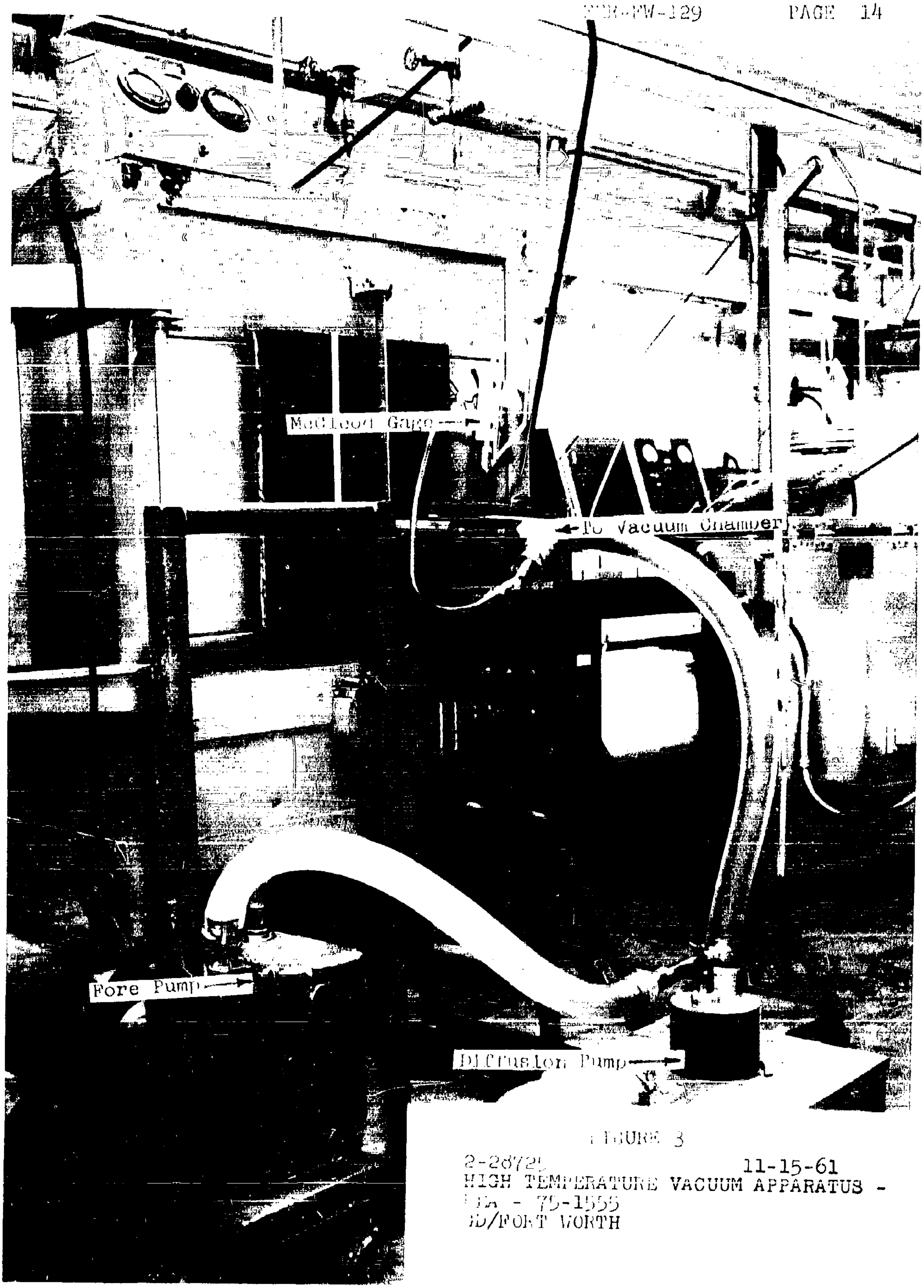

FIGURE 3

2-28725 11-15-61
HIGH TEMPERATURE VACUUM APPARATUS -
FA - 75-1555
GD/FORT WORTH

TABLE I

SUMMARY OF AVERAGE RESULTS OF THE EFFECTS OF SPACE ENVIRONMENTAL CONDITIONS OF TEMPERATURES UP TO 700°F AND VACUUM TO 1 X 10^{-3} mm Hg

