

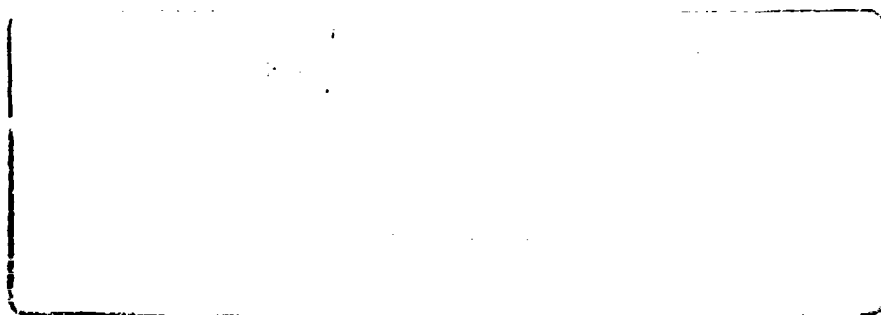
UNCLASSIFIED

AD NUMBER
ADB006345
NEW LIMITATION CHANGE
TO Approved for public release, distribution unlimited
FROM Distribution authorized to U.S. Gov't. agencies only; Proprietary Info.; Oct 1966. Other requests shall be referred to the Army Missile Command, Attn: AMSMI-RPR, Redstone Arsenal, AL 35809.
AUTHORITY
USAMC, per ltr dtd, 11 Dec 1975

THIS PAGE IS UNCLASSIFIED

AD No.
DDC FILE COPY

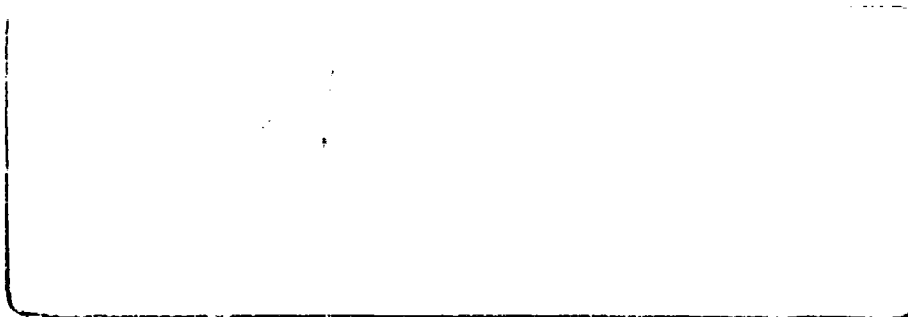
AD B006345



SEP 8 1975
B



Aeronutronic 
Aeronutronic Ford Corporation
Aeronutronic Division
Newport Beach, California



ACCESSION for	
NTIC	Write Section ☐
SUC	Mail Section ☒
UNANNOUNCED	☐
JUSTIFICATION.....	
BY	
DISTRIBUTION/AVAILABILITY CODES	
Dist.	AVAIL. and/or SPECIAL
13	

per letter on file

14
Publication No. U-3792

15
Under Contract:

DA-01-021-AMC-14097(Z)

10 61p.

9
6
TECHNICAL REPORT.

CHAPARRAL WEAPON SYSTEM
BLAST PROTECTIVE COATING INVESTIGATION.

Distribution limited to U.S. Gov't. agencies only;
Proprietary Info.: 37972 Other requests
for this document must be referred to

Prepared for: U. S. Army Missile Command
Redstone Arsenal, Alabama 35809
ATTN: AMSMI-RPR

Prepared by: 10 J. P. Hoffman and H. Hop
Mechanical Engineering Department

Approved by:

W. F. MacInnes
Manager
Mechanical Engineering

R. S. Leith
Technical Manager
Chaparral Weapon System

V. D. Iglesias
Program Manager
Chaparral Weapon System

14
10 Oct 1966

AERONUTRONIC
DIVISION OF PHILCO-CORPORATION
A SUBSIDIARY OF Ford Motor Company,
FORD ROAD NEWPORT BEACH, CALIFORNIA

DDC
REFILED
SEP 8 1965
B

401965

NOTICE

Aeronutronic considers the epoxy-nylon and epoxy-urethane formulations described herein as potentially patentable, and has taken steps to patent them for use as extreme temperature range ablative coatings.

SUMMARY

A literature survey and experimental investigation have been conducted for the purpose of obtaining a coating that could be applied to the surface of the Chaparral fire unit to protect it from the detrimental effects of the Sidewinder motor exhaust during missile launch. The results of the literature survey indicate that a significant amount of experimental work has been performed on the thermal protection of exposed structure for fixed launch sites, but only a limited amount of work has been performed on the protection of structure for mobile launchers. The coating data that is available for mobile launchers has been limited primarily to the investigation of material performance in a rocket exhaust environment. Very little effort has been expended in the evaluation of protective coating materials when exposed to all the other environmental requirements (i.e., temperature extremes, temperature and mechanical shock, humidity, fluid exposure, etc.).

