This ATM presents the results of the S-13G thermal coating investigation conducted in support of the ALSEP Flight 1 central station adhesion paint failure. The report includes a summary of the events that occurred during the investigation, a comprehesive review of S-13G coating application history at BxA, the results of BxA and IITRI coupon tests to determine the cause for the loss of adhesion and establish corrective action, and recommendations for changes to the manufacturing process procedure for subsequent ALSEP painted hardware.

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1.0 SUMMARY OF EVENTS FOR FLIGHT 1 S-13G THERMAL COATING ADHESION INVESTIGATION

A brief description of the events leading to the investigation by Bendix and IITRI of S-13G thermal coating thermal-vacuum adhesion characteristics is included herein for background purposes. This description covers a review of various ALSEP Central Station thermal-vacuum tests conducted prior to the S-13G adhesion failure experienced on the Flight 1 acceptance test and traces the S-13G application history of each unit through the Flight 2 acceptance test.

The ALSEP Flight 1 unit thermal-vacuum acceptance testing was conducted in the BxA 20' x 27' test chamber during the period from 23 June 1968 through 3 July 1968 and was subjected to an accelerated simulated lunar cycle environment. The first portion of the acceptance test simulated lunar night for radiometer reference purposes with the Central Station operating under a lunar night power schedule. In the transition to simulated lunar morning, it was observed that adhesion failure of the S-13G thermal coating had occurred on the sunshield and one side of the primary structure of the Central Station. After removal from the test chamber, the Central Station was examined in an attempt to determine possible causes of failure with the following observations.

1. The adhesion failure occurred at the primer/substrate interface on both the sunshield and primary structure.

2. Failure had occurred at cryogenic temperature in a simulated lunar night environment.

3. The GE SS-4044 primer and/or S-13G thermal coating batches were suspect since Proto A and Qual SA models both had completed thermal-vacuum tests with no evidence of S-13G adhesion failure.

4. Variations in S-13G application technique were suspect since Flight 1 was completely coated by Bendix personnel whereas IITRI had coated all of Proto A and part of Qual SA units.

TABLE I SUMMARY OF ALSEP CENTRAL STATION S-13G COATED SURFACES

						G. E. SS-4044 Primer							S-13G Thermal Coating									
Unit	Item	Drwg. #233	Serial No.	Coated By	Date Cleaned	Date Primed	Time	Batch No.	Man Date	Thick Mils	P. O or RIR	Exp. Date	Date Coated	Time	Batch No.	Man Date	Thick Mils	P. O. or RIR	Exp. Date	O. R's	Pig't. Batch and Date	Remarks
Proto A	T. Plate	0351-1	1	IITRI	6-1-67	6-1-67				O. K.			6-2-67		301	5-23-67	O. K.				298 6-23-67	
	T. Plate Repaint	0351-1	1	IITRI	7-18-67	7-18-67				0.3-0.6			7-18-67		320	7-15-67	9-12				311 6-19-67	
	Primary Structure	0203	1	IITRI	7-18-67	7-18-67				0.3-0.6			7-18-67		320	7-15-67	9-12				311 6-29-67	
	Sunshield	0228	1	IITRI	7-18-67	7-18-67				0.3-0.6			7-18-67		320	7-15-67	9-12				311 6-29-67	
Qual SA	T. Plate	0351-2	2	IITRI	7-18-67	7-18-67				0.3-0.6			7-18-67		320	7-15-67	9-12				311 6-29-67	
	Primary Structure	5088	3	BxA	1-24-68	1-24-68	11:30A			O. K.			1-24-68 1-25-68	2:00P 3:00P 4:30P 10:00A 1:30P 2:30P	474	1-4-68	12-15		2-29-68		473 1-2-68	Catalyst Exp. 3-7-68
	Sunshield	5136	3	BxA	2-11-68	2-12-68	3:00P			O. K.			2-13-68 2-14-68	1:00P 1:00P	474 524		O. K.	78408 78469	2-29-68 4-7-68			Catalyst Exp. 3-7-68 RIR 78469
Flight 1	T. Plate	0351-2	3	BxA		2-23-68	2:30P		2-9-68	0.4-0.5	78469	2-7-68	2-23-68 2-24-68	4:30P 8:30A 1:30P	524	2-7-68	14-15			7162	507, 508 1-30-68	
	Primary Structure	5088	4	BxA	4-9-68	4-9-68	10:00A			O. K	G8699	2-7-69	4-9-68 4-10-68	2:00P 4:00P 9:30A		4-2-68	10-12		6-1-68			
	Primary Structure Repaint	5088	4	BxA	7-8-68	7-8-68	6:30P	349		0.5			7-9-68		742	7-2-68	O. K.		8-2-68			Surface Abraded
	Sunshield	5136	4	BxA	4-26-68	4-26-68	3:00P	316	2-9-68	O. K.	78469	2-7-69	4-26-68 4-27-68	5:30P 10:00A 3:00P			O. K.		6-1-68			
	Sunshield Repaint	5136	4	BxA	7-8-68	7-8-68	6:30P	349		0.5			7-9-68		742	7-2-68	O. K.		8-2-68			Surface Abraded
Flight 2	T. Plate	0351-2	4	BxA	5-31-68	5-31-68		64159 AH		0.4-0.6		3-24-69	5-31-68	2:00P	647	4-2-68	O. K.		6-1-68			
	Primary Structure	5088	5	BxA	6-13-68	6-13-68	4:00P	64159 AH		0.6		3-29-69	6-14-68	9:30A 11:00A 1:10P	724	6-10-68	10-12					
	Sunshield	5136	5	BxA	7-21-68	7-21-68	10:00P	349	7-17-68	0.25 ± 0.1	78959	10-18-68	7-22-68	10:40A 1:15P 2:45P	757	7-18-68	O. K.		8-18-68			Catalyst Exp. 8-18-68 RIR 78959

NOTE: The notation "O. K." for thickness value indicates that thickness was accepted by inspection per M. P. -64 but actual value was not recorded.

A review of records involving all S-13G coated units was made in an effort to determine the exact status of each unit up to the time of the Flight 1 test failure in anticipation of obtaining information that would provide a valid reason for the adhesion failure. This preliminary investigation did not reveal any evidence indicating that improper application of the S-13G coating had occurred on the Flight 1 test or that defective primer or S-13G coating hatches were used. The results of this investigation of records is presented in Table 1 with information gaps readily apparent. The notation of "OK" for primer and S-13G thickness values only indicates that the thickness was accepted by Bendix inspection under Manufacturing Process 64 (M.P.-64); however actual values were not recorded as required (0.3 - 0.9 mils for primer and 9-12 mils for S-13G).

The only item of any possible significance revealed by the records investigation was the length of time between application and thermal-vacuum testing of the various units. The Flight 1 Central Station experienced the least time between paint application and test of all units as shown in the following summary.

Unit	Surface	Time Before Test
Proto A	All	5 mo.
Qual SA	T/P	10 mo.
	P/S	3 mo.
	S/S	3-1/2 mo.
Flight 1	T/P	4 mo.
	P/S	2 mo.
	S/S	2-1/2 mo.

Note: T/P Thermal Plate (Radiator Surface)
P/S Primary Structure
S/S Sunshield

Consultations with IITRI, supplier of S-13G coating materials to Bendix under Subcontract SC-0265, were held to determine the course of action necessary to establish the cause of adhesion failure and formulate meaningful corrective action. IITRI was given the responsibility of investigating the mechanical, chemical and application aspects of S-13G adhesion characteristics concurrent with the Bendix investigation of the thermal-vacuum adhesion characteristics. Some degree of overlap existed in the investigation program due to interrelationships which could not be conveniently separated because of availability of test equipment, instrumentation and facilities.

The following sections of this report present the important aspects of the joint investigation by Bendix Aerospace Systems Division and IITRI in considerable detail. Not all of the aspects of the S-13G thermal coating adhesion problem have been investigated as thoroughly as desired because of time, program schedule, and cost limitations. However, the findings as presented in this report demonstrate a high degree of confidence that the problem has been properly analyzed and the corrective action implemented will provide adequate S-13G thermal coating adhesion integrity to permit the ALSEP thermal control design to function properly under the extremes of actual lunar environment.

2.0 BENDIX TEST FACILITIES

The series of S-13G thermal coating thermal-vacuum adhesion tests were performed in the BxA 4' x 8' Thermal-Vacuum Test Chamber at Bendix Aerospace Systems Division during the period of 13 July 1968 through 6 September 1968. All adhesion tests were conducted using the same basic equipment and instrumentation as listed in Table 2. The 4' x 8' chamber, (Fig. 1) with an integral cold wall for the circulation of liquid nitrogen (LN_2) was used to provide a gross environment of $-300^oF \pm 20^oF$ at a pressure of 1×10^{-5} torr or less throughout the tests. In addition, a copper thermal plate having integral LN_2 coils and electric heaters was used to provide the required thermal environment to all test samples. Thermocouples were attached to the chamber cold wall, thermal plate, and some test samples. All temperature data were monitored and recorded with a Leeds and Northrup potentionmeter and Daystrom multipoint recorder. The tests were also visually monitored by the test engineer and photographically with a Polaroid camera.

3.0 BENDIX TEST PROCEDURE

The S-13G thermal coating thermal-vacuum adhesion tests were performed using a common test procedure and subjecting the test samples to cold and hot temperature cycles. Tests 1, 3, and 4 subjected the test samples to an alternating cold-hot environment (-300^oF to $+200^oF$). Test 2 used a hot-cold cycle ($+200^oF$ to -300^oF) to determine if the equivalent of a vacuum bake-out of the test samples was of any significance to the S-13G thermal coating adhesion. All tests were conducted with two or three complete thermal cycles.

In the cold-hot cycle tests the procedure steps used were as follows:

1. Closed chamber door and initiated evacuation pumps.
2. When chamber pressure reached a value of 1×10^{-5} torr, cold wall and thermal plate LN_2 flow initiated and maintained until thermal plate and test samples reached a temperature of $-300^oF \pm 20^oF$.
3. Cold soaked test samples for 30 minutes (15 minutes for tests, 2, 3, & 4).

TABLE 2 THERMAL-VACUUM TEST EQUIPMENT AND INSTRUMENTATION

ITEM	MANUFACTURER	FUNCTION
1. Vacuum Chamber (4' x 8')	NRC	Provide Vacuum Environment (1 x 10^{-5} Torr or Less)
2. Chamber (4' x 8') Control Console	NRC	Control of Vacuum Environment
3. Thermal Plate	Bendix	Thermal Control of Test Samples
4. Labac Power Controller	R. I.	Thermal Plate Heater Power Control
5. Multipoint Recorder	Daystrom	Chamber, Thermal Plate, & Test Sample Temperature Recording
6. Potentiometer	Leeds & Northrop	Thermal Plate and Test Sample Temperature Monitor
7. Camera	Polaroid	Test Photographic Coverage

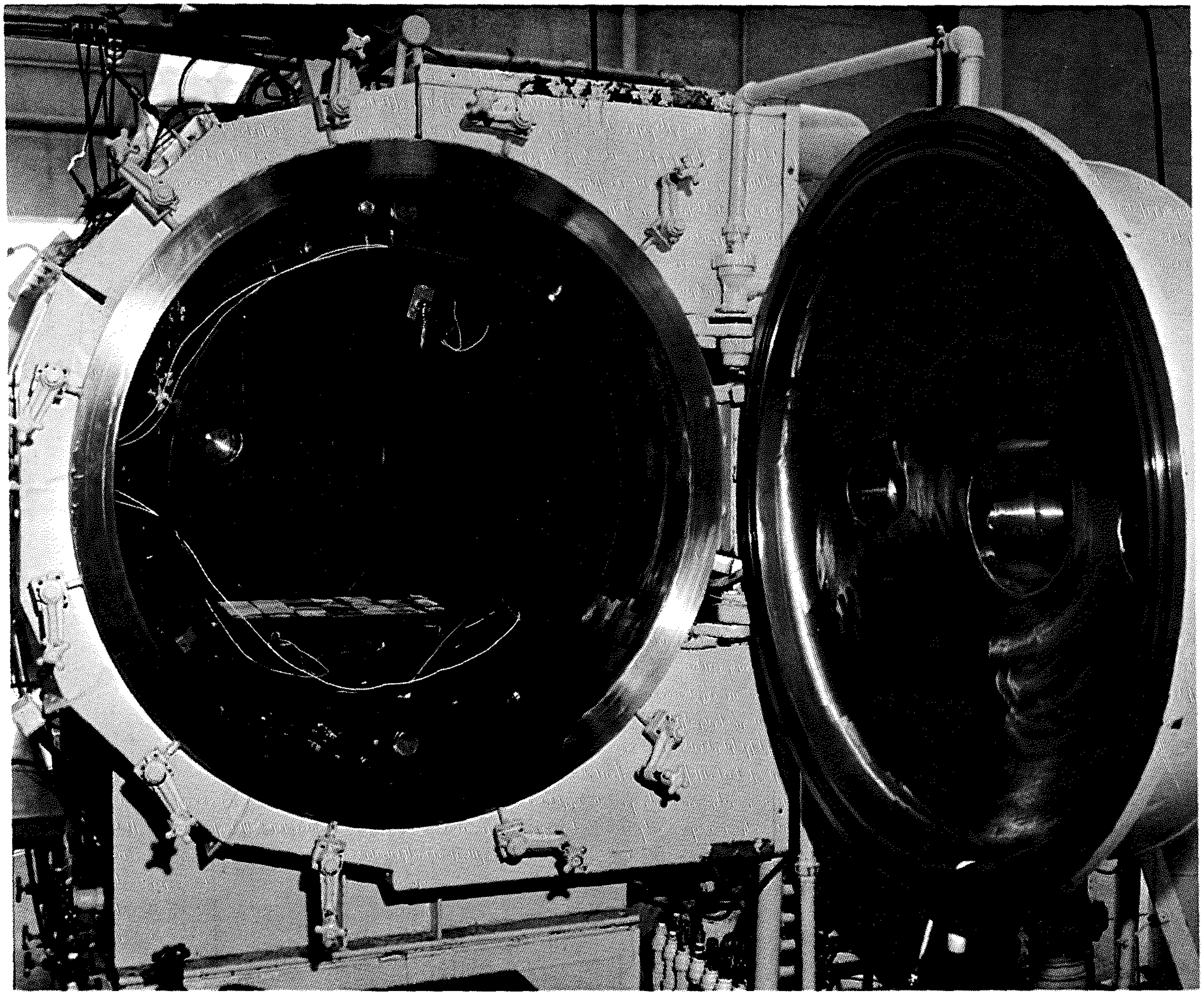

Figure 1 Photograph of BxA 4' x 8" Vacuum Chamber With Test Specimens on Thermal Plate.