Material	Average % Weight Loss After							
	300 hrs. @ 200°F 1 x 10^{-3} mm Hg		300 hrs. @ 200°F no vacuum		10 hrs. @ 700°F 2.5 x 10^{-3} mm Hg		10 hrs @ 700°F no vacuum	
	Uncovered	Covered	Uncovered	Covered	Uncovered	Covered	Uncovered	Covered
Q-2-0103	1.5	1.2	1.9	1.9	13.2	16.0	25.8	31.2
PX - 1040	1.0	-	2.1	-	5.4*	-	31.5*	-
Epon 931	0.6	0.12	0.2	0.28	46.0	45.0	74.9	70.5
Dynabond 118	0.1	0.25	0.1	0.14	4.3	3.9	17.2	13.6
Fairprene 5159	0.6	-	0.2	-	28.7	-	Ash	-

* These specimens aged at 400°F for 300 hours and approximately 1.5 x 10^{-3} mm Hg vacuum.

Note: For explanation of "covered" and "uncovered" see pg. 29.

TABLE I
(continued)

Material	Average Peel Strength In lbs/in. Width* After				Average Shear Strength In psi* After			
	300 hrs. @ 200°F		10 hrs. @ 700°F		300 hrs. @ 200°F		10 hrs. @ 700°F	
	1×10^{-3} mm.Hg. Vac.	No Vac.	2.5×10^{-3} mm.Hg. Vac.	No Vac.	1×10^{-3} mm.Hg. Vac.	No Vac.	2.5×10^{-3} mm.Hg. Vac.	No Vac.
Silicone Rubber Sealant, Dow Corning Q-2-0103 (Original peel = 6.0 lbs/in. width @ R.T.)	10.0	7.0	3.5	decomposed	-	-	-	-
Butyl General Purpose Cement, Permalastic PX - 1040 (Original peel = 3.5 lbs/in. width @ R.T.)	3.5	3.0	3.0**	0.5**	-	-	-	-
Room Temp. Curing Epoxy Adhesive Shell Epon 931 (Original shear strength = 2089 psi @ R.T.)	-	-	-	-	2282	2225	225	fell apart
Phenylsilane based Adhesive, Dynabond 118 (Original shear strength = 2292 psi @ R.T.)	-	-	-	-	2292	2308	785	fell apart
Viton Rubber Cement, Dupont Fairprene 5159 (Original peel = 8.0 lbs/in. width @ R.T.)	5.0	4.0	decomposed	decomposed	-	-	-	-

* Specimens tested at room temperature after being subjected to vacuum-temp. or temp. only.

** These specimens aged at 400°F for 300 hours and approximately 1.5×10^{-3} mm Hg vacuum.

TABLE II

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 200°F FOR 300 HOURS WITH A VACUUM OF 1 X 10^{-3} mm Hg

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss	Percent Loss	Average Percent Loss
Q-2-0103	DC-1	1"x1"x	2.3383	.03320	1.4	
	DC-2	.096"	2.3726	.03720	1.6	1.5
	DC-3		2.3964	.03370	1.4	
PX-1040	B-1	1"x2"x	0.0878	.0005	0.6	
	B-2	.0013"	0.0935	.0010	1.1	1.0
	B-3		0.0947	.0014	1.5	
Epon 931	S-1	1"x1"x	2.2376	.0185	0.8	
	S-2	.096"	2.2858	.0085	0.4	0.6
	S-3		2.2304	.0108	0.5	
Dyna-bond 118	D-1	1"x1"x	1.0739	.0005	0.05	
	D-2	.041"	0.9833	.0015	0.2	0.1
	D-3		1.0375	.0013	0.1	
Fair - prene 5159	V-1	1"x2"x	1.0581	.0067	0.6	
	V-2	.0025"	1.0153	.0061	0.6	0.6
	V-3		1.0796	.0057	0.5	

TABLE III

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 200°F FOR 300 HOURS WITHOUT VACUUM