In view of this fact, an experimental program, Phase I, was conducted at Aeronutronic in which 15 potential coating systems were evaluated under simulated Chaparral environments, excluding jet erosion. The 15 selected materials included the most promising coatings from the results of the jet erosion work performed by the Army Ground Support Equipment Laboratory on the Mauler Program. Also, the remaining coating materials were selected because they exhibited good jet erosion characteristics from tests performed by other agencies. The application techniques required for the 15 coatings consisted of seven trowelable, six precured bonded sheets and one bonded laminate. The results of the experimental investigation showed that only four out of the 15 coatings passed the environmental test program. These four coatings consisted of two phenolics and two of the butadiene acrylonitrile rubbers. All of the successful candidates belong to the precured bonded sheet family of coatings. Since these coatings are rather inflexible, they will present application problems if the surface to be protected is not smooth.

For this reason, a follow-on program, Phase II, was initiated to find a sprayable or trowelable coating that would pass the Chaparral requirements. In addition to looking for new coatings, the trowelable/sprayable coatings, used in Phase I, were modified to help them overcome their deficiencies. Nineteen coating systems were evaluated in Phase II under essentially the

same environmental conditions used in Phase I. The experimental investigation showed that three out of the initial 19 coatings could pass the Chaparral environmental requirements. These three successful candidates are trowelable epoxy-polysulphide, epoxy-nylon and epoxy-urethane type materials. They should be subjected to subscale jet erosion testing in the near future and the most promising candidate recommended for application to Engineering Models 1 through 5.

CONTENTS

SECTION	PAGE
1 INTRODUCTION	1
2 LITERATURE SEARCH.	2
3 COATING DESIGN REQUIREMENTS.	8
4 EXPERIMENTAL INVESTIGATION	
4.1 General Description of the Test Program	10
4.2 Test Conditions and Procedures.	14
4.3 Test Specimen	14
4.4 Selection of Candidate Coatings	15
5 DISCUSSION OF TEST RESULTS	
5.1 Phase I Test Results.	19
5.2 Phase II Test Results	23
6 CONCLUSIONS AND RECOMMENDATIONS.	27
REFERENCES	30
APPENDICES	
A TEST PROCEDURES	
A.1 Phase I Test Procedures	A-1
A.2 Phase II Test Procedures.	A-10
B TEST SPECIMEN PREPARATION	
B.1 Phase I Specimen Preparation.	B-1
B.2 Phase II Specimen Preparation	B-1
DISTRIBUTION	D-1

ILLUSTRATIONS

FIGURE		PAGE
1	Protective Coating Test Tree for Phase I	11
2	Protective Coating Test Tree for Phase II.	12

TABLES

TABLE		PAGE
I	Materials Tested by the Army Ground Support Equipment Laboratory on the Mauler Program	4
II	Materials Tested by Martin Company on the Pershing Program.	6
III	Coatings and Adhesive Systems Selected for Evaluation in Phase I.	16
IV	Coatings and Adhesive Systems Selected for Evaluation in Phase II	17
V	Summary of Phase I Environmental Test Results.	20
VI	Summary of Phase II Environmental Test Results	24
B.1	Phase I Application Procedures - Troweled Coatings .	B-3
B.2	Phase I Application Procedures - Castable Coatings . .	B-5
B.3	Phase I Application Procedures - Bonded Precured Tile Coatings.	B-6
B.4	Phase I Application Procedures - Bonded Laminate/Coatings	B-9
B.5	Phase II Application Procedure Information	B-10
B.6	Phase II Aeronutronic Coating Formulations	B-12

SECTION 1

INTRODUCTION

↙ The purpose of this report is to present the results of the work performed to date directed towards developing a coating to protect the Chaparral Fire Unit from the detrimental effects of the Sidewinder exhaust plume. → p. 27

At the beginning of the Chaparral development program, it was recognized that some method of attenuating or eliminating the missile exhaust blast effects on the launcher structure would have to be provided in order to obtain a lightweight low cost system. Various techniques to overcome the detrimental effects of rocket motor blast on adjacent structure have been considered by other agencies with varying degrees of success (References 1 through 4). The technique of coating the exposed launcher structure with a material that could be readily repaired in the field was selected by Aeronutronic as having the most potential.

Following a survey of the work performed by other agencies, it quickly became obvious that considerable effort had been expended throughout the country in search of low erosion rate materials. Unfortunately, very little information was available to confirm the ability of these same materials to maintain their integrity when exposed to environments other than missile blast. For this reason, an experimental program was initiated at Aeronutronic to identify and solve the problems associated with using erodable coating materials, so that one or more potential coatings could be developed to meet the specified Chaparral environmental requirements.