4. Turned off LN_2 flow to thermal plate upon completion of cold soaking and initiated thermal plate heater power.
5. Adjusted thermal plate heater power to raise thermal plate and test sample temperatures to +200°F ± 20°F (chamber cold wall LN_2 flow continued).
6. Hot soaked test samples for 30 minutes (15 minutes for tests 2, 3, &4) at +200°F ± 20°F.
7. Turned off thermal plate heater power upon completion of hot soaking and turned on LN_2 flow to thermal plate.
8. Repeated cold-hot cycle one or two more times and then terminated test.

A typical cold-hot cycle obtained from the above listed steps is presented in Figure 2. As previously stated, Test 2 was conducted with a hot-cold cycling of the samples.

After removal of test samples from the 4' x 8' chamber upon completion of Tests 3 and 4, those samples which did not exhibit adhesion failure of the S-13G thermal coating were subjected to a thirty (30) second dip in LN_2 as an additional thermal shock test.

Temperature data were recorded continuously throughout the thermal-vacuum tests to verify chamber conditions under which S-13G adhesion failure occurred. Upon removal from the test chamber, all test samples which had failed were examined visually to verify the type failure (primer substrate interface, primer/S-13G interface) which had occurred.

Figure 3 shows typical test sample results and characteristic adhesion failure patterns during thermal-vacuum tests. This photo was taken at the time of removal of samples of Test 4D from the 4' x 8' vacuum chamber. Note the fragments of S-13G coating on the chamber floor and across samples on the thermal plate. The elastic characteristics of the S-13G coating are such as to cause catastrophic adhesion failure on many samples as evidenced in the photograph.

4.0 BENDIX TEST NO. 1

The initial S-13G thermal-vacuum adhesion tests performed by Bendix were conducted on 13 and 14 July 1968. A photograph of Test No. 1 samples S-1 through S-13 is shown in Figure 4. This test utilized the

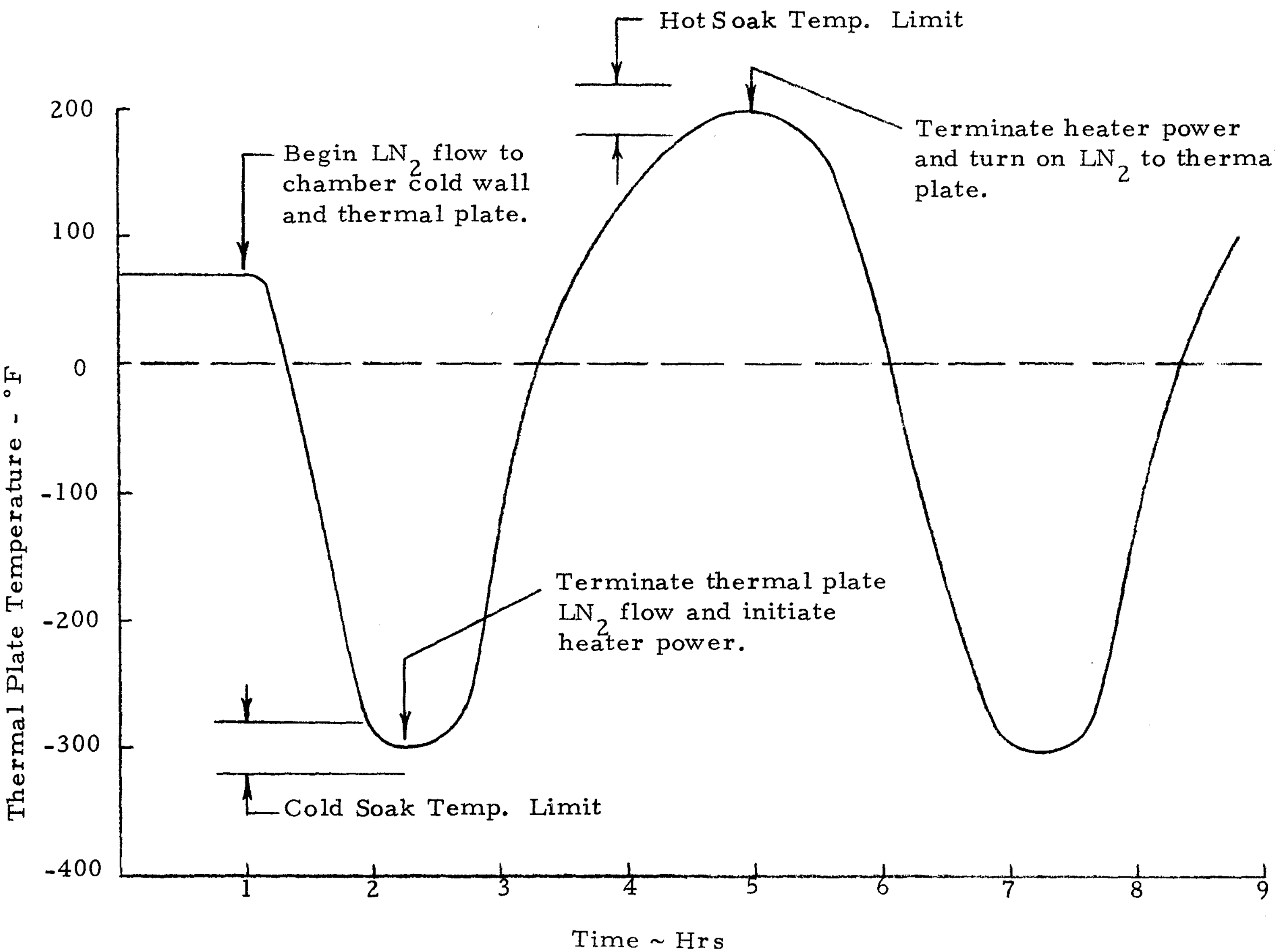

Figure 2. Typical Cold-Hot Temperature Cycle of Thermal Plate

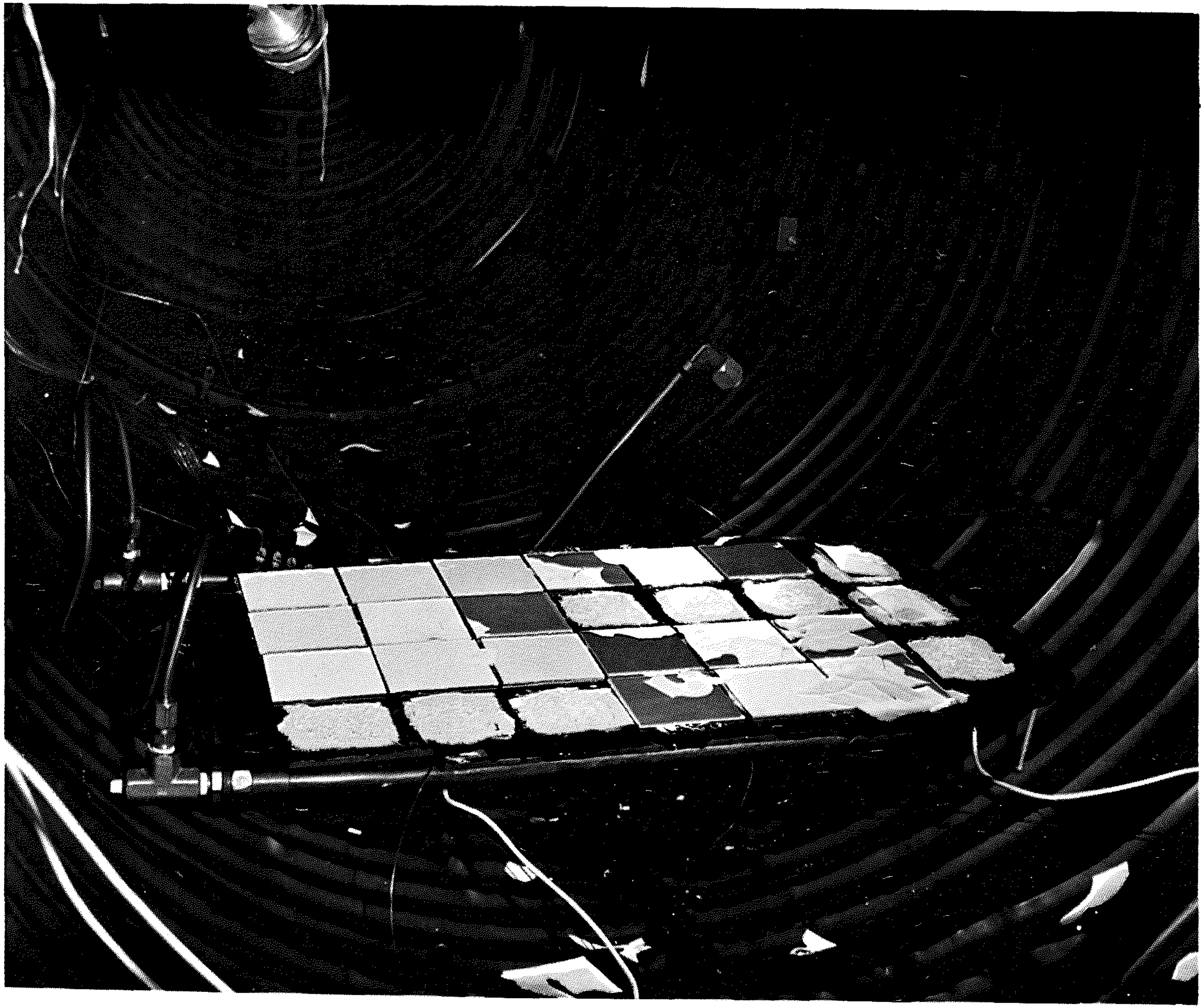

Figure 3 Photograph of Test Specimens Upon Completion of Test 4D.

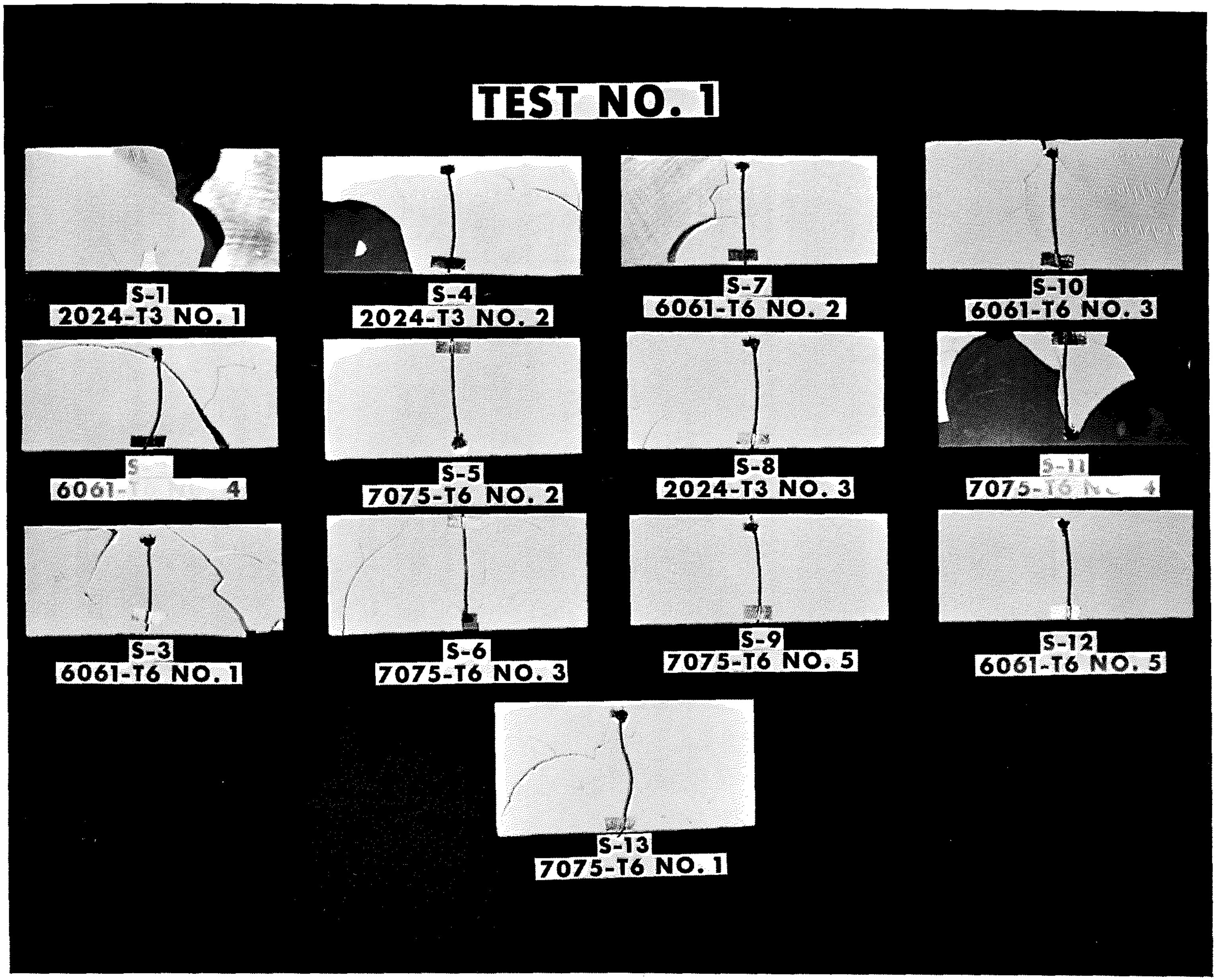

Figure 4 Photograph of Test No. 1 Test Specimens.

three basic substrate materials employed in the ALSEP Central Station sunshield, primary structure, and thermal plate. Each of the substrate materials received specific surface treatments and primer and S-13G thermal coating applications in order to introduce as many major variables as possible which could be related to the Flight 1 adhesion failure of the S-13G thermal coating. The basic test objectives in Test No. 1 are summarized as follows:

1. Determination of substrate material effects upon adhesion.
2. Investigation of the effects of substrate surface preparation.
3. Evaluation of GE SS-4044 primer batch quality.
4. Evaluation of S-13G thermal coating batch quality.
5. Possible degradation of S-13G thermal coating adhesion due to cleaning procedure.
6. Determination of the temperature range in which the adhesion failure occurs.