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss	Percent Loss	Average Percent Loss
Q-2-0103	DC-4	1"x1"x	2.3425	.0450	1.9	
	DC-5	.096	2.2891	.0431	1.9	1.9
	DC-6		2.3428	.0447	1.9	
PX-1040	B-4	1"x2"x	.0765	.0014	1.8	
	B-5	.0013	.0997	.0024	2.4	2.1
	B-6		.0806	.0017	2.1	
Epon 931	S-4	1"x1"x	2.3233	.0039	.17	
	S-5	.096	2.2826	.0046	.20	.20
	S-6		2.2828	.0049	.22	
Dynabond 118	D-4	1"x1"x	1.0895	.0001	.05	
	D-5	.041"	1.0554	.0007	.15	.10
	D-6		0.9898	.0004	.13	
Fairprene	U-4	1"x2"x	1.2354	.0036	.29	
	U-5	.0025	1.0894	.0020	.18	.20
	U-6		0.8968	.0017	.19	

TABLE IV

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 700°F FOR 10 HOURS WITH A VACUUM OF 2.5 X 10^{-3} mm Hg

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss (gms.)	Percent Loss	Average Percent Loss
Q-2-0103	DC-7	1"x1"x	2.3706	.3125	13.2	
	DC-8	.096	2.3722	.3136	13.2	13.2
	DC-9		2.2778	.3019	13.3	
PX-1040	B-7*	1"x2"x	.0750	.0045	6.0	
	B-8*	.0013	.0699	.0040	5.7	5.4
	B-9*		.1035	.0047	4.5	
Epon 931	S-7	1"x2"x	2.2305	1.0120	45.4	
	S-8	.096	2.2034	1.0011	45.4	46.0
	S-9		2.2665	1.0711	47.2	
Dynabond 118	D-7	1"x1"	1.0427	.0439	4.2	
	D-8	.041	1.0450	.0455	4.4	4.3
	D-9		1.0097	.0436	4.3	
Fairprene 5159	V-7	1"x2"	1.6345	0.5190	31.8	
	V-8	.0025	1.5668	0.4264	27.2	28.7
	V-9		1.2908	0.3515	27.2	

* These specimens were subjected to 400°F for 300 hours and approximately 1.5 x 10^{-3} mm Hg vacuum.

TABLE V

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 700°F FOR 10 HOURS WITHOUT VACUUM

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss (gms.)	Percent Loss	Average Percent Loss
Q-2-0103	DC-10	1"x2"x	2.3566	.6385	27.0	
	DC-11	.096	2.3955	.6394	26.7	25.8
	DC-12		2.4152	.5708	23.6	(mtl. spongy)
PX-1040	B-10*	1"x2"x	.0559	.0164	29.3	
	B-11*	.0013	.0372	.0152	40.9	31.5
	B-12*		.0678	.0164	24.2	
Epon 931	S-10	1"x1"x	2.2570	1.708	75.7	
	S-11	.096	2.2250	1.690	75.9	74.9
	S-12		2.2827	1.667	73.0	(ash remained)
Dynabond 118	D-10	1"x1"x	1.0250	0.1710	16.7	
	D-11	.041	0.9813	0.1698	17.3	17.2
	D-12		0.9611	0.1695	17.6	
Fairprene 5159	V-10	1"x1"x	1.8132	Specimens completely decomposed		
	V-11	.0025	1.6945			
	V-12		1.6612			

* These specimens were subjected to 400°F for 300 hours without vacuum.

TABLE VI

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 200°F FOR 300 HOURS WITH AND WITHOUT 1 X 10^{-3} mm Hg VACUUM - MATERIAL SANDWICHED BETWEEN TWO PIECES OF .040" RS 140 TITANIUM

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss (gms.)	Percent Loss	Average Percent Loss
Q-2-0103	DC-1	1"x1"x .096"	0.1540	0.0016	1.04	
	DC-2		0.1663	0.0019	1.14	1.2*
	DC-3		0.1742	0.0025	1.44	
	DC-4		0.2033	0.0040	1.97	
	DC-5		0.1751	0.0031	1.77	1.9
	DC-6		0.1561	0.0030	1.92	
Epon 931	S-1	1"x1"x .096"	0.2205	0.0002	0.091	
	S-2		0.2371	0.0003	0.127	0.12*
	S-3		0.2255	0.0003	0.133	
	S-4		0.3125	0.0004	0.128	
	S-5		0.2259	0.0007	0.310	0.28
	S-6		0.3263	0.0013	0.398	
Dynabond 118	D-1	1"x1"x .040"	0.1097	0.0001	0.273	
	D-2		0.1010	0.0002	0.297	0.25*
	D-3		0.1113	0.0003	0.180	
	D-4		0.1089	0.0001	0.092	
	D-5		0.1075	0.0002	0.186	0.14
	D-6		-.- -			

* Average of specimens subjected to approximately 1 x 10^{-3} mm Hg vacuum and temperature - other specimens subjected to temperature only.

TABLE VII

WEIGHT LOSS OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 200°F FOR 10 HOURS WITH AND WITHOUT 1 x 10^{-3} mm Hg VACUUM - MATERIAL SANDWICHED BETWEEN TWO PIECES OF .040" RS 140 TITANIUM

Material	Spec. No.	Approx. Size	Wt. Prior To Test (gms.)	Weight Loss (gms.)	Percent Loss	Average Percent Loss
Q-2-0103	DC-7	1"x1"x	0.2395	0.0347	14.49	
	DC-8	.096"	0.1692	0.0297	17.55	16.0**
	DC-9		-	-	-	
	DC-10		0.5393	0.1550	28.74	
	DC-11		0.6175	0.1219	35.40	31.2
	DC-12		0.5696	0.1669	29.30	
Epon 931	S-7	1"x1"x	0.1437	0.1090	75.85*	
	S-8	.096"	0.2234	0.1013	45.34	45.0**
	S-9		0.2972	0.1331	44.78	
	S-10		0.1981	0.1405	70.92	
	S-11		0.2492	0.1809	72.59	70.5
	S-12		0.3221	0.2194	68.12	
Dynabond 118	D-7	1"x1"x	-	-	-	
	D-8	.041"	0.1096	0.0042	3.83	3.9**
	D-9		0.1018	0.0041	4.03	
	D-10		0.1033	0.0137	13.26	
	D-11		0.1036	0.0133	12.84	13.6
	D-12		0.1107	0.0163	14.73	

* This value not included in average

** Average of specimens subjected to approximately 1 x 10^{-3} mm Hg vacuum and temperature. Other specimens aged at temperature only.

TABLE VIII

LOSS OF SHEAR STRENGTH OF EPON 931 AND DYNABOND 118 SUBJECTED TO AGING AT 200°F FOR 300 HOURS WITH AND WITHOUT 1.0 x 10^{-3} mm Hg VACUUM - TESTED AT ROOM TEMPERATURE AT 650 LBS/MIN. LOAD RATE

Specimen Number & Material	Area (Sq.In.)	Load To Failure lbs.	Stress To Failure psi	% Coh Failure	Average psi	Vacuum mm Hg
Epon 931						
S-1-6	.532	1200	2256	0		1x10^{-3}
S-1-3	.510	1285	2520	5	2282	↕
S-2-2	.521	1210	2322	5		
S-3-2	.512	1040	2031	5		1x10^{-3}
S-2-4	.504	1225	2431	10		None
S-3-1	.499	1135	2275	5	2225	↕
S-3-5	.508	985	1939	5		
S-3-7	.503	1135	2256	10		None
S-2-6	.505	1055	2089	5	2089	None-Control
Dynabond 118						
D-2-5	.541	1260	2329	90		1x10^{-3}
D-2-6	.536	1205	2248	80		↕
D-3-1	.520	1160	2231	90	2292	
D-3-2	.528	1245	2358	95		1x10^{-3}
D-1-5	.547	1260	2304	90		None
D-2-3	.545	1245	2284	85		↕
D-2-4	.543	1260	2320	80	2308	
D-3-7	.536	1245	2323	85		None
D-3-6	.526	1205	2291	80	2292	None-Control

TABLE IX

LOSS OF SHEAR STRENGTH OF EPON 931 AND DYNABOND 118 SUBJECTED TO AGING AT 700°F FOR 10 HOURS WITH AND WITHOUT 2.5×10^{-3} mm Hg VACUUM - TESTED AT ROOM TEMPERATURE AT 650 LBS/MIN LOAD RATE