4.1 Sample Preparation

Test coupons were prepared in a nominal 6" x 6" size of 2024-T3 Alclad (sunshield), 7075-T6 aluminum (primary structure) and 6061-T6 aluminum (thermal plate). The range of surface treatments investigated are presented in Table 3. A total of thirteen coupons were prepared from the basic substrate materials, three of which were 2024-T3 Alclad, five were 7075-T6 Anodized aluminum and five were 6061-T6 aluminum with Chem Film treatment. The specific surface treatment of each coupon prior to primer application is presented in Table 4. Upon completion of coupon surface treatment all were cleaned and dried before the primer was applied as indicated in Table 3.

The thirteen coupons were coated with GE SS-4044 primer at a thickness of approximately 0.5 mils. Both the original Flight 1 primer batch (316) and a new primer batch (349) were used as indicated in Table 4. After a one hour air dry at room temperature (70 to 80^{o}F), S-13G thermal coating was applied at a thickness between 9 and 12 mils. Both the original Flight 1 and new S-13G batches were used as indicated in Table 4. Upon completion of the S-13G application, the coupons were cured by air drying at room temperature for 48 hrs.

To test for possible cleaning procedure effects, two S-13G coated coupons (S-9 and S-12) were given a simulated cleaning by scrubbing with a 5% Alconox solution follows by a distilled water rinse (procedure used on Flight 1 sunshield prior to submitting for T/V

TABLE 3. BENDIX TEST NO. 1 TEST COUPON SUBSTRATE PREPARATION

Aluminum Material	2024-T3	7075-T6	6061-T6
Normal Surface Treatment	Alclad	Anodized	Chem Film
Central Station Surface	Sunshield	Thermal Plate	Primary Structure
Thermal-Vacuum Test Surface Treatment	1. Normal Surface Treatment 2. Alclad Removed by Abrasion	1. Normal Surface Treatment 2. Anodizing Removed by Abrasion	1. Normal Surface Treatment 2. Chem Film Removed by Abrasion
Cleaning Treatment	Acetone Followed by Distilled Water Rinse Until Break-Free Condition Obtained	Acetone Followed by Distilled Water Rinse Until Break-Free Condition Obtained	Acetone Followed by Distilled Water Rinse Until Break-Free Condition Obtained
Drying Treatment	Air Dry at Room Temperature	Air Dry at Room Temperature	Air Dry at Room Temperature
Test Sample Number	S-1, S-4, & S-8	S-5, S-6, S-9, S-11 & S-13	S-2, S-3, S-7, S-10, & S-12

TABLE 4. BENDIX TEST NO. 1 TEST COUPON COATING APPLICATION

Test Sample Number	Material	Surface Treatment	Primer (GE SS 4044)	S-13G Thermal Coating	Remarks
S-1	2024-T3 No. 1	Abraded	Flt. 1	Flt. 1	
S-2	6061-T6 No. 4	Normal	New	New	
S-3	6061-T6 No. 1	Abraded	Flt. 1	Flt. 1	
S-4	2024-T3 No. 2	Normal	New	New	
S-5	7075-T6 No. 2	Abraded	New	New	
S-6	7075-T6 No. 3	Abraded	Flt. 1	Flt. 1	
S-7	6061-T6 No. 2	Abraded	New	New	
S-8	2024-T3 No. 3	Normal	New	New	
S-9	7075-T6 No. 5	Abraded	New	New	Cleaned with Alconox + distilled water rinse
S-10	6061-T6 No. 3	Normal	Flt. 1	Flt. 1	
S-11	7075-T6 No. 4	Normal	New	New	
S-12	6061-T6 No. 5	Abraded	New	New	Cleaned with Alconox + distilled water rinse
S-13	7075-T6 No. 1	Abraded	Flt. 1	Flt. 1	

NOTE:

1. All test samples had S-13G coating thickness of 9-12 Mils over a primer thickness of 0.6-0.9 Mils.
2. Primer Cure: 1 hr air dry at room temperature (70-80°F)
3. S-13G Cure: 48 hr air dry at room temperature (70-80°F)

acceptance tests). The coupons were then cut in half to provide two test samples approximately 3" x 6" in size. One of the samples from each coupon was retained for future tests and the remaining sample was used for Test No. 1.

4.2 Test Results

Of the thirteen samples, nine exhibited adhesion failure during the first cold cycle of the test. The first adhesion failure (sample S-11) occurred at a nominal temperature of $-142^{\circ}F \pm 20^{\circ}F$ as noted in Table 5. The temperatures at which the remaining failures occurred during the first cold cycle are also noted in Table 5.

All samples which failed during the test did so during the first cold cycle. One sample (S-7) successfully passed two cold soak periods but adhesion failure was observed during the second hot soak period after the thermal plate temperature had inadvertently been allowed to rise to at least $+392^{\circ}F$. This excessively high temperature was reached as a result of improper setting of thermal plate heater voltage. Failure of S-7 was noted after the high temperature was corrected and the exact time of adhesion failure is unknown.

Two distinct types of adhesion failure were observed during Test No. 1. One failure type was the formation of a bubble with cracks developing at the bubble edge. The second failure type was the formation of cracks across the sample surface beginning at the sample edge. The type of adhesion failure was observed to be generally related to the substrate surface treatment. Bubble formation was associated with those samples which had normal surface treatment (Alclad, Anodize, & Chem Film). Initial cracking of the coating was generally associated with those samples which had normal surface treatment films removed by abrasion.

Those test samples (S-5, S-9, S-12) which successfully passed the thermal-vacuum adhesion test were samples which had normal substrate surface treatment films removed by abrasion. Samples S-9 and S-12 were also the two which were scrubbed with a 5% Alconox solution to simulate the cleaning procedure used on the Flight 1 sunshield. From these results it was concluded that the cleaning procedure was not a significant factor in the Flight 1 S-13G adhesion failure.

TABLE 5. BENDIX TEST NO. 1 TEST RESULTS

Test Sample Number	Date	Cycle	Sample Temp. at Failure ~ °F(± 20)	Initial Failure Type
S-11	7-13-68	1st Cold	-142(-122, -162)	Cracks
S-2			-184(-164, -204)	Bubble
S-10			-183(-163, -203)	Bubble
S-6			-181(-161, -201)	Cracks
S-1			-191(-171, -211)	Cracks
S-3			-191(-171, -211)	Cracks
S-4			-254(-234, -274)	Cracks
S-8			-300(-280, -320)	Bubble
S-13			-300(-280, -320)	Cracks
S-7	7-14-68	Unknown	+392°F Min.	Unknown
S-5				
S-9		N. A.	N. A.	Successfully Completed Two (2) Cold-Hot Cycles
S-12				

Generally Test No. 1 provided the following information relative to S-13G adhesion failure under thermal-vacuum conditions:

1. Substrate material was not a significant factor.
2. Substrate surface preparation was a related factor.
3. Primer and S-13G batch quality could not be evaluated properly since samples having the original and new Flight 1 primers and S-13G coatings did not pass the test.
4. Adhesion failure on all samples occurred at the primer/substrate interface.
5. The adhesion failures occurred in a temperature range of approximately -150°F to -300°F during the first cold cycle. The only exception to this was sample S-7.

5.0 BENDIX TEST NO. 2

The second thermal-vacuum adhesion test was performed on 14 and 15 July 1968 using a total of six test samples, four of which were from retained portions which were available from Test No. 1 coupons. Test specimens of Test No. 2 are shown in Figure 5. Two new samples were included from a specially prepared coupon. In Test No. 2, three hot-cold cycles were completed before the test was terminated. A hot soak (+200°F ±20°F) period for 15 minutes was performed first to provide the test samples the equivalent of a vacuum-bake prior to subjecting them to the cold temperature environment.

The basic test objectives in Test No. 2 are summarized as follows:

1. Effect of elevated temperature cure of S-13G thermal coating upon adhesion.
2. Effect of abraded versus normal substrate surface preparation.

5.1 Sample Preparation

Test samples for Test No. 2 were prepared as indicated in Table 6. Four of the six samples were retained from Test No. 1 coupons and are so identified in the table. Two samples were taken from a specially prepared coupon (2024-T3 Alclad No. 5) which had the S-13G coating cured for 24 hrs. at 200°F. This coupon had been

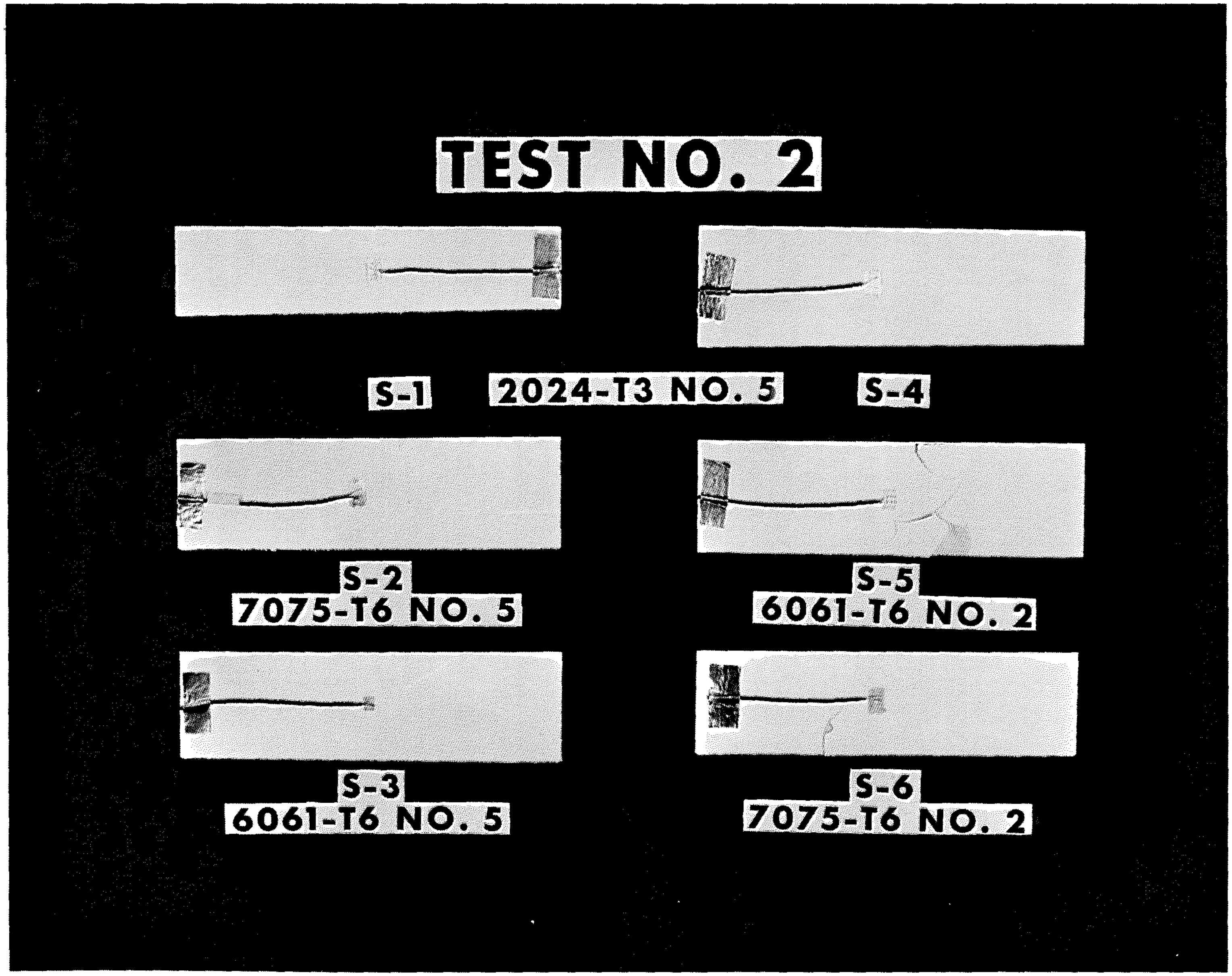

Figure 5. Photograph of Test No. 2 Test Specimens.

TABLE 6 BENDIX TEST NO. 2 TEST COUPON COATING APPLICATION

TEST SAMPLE NO.	MATERIAL	SURFACE TREATMENT	PRIMER (GE SS-4044)	S-13G THERMAL COATING	S-13G CURE	REMARKS
S1	2024-T3 No. 5	Abraded	New	New	24 Hrs. @ 200°F	
S2	7075-T6 No. 5	Abraded	New	New	48 Hr. Air Dry	Retain From Test No. 1
S3	6061-T6 No. 5	Abraded	New	New	48 Hr. Air Dry	Retain From Test No. 1
S4	2024-T3 No. 5	Abraded	New	New	24 Hrs. @ 200°F	
S5	6061-T6 No. 2	Abraded	New	New	48 Hr. Air Dry	Retain From Test No. 1
S6	7075-T6 No. 2	Abraded	New	New	48 Hr. Air Dry	Retain From Test No. 1

NOTE:

1. Primer Thickness: 15 mils
2. Primer Cure: 1 hr. air dry at room temp. (70-80°F)
3. 5-13G coating thickness 9-12 mils

abraded to remove the Alclad surface treatment film and primer was applied at a 0.5 mil thickness. The primer was cured for 1 hr in air at room temperature (70 to 80°F). New primer and new S-13G batches were used on this coupon.

5.2 Test Results

Two of the six samples in Test No. 2 exhibited adhesion failure during the first cold cycle. Sample S-2 (a duplicate of sample S-9 in Test No. 1) failed as did sample S-5 (a duplicate of sample S-7 in Test No. 1).

Of the four samples which successfully passed Test No. 2, two (S-1 and S-4) were the special elevated temperature cured samples. The other two successful samples (S-3 and S-6) had also successfully passed Test No. 1 (as samples S-5 and S-12).