Specimen Number & Material	Area (Sq.In.)	Load To Failure lbs.	Stress To Failure psi	% Coh. Failure	Average psi	Vacuum mm Hg
Epon 931						
S-1-2	-	Specimens broke on removal from jig				2.5×10^{-3}
S-3-6	-					
S-3-4	-					
S-2-7	.548	.123	225	0	225	
S-1-4	-	Specimens fell apart in aging				None
S-1-7	-					
S-2-5	-					
S-2-6	-					
Dynabond 118						
D-3-4	.535	420	785	70	785	2.5×10^{-3}
D-1-1	.537	520	968	60		
D-2-1	.531	410	772	75		
D-2-2	.534	330	618	70		
D-3-5	-	Specimens fell apart in aging				None
D-3-3	-					
D-1-4	-					
D-1-2	-					

TABLE X

PEEL STRENGTH OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 200°F FOR 300 HOURS WITH AND WITHOUT 1×10^{-3} mm Hg VACUUM - SPECIMENS TESTED AT ROOM TEMPERATURE AND 2"/MIN. PEEL RATE

Specimen* Number & Material	Av. Load To Failure (Lbs/In.Width)	Type Failure	Av. Peel Strength (Lbs/In.Width)	Vacuum (mm Hg)
Q-2-0103				
DC-1	10.0	75% Adh. to wire	10.0	$1x10^{-3}$
DC-2	10.0	70% Adh. to wire		
DC-3	9.5	50% Adh. to wire		
DC-4	6.0	80% Adh. to wire	7.0	None
DC-5	7.5	75% Adh. to wire		None
DC-10	6.0	80% Adh. to wire	6.0	None (Control)
PX-1040				
B-1	4.0	95% Adh. to wire	3.5	$1x10^{-3}$
B-2	3.0	80% Adh. to wire		
B-3	3.5	50% Adh. to wire		
B-4	3.5	90% Adh. to wire	3.0	None
B-5	3.0	80% Adh. to wire		None
B-6	3.5	75% Adh. to wire	3.5	None (Control)
Fairprene 5159				
V-1	6.0	50% Adh. to wire	5.0	$1x10^{-3}$
V-2	6.5	60% Adh. to wire		
V-4	3.5	10% Adh. to wire		
V-5	2.5	20% Adh. to wire	4.0	None
V-6	5.0	15% Adh. to wire		None
V-3	8.0	50% Adh. to wire	8.0	None (Control)

* All specimens 1" width.

TABLE XI

PEEL STRENGTH OF DIFFERENT MATERIALS SUBJECTED TO AGING AT 700°F FOR 10 HOURS WITH AND WITHOUT 2.5×10^{-3} mm Hg VACUUM - SPECIMENS TESTED AT ROOM TEMPERATURE AND 2"/MIN. PEEL RATE

Specimen* Number & Material	Load To Failure (Lbs/In.Width)	Type Failure	Av. Peel Strength (Lbs/In.Width)	Vacuum (mm Hg)
Q-2-0103				
DC-8	3.0	10% Adh.	3.5	2.5×10^{-3}
DC-9	4.0	25% to Wire		2.5×10^{-3}
DC-6	Material lost all adhesion		-	None
DC-7	Material lost all adhesion		-	None
PX-1040				
B-8**	3.0	80% Adh.		
B-9**	4.0	75% to	3.0	1.5×10^{-3}
B-10**	3.0	60% Metal		
B-6**	0.6	80% Co-	0.5	None
B-7**	0.4	90% hesive		None
Fairprene 5159				
V-8	Material hard and brittle - lost all adhesion			1.5×10^{-3}
V-10				1.5×10^{-3}
V-7	Specimen fell apart			None
V-9	Specimen fell apart			None

* All specimen 1 inch width.

** These specimens aged at 400°F for 300 hours with and without 1.5×10^{-3} mm Hg in lieu of 700°F.

APPENDIX I

EXPERIMENTAL PROCEDURES

A. Modification of Vacuum Chamber for 200° and 400°F Tests

A small vacuum oven with a 9" diameter by 10" length vacuum chamber was modified for use in this research program by increasing the vacuum inlet tube diameter and the fabrication of a silicone rubber seal for the door. The system was attached to a one inch oil diffusion pump and a Welch Duoseal fore pump by heavy Tygon vacuum tubing. The specimens were placed flat on wire racks inside the chamber, the racks being approximately 5" apart. Figure 1 is a photograph of the vacuum oven setup. The same diffusion and fore pumps were used as shown in Figure 3. Vacuum was measured by a thermocouple gage and McCleod gage.

B. Fabrication of 700°F Vacuum Chamber

A vacuum chamber was designed and fabricated from stainless steel with a compression-shear type soft copper seal, having an inside diameter of approximately 10.5" by 12" in length as shown in Figure 2. The setup with chamber in the 700°F oven is shown in Figure 3. Vacuum was measured by a thermocouple gage and McCleod gage.

C. <u>Cleaning of Titanium Metal and Monel Wire Screen</u>

1. All RS 140 titanium used in this test was cleaned as follows:

   a. Immersed for 30-60 seconds at room temperature in a pickle solution with the following composition:

      1) Nitric acid, 15% by volume

      2) Hydrofluoric acid, 3% by volume

      3) Tap water, 82% by volume

   b. Rinsed in clean running tap water

   c. Immersed in the following solution for 2 minutes at room temperature:

      1) Trisodium phosphate, 50 grams/liter

      2) Sodium fluoride, 8.9 grams/liter

      3) Hydrofluoric acid, 31 grams/liter

      4) Tap water, enough to make one liter solution

   d. Rinsed in running tap water

   e. Immersed in 130-150°F tap water for 15 minutes

   f. Rinsed with a spray of distilled water and then dried in 160°F drying oven.

2. The Monel wire screen was cleaned as follows:

   a. MEK wiped

   b. Cleaned with A-3 cleaner

   c. Spray-rinsed with distilled water

   d. Dried in 160°F oven.

D. Preparation of Specimens

1. Preparation of Silicone Sealant Q-2-0103 Specimens

Ten grams of XY-115 catalyst was mixed thoroughly with 100 grams of Q-2-0103 with a spatula until a uniform color was obtained.

a. Peel Specimens

An 0.060" coat of the mixed sealant was then applied to 1" x 8" x 0.040" RS-140 titanium strips previously cleaned and then primed with QA-2-1011. A 1" strip of Monel wire mesh, previously cleaned with A-3 cleaner and primed with QA-2-1011, was placed in contact with the sealant. Another layer of sealant, approximately .040" thick, was applied over the Monel wire screen which was aligned with the titanium strip. The specimens were then placed in a bonding press between two layers of 3 mil Mylar film and compressed to approximately 80 mils sealant-wire thickness by using spacers to maintain thickness. Pressure was maintained for about 4 hours until sealant had set. The specimens were allowed to cure 72 hours before testing. A total of 10 specimens were prepared with a peel area of approximately 1" x 6".

b. Uncovered and Covered Weight Loss Specimens

The terms uncovered and covered as used with all weight loss specimens and referred to as such in this report, are defined as follows: the material cured and not sandwiched between titanium is referred to as uncovered and the material sandwiched between titanium as covered.

Uncovered weight loss specimens were prepared by compressing a portion of catalyzed sealant between two layers of Mylar film to approximately 96 mils and allowing the material to cure for 24 hours under pressure before being cut into 1" x 1" x .096" pieces and removing the Mylar film. Another portion of the catalyzed sealant was spread on 1" x 1" x .040" pieces of titanium cleaned per C.1. above. Similar 1" x 1" x .040" pieces of titanium were placed on top to form a sandwich type specimen such that a sealant thickness of approximately .025" is obtained.

The specimens were allowed to cure for 72 hours prior to test and this type specimen is referred to as (covered) elsewhere in this report

2. Preparation of Butyl Rubber Cement PX-1040 Specimens

a. Peel Specimens

Permalastic PX-1040 butyl adhesive was employed in fabrication of 10 peel specimens. Titanium strips 1" x 8" were prepared as described in C.1 and two heavy coats of Permalastic PX-1040 cement were applied to the titanium strips, allowing 30 minute drying time between coats. Glass fabric 183, Volan A finish, was cut into 1" x 14" strips and one coat of Permalastic cement was used to bond 8" of one end of two strips of 183 cloth together. Two heavy coats of Permalastic was then applied to the 1" x 8" surface of the two laminated strips, allowing 30 minutes drying time between coats. While the last coat was still tacky the two-ply laminate of cloth was pressed in firm contact with the mating titanium strip giving a total adhesive thickness of about .008". Two plies of cloth were used to prevent adhesive failure to the cloth.

b. Weight Loss Specimens

Weight loss specimens were fabricated by coating .004" aluminum foil with the Permalastic PX-1040 to a dry thickness of 2.5 mils. When the cement had dried tack free, the foil coated with the butyl cement was cut into strips 1" x 2" which were then air dried 72 hours before testing.