Visual examination of the samples after removal from the 4' x 8' test chamber showed the same primer/substrate interface failure as observed in Test No. 1. The 15 minute vacuum bake simulated at the beginning of the Test No. 2 by performing the hot soak first was considered to be of little if any benefit due to the failure of sample S-2.

From the samples which passed Test No. 2 (and Test No. 1 also), the following basic conclusions were drawn:

1. Elevated temperature cure of the S-13G thermal coating is beneficial in obtaining adhesion.
2. Abrasion of substrate surface to remove all normal surface treatment improves adhesion characteristics of S-13G thermal coating.
3. Adhesion failure is primer related.
4. Vacuum bake is not significant in promoting adhesion.

6.0 BENDIX TEST NO. 3

Test No. 3 was conducted on July 24-25, 1968 and is essentially a repetition of Tests 1 and 2 with one basic difference. The remaining portions of coupons fabricated for Tests 1 and 2 were the source of samples for Test No. 3. Test specimens of Test No. 3 are shown in Figure 6. A test sample was cut from each of the coupon portions

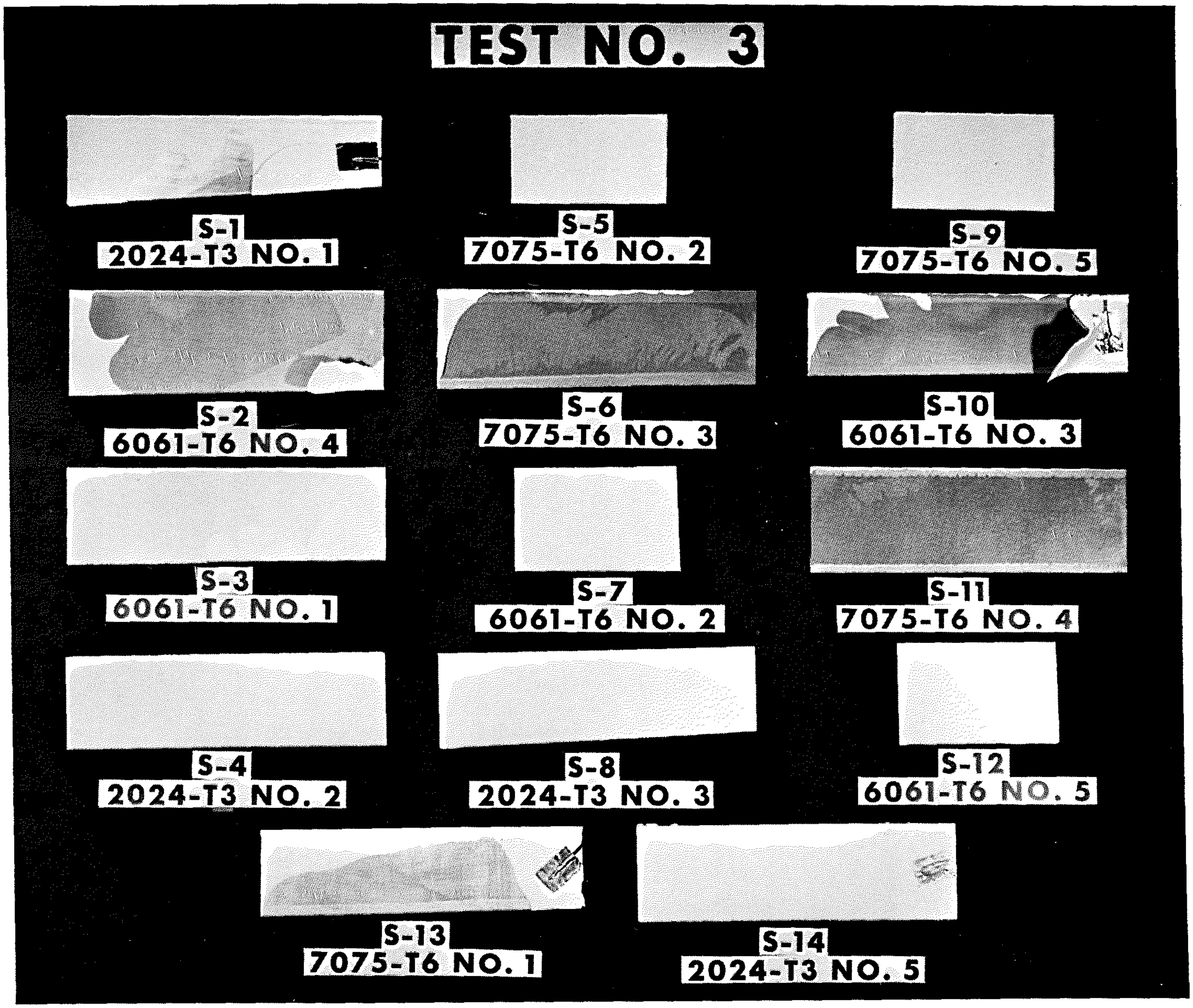

Figure 6. Photograph of Test No. 3 Test Specimens.

available to produce a total of fourteen test samples. Thirteen of the samples were from Test No. 1 coupons and one sample was taken from the special coupon from Test No. 2. All test samples then were baked for 24 hours at a temperature of 150°F.

The basic objectives of Test No. 3 were to determine the following:

1. Beneficial effect of elevated temperature cure upon previous test samples.
2. Duplication of previous successful test results.
3. Formulate interim changes to M.P. 64 (S-13G Thermal Coating Application Procedure) to prevent adhesion failure on ALSEP Flight 2 hardware which had already been coated with S-13G.

6.1 Sample Preparation

As previously stated, Test No. 3 was conducted with samples from coupons fabricated for Tests No. 1 and No. 2. A total of 14 test samples were prepared from the retained coupon portions; each sample being approximately 1" x 6" or 1-1/2" x 3" in size. None of the samples were instrumented with thermocouples as in Tests No. 1 and No. 2 but were attached to the test chamber thermal plate with Dow Corning 340 Heat Sink Compound to insure adequate contact between each sample and the thermal plate. Thermocouples installed on the thermal plate were monitored to determine establishment of thermal soak conditions. Test sample identification for Test No. 3 is presented in Table 7.

6.2 Test Results

Of the fourteen test samples, seven successfully passed three complete cold-hot cycles in the 4' x 8' test chamber. Upon removal from the chamber the seven successful samples were subjected to an ambient pressure LN_2 dip test without exhibiting adhesion failure. The seven test samples which successfully completed Test No. 3 are noted in Table 7 which summarizes the overall results of the test and correlates them to Test No. 1 and No. 2.

Of the seven successful samples, three duplicated Test No. 1 successes and four duplicated Test No. 2 successes. One sample which had previously failed during Tests No. 1 and No. 2 successfully passed

TABLE 7 BENDIX TEST NO. 3 TEST SAMPLE IDENTIFICATION

Test Sample Number	Mat'l	Surface Treatment	Test No. 1 I. D.	Test No. 1 Result	Test No. 2 I. D.	Test No. 2 Result	Test No. 3 Result
S-1	2024-T3 No. 1	Abraded	S-1	Failed	-	-	Failed
S-2	6061-T6 No. 4	Normal	S-2	Failed	-	-	Failed
S-3	6061-T6 No. 1	Abraded	S-3	Failed	-	-	Failed
S-4	2024-T3 No. 2	Normal	S-4	Failed	-	-	Passed
S-5	7075-T6 No. 2	Abraded	S-5	Passed	S-6	Passed	Passed
S-6	7075-T6 No. 3	Abraded	S-6	Failed	-	-	Failed
S-7	6061-T6 No. 2	Abraded	S-7	Failed	S-5	Failed	Passed
S-8	2024-T3 No. 3	Normal	S-8	Failed	-	-	Passed
S-9	7075-T6 No. 5	Abraded	S-9	Passed	S-2	Failed	Passed
S-10	6061-T6 No. 3	Normal	S-10	Failed	-	-	Failed
S-11	7075-T6 No. 4	Normal	S-11	Failed	-	-	Failed
S-12	6061-T6 No. 5	Abraded	S-12	Passed	S-3	Passed	Passed
S-13	7075-T6 No. 1	Abraded	S-13	Failed	-	-	Failed
S-14	2024-T3	Abraded	-	-	S-1 & S-4	Passed	Passed

FOR COMPLETE SAMPLE PREPARATION SEE TABLES 4 AND 6

Test No. 3. Two other samples having failed Test No. 1 successfully passed Test No. 3. Sample (S-4) which passed Test No. 1 only to fail in Test No. 2 successfully passed Test No. 3.

The results of Test No. 3 led to the following observations relative to the S-13G adhesion failure:

1. Elevated temperature cure is beneficial to adhesion characteristics.
2. Randomness of previous test successes had been considerably narrowed.
3. All adhesion failures consistently occurred at the primer/substrate interface during the first cold cycle at cryogenic temperatures.
4. Basic changes to M.P. 64 in area of substrate surface treatment, primer and S-13G cure are indicated by test results.

7.0 BENDIX TEST NO. 4

Upon completion of the first three Bendix thermal-vacuum adhesion tests of S-13G thermal coating, a fourth test was formulated to investigate various combinations of primer thickness, cure, S-13G coating thickness and cure. The results of Tests No. 1, No. 2 and No. 3 produced general adhesion failures but did not cover enough primer and S-13G coating parameters to determine the exact cause. Consultation with IITRI on the results of their tests on the failure problem isolated the following basic parameters to be investigated in Test No. 4.

1. Effect of primer thickness upon adhesion.
2. Variation of primer cure process.
3. Effect of S-13G coating thickness upon adhesion.
4. Evaluation of S-13G coating cure process.
5. Single vs multilayer build up of both primer and S-13G coating.
6. S-13G application schedule effects.

Test No. 4 was conducted in five phases during the period 3 September through 11 September 1968 and used a basic set of 54 test coupons (approx. 6" x 6" in size). Test specimens of Tests 4A through 4E are shown in Figures 7-11. Test samples from each of the coupons were