3. Preparation of Epon 931 Adhesive Specimens

a. Lapshear Specimens

The Epon 931 adhesive, part A, was mixed thoroughly with part B in the ratio of 100 parts A to 9 parts B. The adhesive was applied to cleaned RS-140 (4" x 9" x .064") titanium cutaway panels and the panels fabricated into lapshear pads with 1/2" overlap. The glueline thickness was controlled with glass threads laid in the center of each specimen parallel to the shearing force to be applied when tested. Specimens were bonded at 5 psi in the bonding press for 24 hours at R.T.* and allowed to cure an additional 48 hours at R. T. without pressure.

b. Weight Loss Specimens

Weight loss specimens were fabricated by placing a portion of the catalyzed adhesive between two sheets of Mylar film and compressing it, as regulated by spacers, to approximately .096" thickness. Specimens were then cut, after curing of material, to 1" x 1" x .096". Also 1" x 1" weight loss specimens were cut from pads prepared by placing catalyzed adhesive between two 4" x 4" x .040" pieces of cleaned RS 140 titanium and bonding in a bonding press for 24 hours at room temperature.

4. Preparing of Dynabond 118 Adhesive Specimens

a. Lapshear Specimens

General Dynamics/Fort Worth Engineering Chemistry Laboratory developed adhesive[3] was applied to one faying surface of cleaned titanium cutaway skins as described in D.3 for Epon 931 specimens. The lapshear pads were placed on a lapshear layup pad and bonded in a steam heated bonding press for 2 hours at 350°F and 100 psi pressure.

b. Weight Loss Specimens

Weight loss specimens were prepared by bonding 8

* R.T. = Room Temperature

layers of the adhesive between two pieces of .002" aluminum foil resulting in a thickness of approximately .040". Covered weight loss specimens were prepared as described in 3.b. above except the bonding conditions were 100 psi at 350°F for 2 hours.

5. Preparation of Viton Cement Fairprene 5159 Specimens

a. Peel Specimens

DuPont's Viton cement was catalyzed with 1% of catalyst and mixed thoroughly. Cleaned strips of titanium, 1" x 8" x .040", and Monel screen, 1" x 14", were primed with a light coat of Chemlok 607. After allowing the prime to dry for one hour, the catalyzed cement was applied to both Monel and titanium strips over an area of approximately 1" x 6" and the coating allowed to dry for 30 minutes before application of another coat. Six coats were applied. After the last coat was applied and dried for 30 minutes, the surfaces were reactivated with MEK and the Monel and titanium strips were pressed together in a bonding press. The specimens were allowed to air cure for 48 hours at room temperature followed by a post cure of 8 hours at 220°F, preceded by a 4 hour rise from room temperature to 220°F.

b. Weight Loss Specimens

Weight loss specimens were prepared by applying the catalyzed Viton cement to Chemlok 607 primed .004" aluminum foil with a resulting film thickness of

.010" - .015". The same cure was used as for the peel specimens.

E. Subjecting Specimens to Simulated Space Environments

1. Specimens tested at 200°F for 300 hours in air and at a pressure of 1×10^{-3} mm Hg (mercury)

The vacuum pressures in this report are all listed in mm of Hg and are considered to be approximate values whether stated or not.

Three specimens of all types described in D. above except 6 of each type shear specimens were placed in a vacuum chamber as described in A. and a vacuum was obtained of approximately 1×10^{-3} mm Hg by using a fore pump and a 1" diffusion pump. The setup was allowed to outgas for 24 hours after which the oven was turned on and the temperature gradually increased to 200°F in approximately 4 hours. As controls, a similar number of specimens were placed in an oven at 200°F without vacuum. The type vacuum setup for the 200°F test for 300 hours is depicted in Figure 1.

2. Specimens aged at 400°F for 200 hours at 1.5×10^{-3} mm Hg vacuum

Following the 200°F test, another run was made at 400°F with only the butyl rubber cement specimens being tested at this temperature. The same general procedure was followed including specimens aged at 400°F without vacuum.