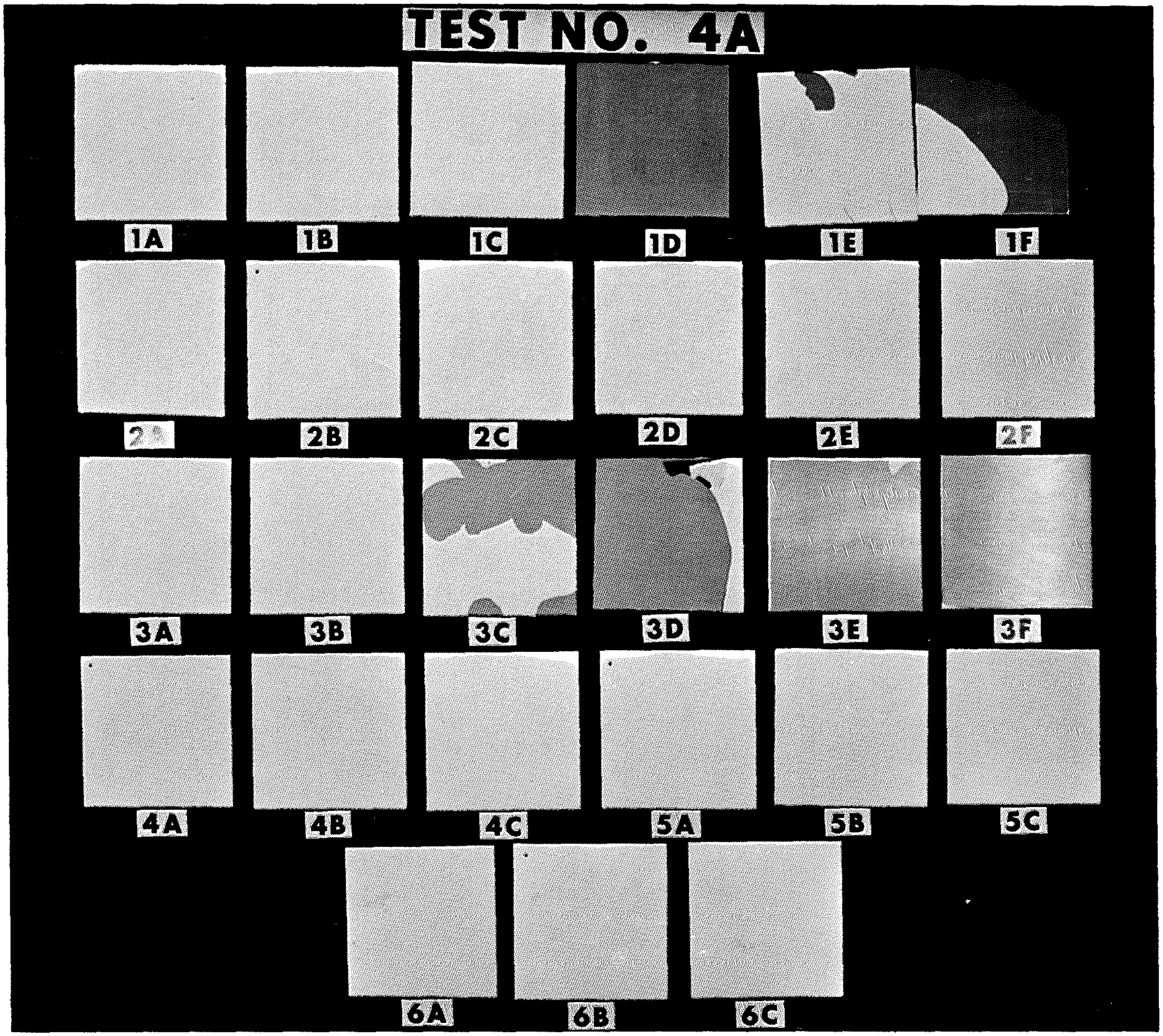

Figure 7. Photograph of Test No. 4A Test Specimens

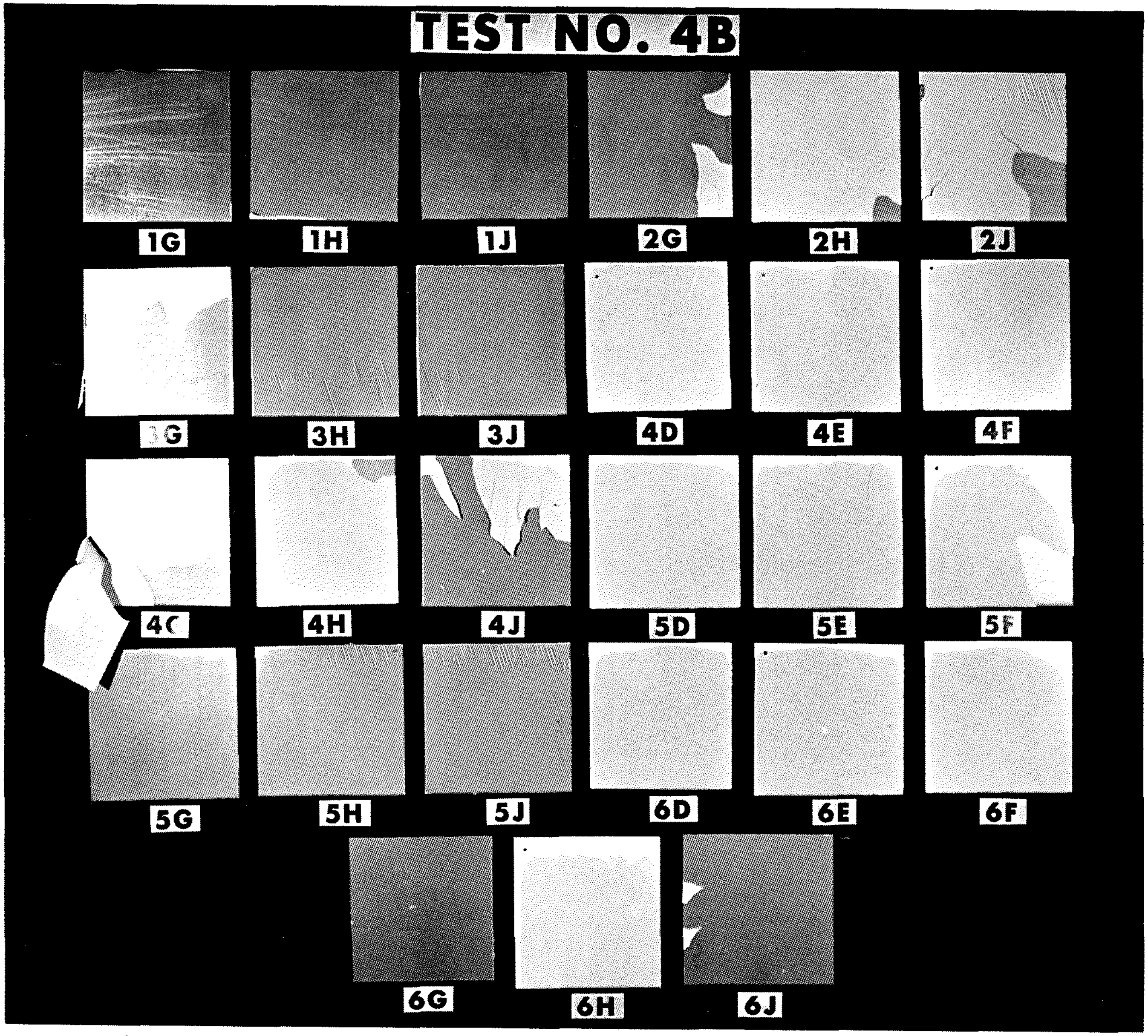

Figure 8. Photograph of Test No. 4B Test Specimens

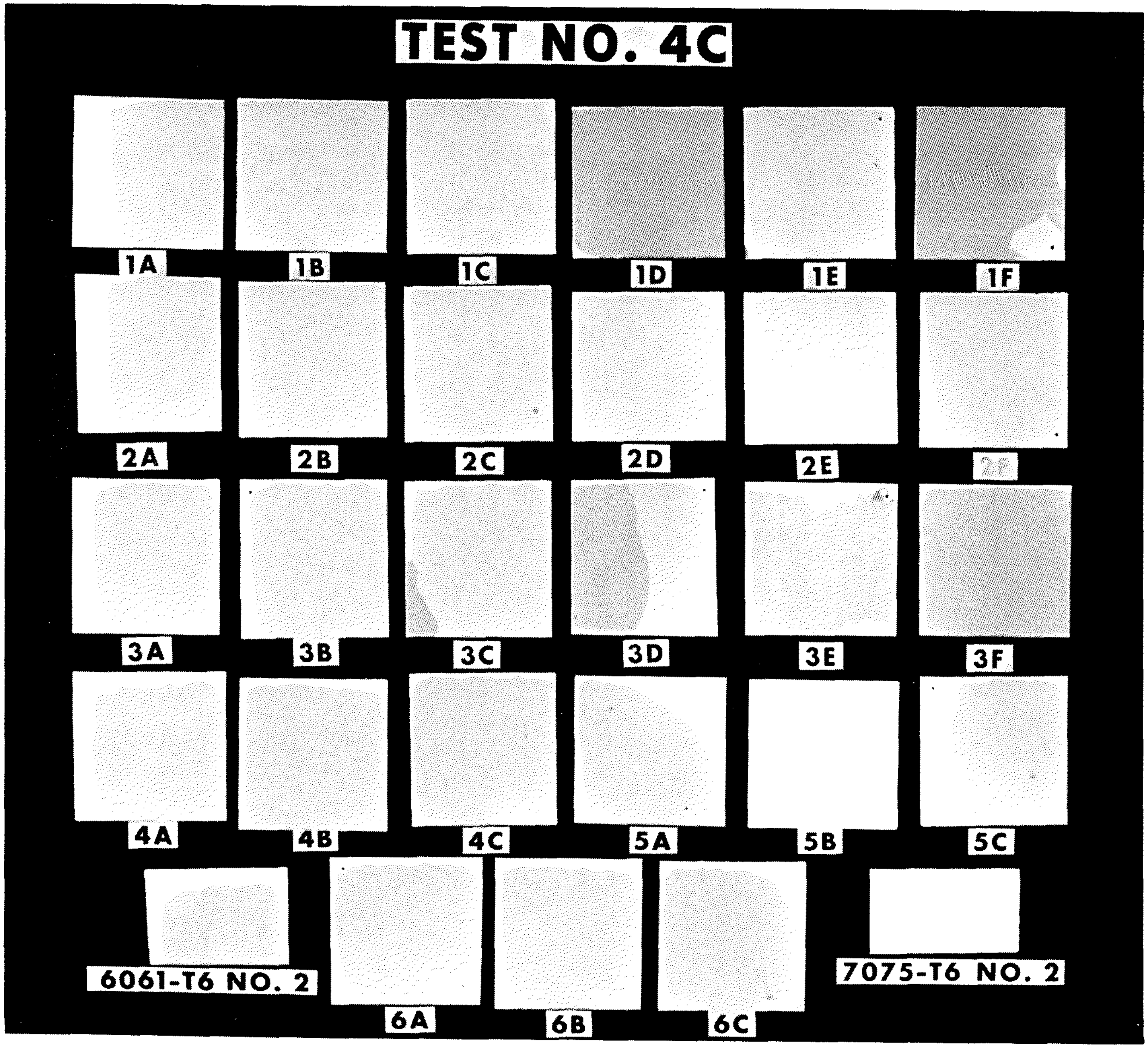

Figure 9. Photograph of Test No. 4C Test Specimens

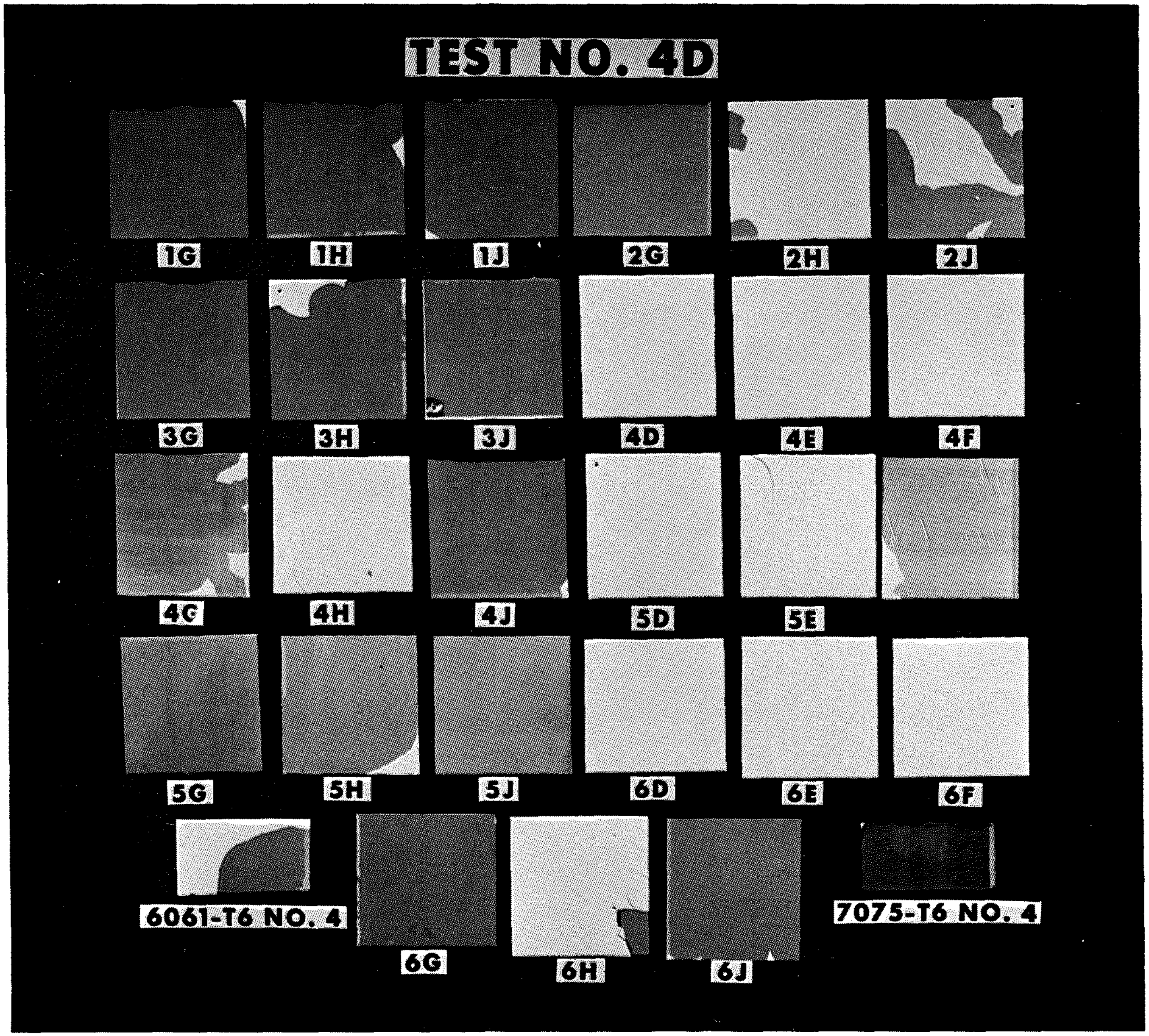

Figure 10. Photograph of Test No. 4D Test Specimens

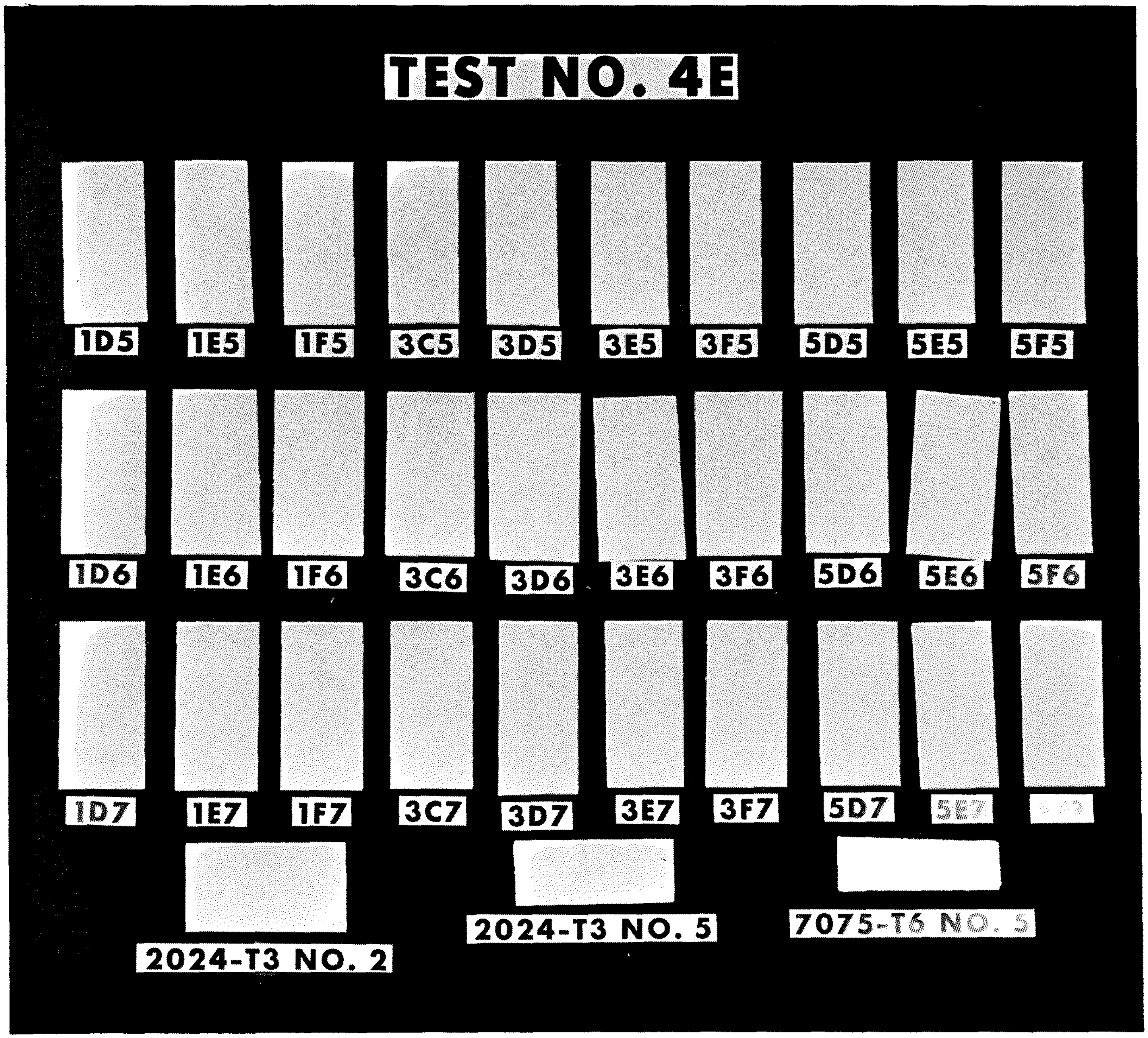

Figure 11. Photograph of Test No. 4E Test Specimens

subjected to thermal-vacuum environment in the same manner as in Tests No. 1, No. 2 and No. 3 in the 4' x 8' test chamber and also included LN_2 dip tests. Each phase of Test No. 4 subjected a group of samples attached to the thermal plate with Dow Corning 340 Heat Sink compound to two complete cold-hot cycles.

7.1 Sample Preparation

The 54 basic test coupons were prepared to provide test samples having specific values of primer and S-13G thickness, controlled primer and S-13G cure process, and variations in primer and S-13G application schedule. Table 8 presents the test coupon description in terms of these variables. All coupons were prepared using a common substrate material (7075-T6 Anodized Aluminum) with the normal surface treatment film removed by abrasion on all coupons except the No. 6 series coupons. On the No. 6 series coupons the normal substrate surface treatment film was retained as indicated in Table 8.

The coupon preparation was formulated to provide three basic primer thickness groups of 0.25 $\pm$0.1 mil, 0.5 $\pm$0.1 mil and 0.9 $\pm$0.1 mil. Permascope measurements were performed to determine actual primer thickness of each coupon. Upon completion of the primer cure process the S-13G was applied and measured with the permascope to determine its thickness relative to the desired value by which coupons were categorized. As indicated in Table 8 not all coupons were within the intended primer and S-13G thickness limits specified but they did exhibit values over the desired ranges of interest. Test results did not reflect any trends in adhesion failure which could be attributed to these primer and S-13G thickness variations within each group.

Actual primer and S-13G coating application was performed on a schedule similar to that used by Bendix shop personnel in coating actual hardware, i.e., both primer and S-13G thicknesses were obtained in one or more spray operations. Thus, the thin primer thickness (0.25 mil range) was achieved in one spray period, the medium thickness (0.5 mil) in two spray periods, and the heavy thickness (0.9 mil) was achieved in three spray periods. The No. 5 series coupons were given two applications of S-13G to achieve the desired thickness, with time between the first and second coat being controlled at 2, 6 and 16 hours. All coupons were coated with fresh GE SS-4044 primer and S-13G thermal coating supplied by IITRI specifically for Bendix Test No. 4 in an effort to eliminate any age variations between primer and S-13G batches.

TABLE 8. BENDIX TEST NO. 4 TEST COUPON PREPARATION

S-13G Thermal Coating Application Parameters		Primer Application Parameters								
		Thickness; 0.25 ± 0.1 Mil			Thickness; 0.50 ± 0.1 Mil			Thickness; 0.90 ± 0.1 Mil		
		1 Hr Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F	1 H4 Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F	1 Hr Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F
Thickness; 6-8 Mils	48 Hr Air Dry	0.10(1) 1A 5.0(1)	0.10(1) 1B 5.0(1)	0.10(1) 1C 6.0(1)	0.35(2) 1D 6.5(1)	0.35(2) 1E 5.5(1)	0.30(2) 1F 6.0(1)	0.80(3) 1G 6.0(1)	0.90(3) 1H 6.0(1)	0.90(3) 1J 6.0(1)
Thickness; 6-8 Mils	48 Hr Air Dry + 24 Hr @ 150°F	0.15(1) 2A 6.0(1)	0.10(1) 2B 6.0(1)	0.15(1) 2C 6.0(1)	0.40(2) 2D 5.5(1)	0.35(2) 2E 6.0(1)	0.35(2) 2F 6.0(1)	0.80(3) 2G 6.5(1)	0.95(3) 2H 6.5(1)	0.90(3) 2J 6.5(1)
Thickness; 9-12 Mils	48 Hr Air Dry	0. 0.15(1) 3A 6.0(1)	0.10(1) 3B 6.5(1)	0.20(1) 3C 5.5(1)	0.35(2) 3D 6.5(1)	0.45(2) 3E 6.5(1)	0.30(2) 3F 5.5(1)	0.95(3) 3G 6.5(1)	0.90(3) 3H 6.5(1)	0.85(3) 3J 6.0(1)
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	0.15(1) 4A 6.0(1)	0.15(1) 4B 6.0(1)	0.10(1) 4C 5.5(1)	0.30(2) 4D 6.0(1)	0.30(2) 4E 6.5(1)	0.25(2) 4F 6.0(1)	0.90(3) 4G 6.5(1)	9.90(3) 4H 7.0(1)	0.90(3) 4J 5.5(1)
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	0.15(1) 5A 10.0(2)	0.15(1) 5B 11.0(2)	0.15(1) 5C 11.0(2)	0.25(2) 5D 10.5(2)	0.45(2) 5E 10.0(2)	0.35(2) 5F 11.0(2)	0.95(3) 5G 10.5(2)	1.0(3) 5H 12.0(2)	0.85(1) 5J 10.5(2)
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	0.10(1) 6A 6.0(1)	0.10(1) 6B 6.5(1)	0.10(1) 6C 5.5(1)	0.35(2) 6D 6.5(1)	0.35(2) 6E 6.5(1)	0.25(2) 6F 6.0(1)	0.95(3) 6G 6.5(1)	0.90(3) 6H 6.5(1)	0.85(3) 6J 6.0(1)

NOTES:
1. All coupon substrate surface teatment removed by abrasion except No. 6 series coupons (7075-T6 anodized)
2. Coupons having 2 primer coats were coated within 40 min.
3. Coupons having 3 primer coats were coated within 1 hr 5 min.
4. Coupons 5C, 5F, & 5J were coated with S-13G 2 hrs between coats
5. Coupons 5B, 5E, & 5H were coated with S-13G 6 hrs between coats
6. Coupons 5A, 5D, & 5G were coated with S-13G 16 hrs between coats

Upon completion of coupon preparation, test samples (3" x 3" in size) were cut from the coupons and submitted for thermal-vacuum testing according to the schedule shown in Tables 9, 10, & 11. Test phases 4A and 4B used 27 test samples each and those samples which successfully completed two cold-hot thermal-vacuum cycles were given an LN_2 dip dip test upon removal from the test chamber. In test phases 4C and 4D, the LN_2 dip test was performed prior to submitting the samples for the cold-hot thermal-vacuum cycles. Test 4E used only cold-hot thermal-vacuum cycling.

7.2 Test Results

The results of Test No. 4 provided confirmation of those parameters critical to the S-13G adhesion problem in terms of proper application technique of both primer and S-13G thermal coating. Specifically, the following general comments summarize the findings of the test:

1. Primer thickness is a critical parameter.
2. Primer application technique is important.
3. Primer cure is important but not critical depending upon the S-13G cure.
4. S-13G cure is of major importance.
5. S-13G thickness and/or application schedule were not significant parameters.

In test phases 4A and 4B, a total of 54 test samples (27 in each phase), were subjected to two complete thermal-vacuum cold-hot cycles and LN_2 dip. In the first cold cycle a total of 26 test samples experienced adhesion failure. All failures occurred in a temperature range between $-250^{\circ}F$ and $-300^{\circ}F$. Table 9 presents the failures according to the coupon preparation parameters for each sample. The adhesion failure pattern is well defined in terms of the preparation parameters as shown in Table 9. Only samples 3G and 6H do not conform well to the overall pattern.

Analysis of the test 4A and 4B results present a definite set of general conclusions in terms of preparation parameters for obtaining satisfactory S-13G thermal vacuum adhesion characteristics. These conclusions are:

1. A thin primer application (0.25 mils or less) produced good adhesion characteristics regardless of primer cure process, S-13G coating thickness, or S-13G cure process.

TABLE 9 TEST 4A AND 4B RESULTS

S-13G Application Parameters	Cure Process	Primer Application Parameters: Thickness; 0.25 ± 0.1 Mils — 1 Hr Air Dry	Thickness; 0.25 ± 0.1 Mils — 16 Hr Air Dry	Thickness; 0.25 ± 0.1 Mils — 16 Hr Air Dry + 16 Hrs @ 150°F	Thickness; 0.5 ± 0.1 Mils — 1 Hr Air Dry	Thickness; 0.5 ± 0.1 Mils — 16 Hr Air Dry	Thickness; 0.5 ± 0.1 Mils — 16 Hr Air Dry + 16 Hrs @ 150°F	Thickness; 0.9 ± 0.1 Mils — 1 Hr Air Dry	Thickness; 0.9 ± 0.1 Mils — 16 Hr Air Dry	Thickness; 0.9 ± 0.1 Mils — 16 Hr Air Dry + 16 Hrs @ 150°F
Thickness; 9-12 Mils	48 Hr Air Dry	1A	1B	1C	1D X	1E X	1F X	1G X	1H X	1J X
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hrs @ 150°F	2A	2B	2C	2D	2E	2F	2G X	2H X	2J X
Thickness; 6-8 Mils	48 Hr Air Dry	3A	3B	3C X	3D X	3E X	3F X	3G X	3H X	3J X
Thickness; 6-8 Mils	48 Hr Air Dry + 24 Hrs @ 150°F	4A	4B	4C	4D	4E	4F	4G X	4H X	4J X
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hrs @ 150°F	5A	5B	5C	5D	5E X	5F X	5G X	5H X	5J X
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hrs @ 150°F	6A	6B	6C	6D	6E	6F	6G X	6H	6J X

X Denotes thermal vacuum adhesion failure

TABLE 10. TEST 4C AND 4D RESULTS

S-13G Application Parameters		Primer Application Parameters								
		Thickness; 0.25± 0.1 Mils			Thickness; 0.5 ± 0.1 Mils			Thickness; 0.9 ± 0.1 Mils		
	Cure Process	1 Hr Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F	1 Hr Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F	1 Hr Air Dry	16 Hr Air Dry	1 Hr Air Dry + 16 Hr @ 150°F
Thickness; 9-12 Mils	48 Hr Air Dry	1A	1B	1C	1D X	1E X	1F X	1G O	1H O	1J O
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	2A	2B	2C	2D	2E	2F	2G X	2H X	2J X
Thickness; 6-8 Mils	48 Hr Air Dry	3A	3B	3C X	3D O	3E O	3F X	3G O	3H O	3J O
Thickness; 6-8 Mils	48 Hr Air Dry + 24 Hr @ 150°F	4A	4B	4C	4D	4E	4F	4G X	4H X	4J X
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	5A	5B	5C	5D	5E X	5F X	5G O	5H O	5J O
Thickness; 9-12 Mils	48 Hr Air Dry + 24 Hr @ 150°F	6A	6B	6C	6D	6E	6F	6G X	6H X	6J X

O Denotes LN_2 dip test adhesion failure
X Denotes thermal-vacuum adhesion failure

TABLE 11. TEST 4E TEST SAMPLES

Elevated Temperature Cure	Sample Number
4 HR @ 150°F	105, 1E5, 1F5 3C5, 3D5, 3E5, 3F5 5D5, 5E5, 5F5
8 HR @ 150°F	1D6, 1E6, 1F6 3C6, 3D6, 3E6, 3F6 5D6, 5E6, 5F6
16 HR @ 150°F	1D7, 1E7, 1F7 3C7, 3D7, 3E7, 3F7 5D7, 5E7, 5F7 2024-T3 No. 5 (S-1 & S-4 In Test No. 2)
24 HR @ 150°F	7075-T6 No. 5 (S-9 In Test No. 1) 2024-T3 No. 2 (S-4 In Test No. 1) These Samples Cured With Flight 1 Sunshield

2. A medium primer application (0.30 mils to 0.5 mils) was unsatisfactory regardless of primer cure process or S-13G coating thickness if an S-13G air dry room temperature cure process was used.
3. A medium primer application was satisfactory if the S-13G coating was cured at elevated temperatures.
4. A heavy primer application (0.6 mils to 0.9 mils or greater) was unsatisfactory regardless of primer cure, S-13G thickness or S-13G cure process.

Tests 4C and 4D were performed to minimize any possible randomness and substantiate results obtained in 4A and 4B. Since all adhesion failures occurred in thermal-vacuum conditions in tests 4A and 4B, the duplicate samples used for tests 4C and 4D were subjected to an LN_2 dip test prior to thermal-vacuum testing. In performing this test, 11 test samples failed in adhesion as noted in Table 10 which presents tests 4C and 4D results. These 11 failures duplicated 11 of the 26 failures experienced in tests 4A and 4B. The remaining 43 test samples were then subjected to thermal-vacuum conditions and 16 additional adhesion failures were experienced providing 100 percent duplication of results obtained in tests 4A and 4B as well as obtaining failure of sample 6H. The test procedure variation used in tests 4C and 4D of performing the LN_2 dip test first raises a question regarding the validity of the LN_2 dip test as a satisfactory adhesion test since it could only duplicate 11 of the 26 failures obtained under thermal-vacuum conditions. In any event, the results of tests 4C and 4D did verify the conclusions reached by the results of tests 4A and 4B. Test 4C included two samples (S-5 and S-7) available from Test No. 1, both of which successfully passed Test 4C. One (S-7) of these had experienced adhesion failure in Test No. 1.

Also included in Test 4D were two samples (S-11 and S-2) available from Test No. 1 which had been curing at room temperature for approximately 1 1/2 months. These two samples both failed again under thermal-vacuum conditions indicating that any benefit derived from a long term room temperature air dry cure would have to be realized over a greater time period than they experienced. It should be noted that these two samples were representative of the condition of the Flight 1 central station radiator surface at the time of Test 4.

Test 4E was performed in an attempt to determine the general relationship between cure time at elevated temperature and the establishment of acceptable thermal-vacuum adhesion characteristics. A total of

30 test samples were used generally representing all air dried thin and medium primer thickness samples which had failed in tests 4A, 4B, 4C and 4D as indicated in Tables 9 and 10. Three additional test samples were also included which had been retained from Tests No. 1 and No. 2. These samples were cured at 150°F at intervals of 4, 8 and 16 hours as shown in Table 11 and then tested under thermal-vacuum conditions for two complete cold-hot cycles. All coupons successfully passed this test and no definite conclusions could be drawn regarding the elevated temperature cure time-temperature relationship oterh than the fact that a short baking time is adequate if the primer thickness is relatively thin and a sufficient air dry cure has been performed.