3. Specimens Aged at 700°F for 10 Hours and 2.5×10^{-3} mm Hg Vacuum

Three specimens of each material and type described in D. above were placed in a stainless steel vacuum chamber as shown in Figure 2. All specimens were tied in place to the racks with stainless steel wire and the racks positioned in the chamber. A copper gasket was placed in the "V" groove and the lid was bolted in position using a torque wrench. The torque applied was 100 ft. lbs. The chamber was then checked for leaks and placed in the high temperature oven in a setup as shown in Figure 3. A vacuum was applied and the system allowed to outgas over night. The oven was then turned on and the test temperature was reached in approximately 3 hours. Temperature was monitored by a thermocouple attached to the outside chamber wall and was maintained at 700°F for 10 hours and 2.5×10^{-3} mm Hg. Control specimens were run at 700°F without vacuum.

F. Physical Testing of Specimens

1. Determination of Weight Loss

Covered and uncovered specimens were weighed on an analytical balance before and after being subjected to environmental conditions. The weight loss was used to determine the percent weight loss for each simulated space environment. The weight loss of the covered specimens was determined after space environmental testing by removing the adhesive material from the 1" x 1" x .040" pieces of titanium in concentrated nitric acid and reweighing the metal pieces. Titanium is relatively passive in concentrated nitric acid and the error here was insignificant due to loss of metal.

$$\frac{W2 - T}{W_1 - T} \times 100 = \% \text{ wt. loss}$$

W2 = Weight of specimen after environmental exposure

W1 = Weight of specimen prior to environmental exposure

T = Weight of both .040" titanium skins stripped of material.

2. Testing Peel Specimens

Peel specimens obtained from the 3 simulated space environmental tests with the controls (no vacuum) and room temperature non-aged controls were tested in the Scott Tensile test machine using a 180° type peel at 2 inch head travel/minute. Peel strength was obtained as lbs/inch width.

3. Testing Lapshear Specimens

Lapshear specimens were cut up into individual specimens prior to vacuum - temperature exposures. After these exposures the specimens were tested along with controls (no vacuum) and room temperature (no age) controls in shear using a loading rate of 650 lbs. per minute and the shear strength expressed in psi.

APPENDIX II

DESCRIPTION OF MATERIALS AND EQUIPMENT

A. MATERIALS

	MATERIALS	SOURCE
1.	Silicone Rubber Sealant Q-2-0103 with Q-2-0103-2 Catalyst (Lot 060207)	Dow Corning Corporation Midland, Michigan
2.	Butyl General Purpose Cement PX-1040	Permalastic Products Detroit, Michigan
3.	Epoxy Room Temperature Curing Adhesive Epon 931	Shell Chemical Company New York, N. Y.
4.	Phenylsilane Based Adhesive - Dynabond 118	General Dynamics/Ft.Worth Fort Worth, Texas
5.	Viton Cement Fairprene 5159	E. I. duPont de Nemours & Co. (Incorporated) Fabrics Division Fairfield, Conn.
6.	Chemlock 607	Lord Mfg. Company Erie, Pennsylvania
7.	R.S. - 140 Titanium Sheet .040" x 1" x 8" and .040" x 4" x 4"	Republic Steel Corporation Masillon, Ohio
8.	Monel Screen Wire	Phoenix Wire Works, Inc. Detroit, Michigan
9.	Aluminum Foil .004" Thick	General Dynamics/Fort Worth Stock
10.	Tygon Tubing	Fisher Scientific Company, Fort Worth, Texas

B. EQUIPMENT	SOURCE
1. Vacuum Fore Pump	W. M. Welch Mfg. Company Chicago, Ill.
2. Vacuum Diffusion Pump (1 inch)	Consolidated Vacuum Corp. Rochester, New York
3. Cooley RB-2 Furnace	Cooley Electric Mfg. Corp. Indianapolis, Indiana
4. Vacuum Oven (Modified by GD/FW Eng. Test Lab.)	Precision Scientific Co. Chicago, Ill.
5. Stainless Steel Vacuum Chamber	General Dynamics/Fort Worth Designed and Fabricated
6. Steam Heated Bonding Press	Pasadena Hydraulics, Inc. El Monte, California
7. McCleod Gage	Scientific Glass Apparatus Co., Inc. Bloomfield, New Jersey