8.0 IITRI TEST PROGRAM

The investigation performed at IITRI as a part of the overall S-13G thermal coating adhesion failure study was performed by F.O. Rogers of IITRI. This portion of the study was primarily mechanical, chemical, and application oriented and served as a guide to thermal-vacuum tests performed by Bendix. This section of this report specifically covers the IITRI investigation and presents the results of those tests performed at IITRI.

A preliminary diagnosis indicated that adhesion loss occurred at the interface between the substrate and primer. Subsequent diagnosis proved that the failure was due to a catastrophic loss of adhesion between the substrate and the SS-4044 primer. The photomicrograph (200X) presented in Figure 12 clearly shows the bulk of the primer to be attached to the "failed" coating, which was taken from failed ALSEP hardware.

Mechanical adhesion tests, bending, and LN^2 dip were the primary techniques used in determination of both primer and S-13G adhesion characteristics. Chemical tests with solvents and dyes were used to determine the effectiveness of primer and S-13G cure and to substantiate the region of adhesion failure.

8.1 Batch Defect Tests

The possibility of Flight 1 batch defects was investigated using substrate panels and coating them with retained amounts of the GE SS-4044 primer lot used on the Flight 1 sunshield and S-13G (batch A-524).

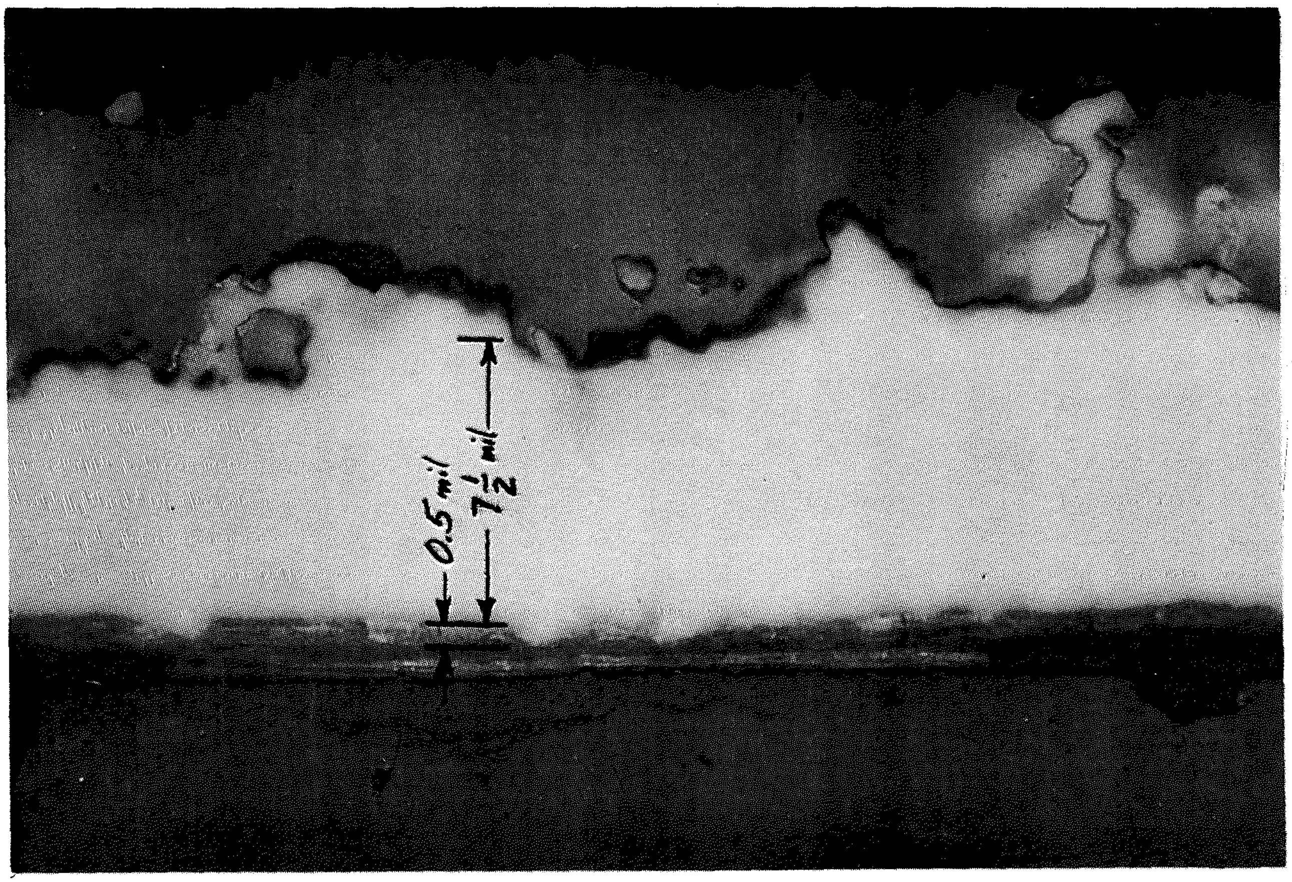

Figure 12 Photomicrograph (200X) of Cross-Section of S-13G Spalled from ALSEP Flight Model.

The primer was applied to the clean etched panel at a 0.3 mil thickness. After a 1 hour air cure at room temperature, S-13G was applied over the primer at an 8.0 mil thickness and allowed to air dry for 24 hours at room temperature. The test panel was then dipped in liquid nitrogen (LN_2) for 20 minutes and upon removal no loss of adhesion was apparent. An original cure test panel of batch A-524 was taken from the laboratory files and bent over a mandrel of approximately 1/2 inch radius. This panel when dipped in LN_2 for 20 minutes did not exhibit adhesion failure.

Three different primer batches (316, 348 and 278) were applied to a clean etched 2024 aluminum panel at a 0.3 mil thickness. Each batch sample was used to cover one third of the panel. After a 1 hour air cure at room temperature, 10 mils of S-13G from batch A-742 was applied over the entire panel and then air dried at room temperature for four days. The panel was then placed in an oven for four hours at 230°F. Upon removal from the oven, the sample was then placed in a vacuum overnight and then dipped in LN_2 the following morning for 20 minutes. No loss of adhesion was observed on any portion of the test panel.

The conclusions reached from the batch tests are as follow:

1. No differences in adhesion using the Flight 1 primer batch and new primer batches were apparent. All were satisfactory under test conditions.
2. The length of time on the substrate does not account for loss of adhesion on Flight 1 and age is not a significant factor.

8.2 Substrate Surface Preparation

Tests to determine effect of substrate surface preparation upon adhesion characteristics were performed using three basis substrate materials supplied by Bendix. Samples of each of the materials were prepared, primed, coated with S-13G and then dipped in LN_2 for 20 minutes.

The three basic aluminum substrate materials used were; (1) 2024-T3 Alclad, (2) 6061-T6 Chem Film, and (3) 7075-T6 Anodized. Six samples of each substrate material were then prepared as follows to produce a total of 18 test specimens:

1. No cleaning of substrate (as received condition).
2. Surface cleaned with Alconox solution.
3. Surface solvent-wiped only.
4. Surface abraded and then solvent-wiped.
5. Surface degreased; etched with 5% NaOH, rinsed; de-smutted with 15% HNO_3, rinsed; and dried with alcohol.
6. Surface etched as in number 5 then contaminated with grease and then solvent-wiped.

To each sample, 0.3 mils of SS-4044 primer (batch 316) was applied and air dried one hour at room temperature. Each sample then was coated with S-13G at a thickness of 10 mils and then air dried 48 hours at room temperature. Upon completion of air dry cure, the samples then were cured 2 hours at 230°F. After baking, they were placed in a vacuum for 2 hours and then were dipped in LN_2 for 20 minutes. No adhesion failures were observed on any of the test samples regardless of substrate surface preparation or treatment.

It was concluded from this test that the degree of cleaning or surface preparation of the substrate was of little or no importance with the primer applied at a 0.3 mil thickness.

8.3 Primer Thickness Test

To determine the effect of primer thickness upon S-13G adhesion characteristics, primer was applied to clean etched 2024 aluminum panels at various thicknesses. The primer was air dried at room temperature for 1 hour prior to receiving 10 mils of S-13G. The S-13G was air dried 16 hours at room temperature and then cured 2 hours at 230°F. The panels were then placed in a vacuum for 16 hours and then dipped in LN_2. Table 12 summarizes the test results for the various primer thickness values tested.

The primer thickness test results showed that primer thicknesses of 0.8 mils or greater failed in adhesion under the LN_2 dip test. Examination of the failures showed that the failure occurring at the primer/substrate interface was similar to that experienced on Flight 1 hardware.

8.4 Colored Primer Test

To verify the primer/substrate failure, test samples of abraded 2024 Alclad with SS-4044 primer pigmented with light red oxide (1 ounce

TABLE 12 IITRI PRIMER THICKNESS TEST

Primer Thickness	LN_2 Dip Test Result
1. No Primer	No Adhesion Failure
2. 0.05 Mils	No Adhesion Failure
3. 0.25 Mils	No Adhesion Failure
4. 0.80 Mils	Failed in Adhesion
5. 1.10 Mils	Failed in Adhesion
6. 1.80 Mils	Failed in Adhesion

NOTE: Sample Preparation

1. 2024 Cleaned and etched
2. Primer batch; No. 316 (same as flight 1)
3. Primer Cure; 1 hr air dry @ room temp
4. S-13G batch; A-742
5. S-13G thickness; 10 Mils
6. S-13G cure; 16 hrs @ room temp., 2 hrs @ 230°F, 16 hrs in vacuum.

oxide for 1 gal of primer) were fabricated and tested. Three panels (3" x 6") were prepared using various primer thicknesses and cures prior to S-13G application as shown in Table 13. Only one adhesion failure was encountered in the LN_2 dip test (panel No. 3) with the underside of the S-13G film being a deep red in color confirming the primer/substrate interface type adhesion failure. The metal face showed only slight traces of red in the pores of the metal.

The results of the colored primer tests led to the following conclusions:

1. A thick primer (0.8 mils or greater) results in adhesion failure.
2. Length of primer cure time of 1 hour, 16 hours or at elevated temperature is not significant compared to primer thickness effects.
3. Thickness of S-13G coating is apparently of no consequence compared to primer thickness.

All of the coatings failed (the films lifted off) except the specimens primed with 0.8 mils of baked red and clear primer; these, however, showed incipient blisters. A photomicrograph (200X) presented in Figure 13, was taken of the failed system prepared from red primer (0.8 mil) and 10 mils of S-13G. The figure clearly shows the line of demarcation between S-13G and primer.

8.5 Primer Thickness and Cure Test

To insure that the use of red oxide as a primer coloring agent did not alter primer adhesion characteristics, a special primer thickness and cure test was performed to verify previous test results. Twelve 2024 Alclad test samples were prepared (abraded and solvent wiped) and primed as shown in Table 14. After completion of primer cure, all samples were given a 10 mil application of S-13G and air dried 24 hours at room temperature. The samples were then dipped in LN_2 with the results as indicated in Table 14. All samples except the 0.8 mil primer thickness ones showed adhesion failure. The 0.8 mil primer thickness samples showed damage in the form of incipient blisters.

It is concluded from this test that: (1) all primer application should be kept below 0.8 mils thickness, (2) elevated temperature cure improves primer adhesion only in borderline cases and (3) the red oxide did not affect primer adhesion.

TABLE 13 IITRI COLORED PRIMER TEST

Panel No.	Red Primer Thickness	Red Primer Cure	S-13G Thickness	S-13G Cure	LN_2 Dip Test Result
1	0.5 Mils	1/2 16 hrs @ Room Temp 1/2 1 hr @ Room Temp	10 Mils	96 hrs @ Room Temp.	No Adhesion Failure
2	0.25 Mils	16 hrs @ 230°F	1/2 7 Mils 1/2 14 Mils	96 hrs @ Room Temp.	No Adhesion Failure
3	1/2 0.2 Mils 1/2 1.2 Mils	16 hrs @ Room Temp.	10 Mils	96 hrs @ Room Temp.	1/2 Having 1.2 Mil Primer Thickness Failed. Other 1/2 Had No Adhesion Failure

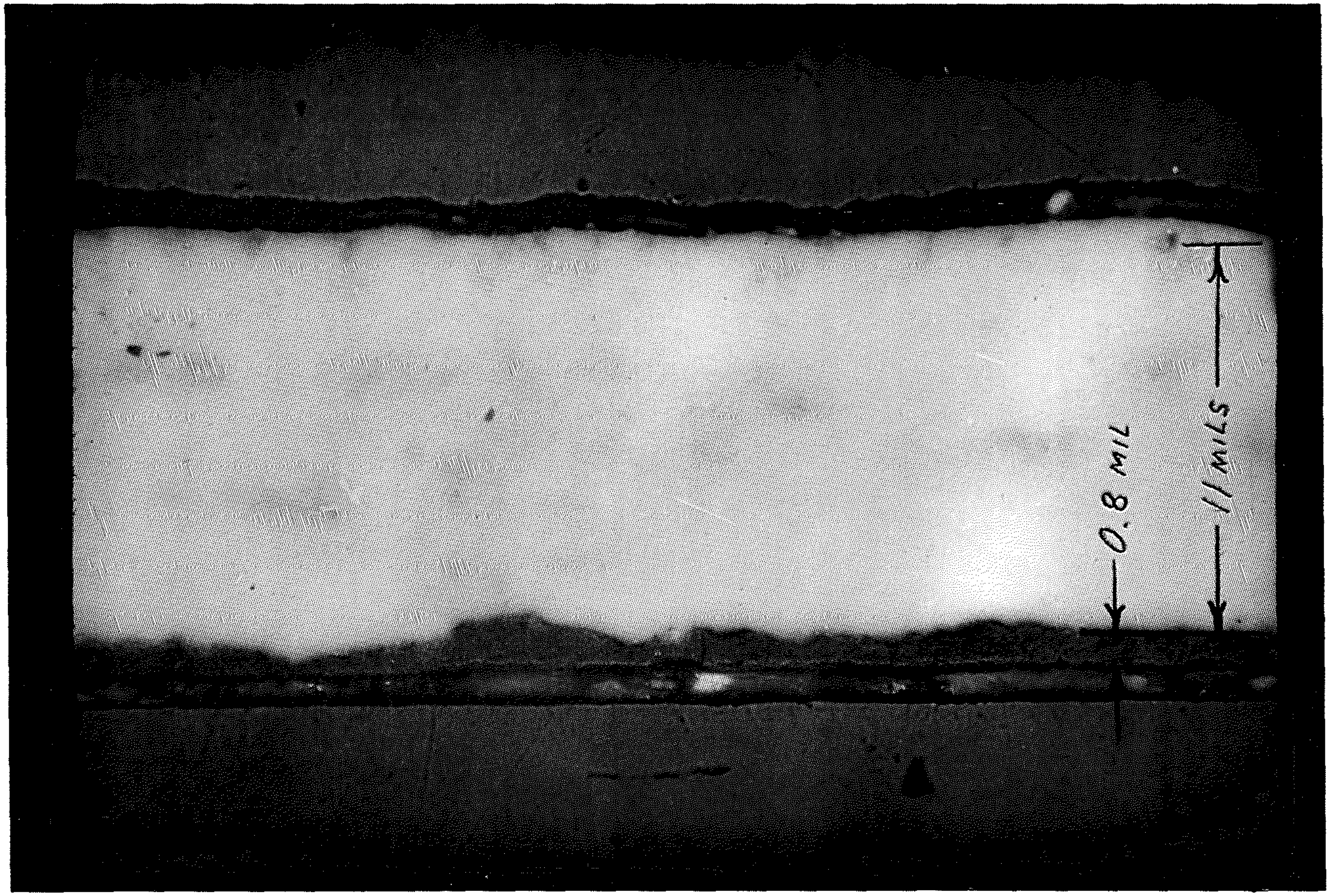

Figure 13 Photomicrograph (200X) of Cross-Section of S-13G Test Coating With Red-iron Oxide-pigmented S. S. 4044 Primer.

TABLE 14 IITRI PRIMER THICKNESS AND CURE TEST

Primer Color	Cure	Thickness ~ Mils		
Clear	16 hrs @ Room Temp.	0.8	1.2	1.6
Clear	16 hrs @ 230°F	0.8	1.2	1.6
Red	16 hrs @ Room Temp.	0.8	1.2	1.6
Red	16 hrs @ 230°F	0.8	1.2	1.6

Substrate: 2024 Alclad abraded and solvent wiped

S-13G Application: 10 Mils with 24 hr room temp. cure

9.0 CORRECTIVE ACTION FOR FLIGHT HARDWARE

At the time of the S-13G adhesion failure during the Flight 1, acceptance test, portions of Flight 2 hardware (thermal plate and primary structure) had already received final application of S-13G thermal coating per the same manufacturing process (MP-64) as used for Flight 1. To minimize impact upon delivery schedule of all hardware units, interim changes to the application procedure were implemented which if proven by the Bendix and IITRI adhesion tests would result in acceptable S-13G thermal coating adhesion. This section of the report details the action taken on each Flight hardware unit affected (Table 15) and also includes the final revisions to M.P. 64 for all units receiving S-13G application subsequent to 16 Sept. 1968.

9.1 Flight 1 Recoating

The failed surfaces of the Flight 1 unit (sunshield and primary structure) were stripped of all remaining S-13G thermal coating after completion of the acceptance test. These surfaces were then abraded to remove all normal substrate surface treatment films. After cleaning and rinsing to a water break-free condition, the surfaces were air dried and new GE SS-4044 primer (batch 349) was applied at a 0.5 mil thickness. The primer coat was air dried at room temperature for 16 hours and S-13G then was applied at 9-12 mils thickness. After a 48 hour air dry at room temperature, the Flight 1 model was then reassembled and allowed to continue air curing at room temperature over an approximate 1 1/2 month period in a protected environment pending adhesion test results.

Upon completion of Test No. 4D it was concluded that the test results indicated with the primer thickness of 0.5 mils used in the recoating, a 24 hour cure at 150^{o}F was sufficient to provide successful adhesion of S-13G on the sunshield and primary structure. To demonstrate the correctness of this action, two test sample portions remaining from Test No. 1 (2024-T3 No. 2 and 7075-T6 No. 5) were cured along with the sunshield and primary structure. These samples then were tested in Test No. 4E and successfully completed the test from which it was concluded that the sunshield and primary structure recoating and cure procedure was adequate based upon all test results to date.

TABLE 15 FLIGHT 1 & 2 HARDWARE S-13G COATING STATUS

Unit	Surface	Date Primed	Primer Thickness	Primer Cure	Date Coated	Coating Thickness	Coating Cure	Remarks
Flight 1	T. Plate	2-23-68	0.4-0.5 mils	1 hr @ Room Temp.	2-24-68	14-15 mils	48 hr @ Room Temp.	Passed Thermal-Vacuum Acceptance Test
	P. Structure	7-8-68 (Recoat)	0.5 mils	1 hr @ Room Temp.	7-9-68 (Recoat)	9-12 mils	48 hr @ Room Temp.	Abraded Substrate: This surface has additional cure of 24 hrs @ 150°F, samples have passed T/V tests
	S. Shield	7-8-68 (Recoat)	0.5 mils	1 hr @ Room Temp.	7-9-68 (Recoat)	9-12 mils	48 hr @ Room Temp.	Abraded Substrate: This surface has additional cure of 24 hrs @ 150°F, samples have passed T/V tests
Flight 2	T. Plate	5-31-68	0.4-0.6 mils	1 hr @ Room Temp.	5-31-68	9-12 mils	48 hr @ Room Temp.	T/V Acceptance Test to verify adhesion
	P. Structure	6-13-68	0.6 mils	1 hr @ Room Temp.	6-14-68	12-12 mils	48 hr @ Room Temp.	Substrate not abraded; anodized surface T/V Acceptance Test to verify adhesion
	S. Shield	7-21-68	0.25 ± 0.1 mils	16 hr @ Room Temp.	7-22-68	6-8 mils	48 hr @ Room Temp. + 24 hrs @ 150°F	Abraded substrate: Control coupons fabricated New QC documentation used

10.0 FLIGHT 2 STATUS

The Flight 2 thermal plate was coated with S-13G prior to the Flight 1 model adhesion failure under the same M.P.-64 procedure was originally used on Flight 1. The Flight 2 thermal plate was subjected to T/Vacceptance tests from 24 September to 3 October 1969 during which time the adhesion characteristics of the S-13G coating were adequately demonstrated. The Flight 2 primary structure was also coated prior to the Flight 1 model failure in the same manner as Flight 1 components. One additional step performed on the primary structure was to cure at 150^{o} for 24 hours as indicated by adhesion test results. The adhesion characteristics of the primary structure were also demonstrated during the above referenced T/V acceptance testing.

Flight 2 sunshield was not coated with S-13G until after Tests No. 1 and No. 2 were completed and some information from IITRI tests was available also. Thus this surface is significantly different in coating application from previous hardware and was coated in a manner nearly identical to that indicated by adhesion test results. The Flight T/V testing again indicated successful adhesion of the coating to the sunshield surface.

11.0 FLIGHT 3 AND SUBSEQUENT FLIGHTS

Effective 16 September 1968, all flight hardware surfaces receiving S-13G thermal coating were processed under the revised M.P.-64. The new application procedure (Table 16) placed emphasis upon maintaining a thin primer thickness, adequate cure of primer and S-13G coating, revised shelf life requirements of S-13G thermal coating materials, coupon control and revised documentation verifying all primer and coating process steps. Several of the changes in the revised M.P.-64 are precautionary in nature since the thermal-vacuum tests were not of as wide a scope as desired. All changes are designed to provide the best possible S-13G adhesion characteristics within the framework of the test program reported herein.

12.0 COMMENTS ON APPLICATION OF TEST RESULTS

A general analysis of all test results (both Bendix and IITRI) show that the adhesion characteristics of S-13G thermal coating at cryogenic temperatures is a function of many interrelated factors. The tests point out however a few basic precautions that must be observed

TABLE 16 M. P. -64 REVISION FOR FLIGHT 3 AND SUBSEQUENT FLIGHTS

1.	Primer Thickness	0.1 to 0.25 mils
2.	Primer Cure	16 hrs @ Room Temp.
3.	S-13G Thickness	9-12 mils
4.	S-13G Cure	48 hr @ Room Temp. Plus 24 hrs @ 150°F
5.	Materials Shelf Life	
	GE SS-4044 Primer	3 Months Under Refrigeration
	S-13G Thermal Coating	30 Days Under Refrigeration
	SRC-05 Catalyst	30 Days Under Refrigeration

6. Substrate Preparation

   Abrade to remove all normal substrate treatment prior to primer application

7. Cleaning Solutions

   5% Alconox or liquid Joy (no abrasive cleansers)

8. Wipe Cleaning

   200% Eythl alcohol (Anhydrous Anhydral)

9. Documentation required on all primer and coating process steps

10. Coupon control to provide hardware samples

at all times to obtain satisfactory adhesion on a routine basis under normal manufacturing conditions. This section summarizes the factors which significantly affect S-13G adhesion and can be reasonably controlled during application.

The thickness at which the GE SS-4044 primer is applied is a major consideration in S-13G coating operations. Even though tests results indicate that primer thicknesses of as great as 0.5 mil can be made to adhere properly, the applied thickness should not exceed a value of approximately 0.25 mil in order to provide an adequate margin of safety. The tests also indicate that multi-layer primer applications are undesirable and should be avoided if possible. It is recognized that in some hardware applications, multi-layer primer application may be unavoidable due to hardware configuration and such applications must be cured properly to prevent degradation of the initial layers by solvents and volatiles contained in the overcoat. Whenever multi-layer primer application is used (even though final film thickness does not exceed 0.25 mil), a 16 hour, room temperature, air dry is recommended with a 24 hour elevated temperature cure (150^{o} -200^{o}F) of the applied S-13G coating.

Single layer primer applications which are room temperature air cured for 1 hour or more can give good adhesion if the thickness is held to 0.15 mil or less and the S-13G is cured at room temperature for a minimum of 48 hours. To provide an adequate safety margin it is recommended that all S-13G applications be cured an additional 24 hours at an elevated temperature of 150^{o}F since test results showed a definite improvement in overall adhesion characteristics due to this additional cure process step.

The thickness of the S-13G coating of either single or multi-layer application apparently is not a significant factor in obtaining good adhesion if proper curing of the coating occurs. Application schedule of the S-13G coating appears to have little effect upon adhesion characteristics if properly cured. Although various cure times of the S-13G were not explored in detail, a minimum of 48 hours at room temperature is recommended prior to the elevated temperature (150^{o}F cure. This allows sufficient time for solvents and volatiles to evaporate, thus minimizing any danger of entrapment to form blisters or bubbles at the higher temperatures.

Substrate surface treatment apparently is of secondary importance since good adhesion was experienced with little or no special preparation in several cases. The controlling factor is most probably associated with the degree of surface porosity and general cleanliness. The presence of Alclad, Anodize or other surface treatment film apparently minimizes surface porosity resulting in a lower adhesive capability. Removal of these treatment films by abrasion considerably enhances adhesion success and may save a marginal application of S-13G coating. To provide an additional margin of safety it is recommended that all surface treatment films be removed by abrasion wherever possible. General surface cleanliness and dryness should also be observed on any substrate prior to primer application to minimize contamination at the primer/substrate interface.

Quality of primer and S-13G coating may have a significant effect upon adhesion characteristics. Although these aspects were not examined in great detail, there were indications that materials degradation from shelf environment may be contributory to adhesive failure. Obvious materials defects of S-13G have been detected from time to time even though the expiration dates had not been exceeded. To minimize the occurrence of such defects, shelf life requirements have been revised at Bendix (see M.P.-64 revisions) to insure acceptable coating materials quality.

Application technique during primer and coating operations is important in order to maintain uniform primer thickness. Although no particular problems were encountered in the preparation of test coupons, the irregular surfaces encountered on actual hardware often make it difficult to obtain a uniform primer thickness. Build up of primer may occur in corners, fillets, joints, etc. resulting in a thickness susceptible to adhesion failure. A single application of primer to the proper thickness is desirable, however, an area which may have a thickness less than specified should not be recoated if the first coat is continuous with no bare spots. Any excess overlap in attempting to come within specified thickness can be more detrimental than thinness of coating if a room temperature cure of both primer and coating is used. Thus application technique is interrelated to thickness and cure process. The particular method used must be compatible with both and any touch-up or recoat operations must be well controlled to prevent any adhesion problems. Basically application technique is an operator proficiency problem and thickness measurements should be made at critical points in coating operations.

An area of concern resulting from the Bendix tests was inability of the LN_2 dip test to duplicate the adhesion failures experienced in a thermal-vacuum environment. In Tests 4C and 4D, only 42 percent of the failures obtained in Tests 4A and 4B were duplicated by the LN_2 dip test.

The only logical conclusion that seems to explain this discrepancy is that the adhesion failure mechanism is not solely a thermal shock phenomenon. The thermal shock experienced by the coating in an LN_2 dip test is very severe compared to that experienced in thermal-vacuum thermal cycling but thermal vacuum tests produced more adhesion failures. It is theorized that the adhesion failure mechanism is a combination thermal shock-vapor pressure phenomenon. This would explain in part the onset of adhesion failure in the formation of a bubble observed in Test No. 1 and on the primary structure in the Flight 1 acceptance test.

13.0 ADDITIONAL PROPOSED S-13G ADHESION TESTING

Based on the previous testing and results obtained, it is recommended that additional adhesion tests be conducted to:

1. Develop the basic primer cure processes.

   (a) Air dry (time and temp.)
   (b) Elevated temp. (time and temp.)

2. Investigate the primer adhesion (multi-layer) as influenced by the application schedule.

3. Develop the basic S-13G cure process compatible with primer application.

4. Determine the influence of S-13G application schedule on adhesion characteristics.

5. Investigate the adequacy of the LN_2 quench test as a legitimate standard adhesion test.

6. Evaluate additional primers and substrates.

7. Investigate different thinner spraying solutions, i.e., replace toluene.