SECTION 2

LITERATURE SEARCH

In the past 20 years, a significant amount of theoretical and experimental work (References 5, 6, 7, 8, and 18) has been performed on the problem of providing thermal protection for exposed structure around fixed launch sites. Since the fixed launch sites did not have to contend with environmental extremes and weight restrictions, it was possible to consider refractory materials (i.e., firebrick, cement, fused silica, etc.). Refractories did provide the low cost, low erosion rate coatings necessary to protect first generation IRBM and ICBM launch pads. Even to this time refractories are still being used for the thermal protection on fixed launch sites.

With the advent of smaller tactical launch systems such as Pershing and Mauler, it was recognized that application of conventional refractories to these systems for blast protection would greatly reduce their effectiveness in terms of field mobility and cost. Consequently, all of the effort on blast protection for these systems has attempted to take advantage of the state-of-the-art development of high temperature materials. Unfortunately, many of the materials that have been developed in related fields (re-entry nose cones, propulsion chamber insulation, etc.) are impractical for systems like the Chaparral because of their brittleness and high initial cost to say nothing of the field maintainability problems.

One of the agencies that has performed work pertinent to this field is the Army Ground Support Equipment Laboratory (GSEL) on the Mauler Program. They have exposed many classes of blast protective materials to the Mauler launch blast environment. This blast environment consisted of exposure of the materials for about 0.1 second, to an exhaust stream having a downstream stagnation pressure and temperature of 330 psia and 5800°F, respectively. The aluminum oxide content in the exhaust was approximately 33 percent by weight. The

specimens were positioned so that the impingement of the exhaust stream was within 15 degrees of the material surface normal on all firings. The jet erosion results are presented in Table I for all materials which could have potential application to the Chaparral system. The amount of material erosion that occurred from a Mauler firing is presented for each coating. If a section of the coating was torn away during a firing, the coating was considered to be partially destroyed. A more detailed description of the test procedures and test results can be obtained in Reference 1. It should be noted that the primary objective of the Army GSEL work was to obtain material jet erosion data. Hence, no information was obtained on the ability of these materials to withstand other types of environments.

The one other company that has performed pertinent jet erosion work is the Martin Company on the Pershing Program. They exposed approximately 40 materials, all 1/2-inch thick, to the exhaust blast from a Pershing booster simulator. The simulator provided hot gases for 1.2 seconds at an estimated stagnation pressure and temperature of 200 psia and 5000°F. The exhaust stream had an aluminum oxide content of 40 percent by weight. The jet impinged normal to the material surface during all firings. The test results for the materials that might have potential application to the Chaparral Weapon System are presented in Table II. The primary consideration on this program, also, was the jet erosion performance of the materials. Therefore, no environmental performance data were available on the materials.

TABLE I
MATERIALS TESTED BY THE ARMY GROUND SUPPORT
EQUIPMENT LABORATORY ON THE MAULER PROGRAM

<u>Manufacturer and Designation</u>	<u>Filler Materials</u>	<u>Maximum Erosion Depth per Firing (inches)</u>
Epoxies		
Schramm 7594Y41	Fiberglass	0.030
Raytheon 2145	Unknown	0.030
Raytheon 2138	Unknown	0.040
Product Techniques PT2090	Unknown	0.045
CCL-Aberdeen 480-1063	Unknown	0.070
CCL-Aberdeen 480-1063	Unknown	0.070
Devcon and GSEL	Stainless steel and silicate particles	0.080
Dynatherm E 300	Unknown	0.125
Raytheon 2140	Unknown	≈ 0.1
Siliconc Rubbers		
Lord Mfg. 336-1516-(8)	Unknown	0.012
Lord Mfg. 336-1650-(0)	Unknown	0.012
Lord Mfg. 336-1949-(9)	Unknown	0.015
Lord Mfg. 336-1651-(1)	Unknown	0.015

TABLE I (Continued)

<u>Manufacturer and Designation</u>		<u>Filler Materials</u>	<u>Maximum Erosion Depth per Firing (inches)</u>
Miscellaneous			
1/4 inch thick 17-4 Steel plate			Not detectable
Enjay Chem	WSX-5833	1/4 inch alkali metal silicate	≈ 0.02
Enjay Chem	LD-3076-M	1/4 inch alkali metal silicate	Not detectable
Raybestos-Manhattan	45 RPD	Unspecified	Not detectable
Raybestos-Manhattan	110 RPD	Unspecified	Not detectable
Raybestos-Manhattan	22 RPD	Unspecified	Partially destroyed
GSEL	64-31-A	Calcium oxide with unknown fillers	0.055
GSEL	64-32-A	Calcium oxide with unknown fillers	Partially destroyed

TABLE II
MATERIALS TESTED BY MARTIN COMPANY
ON THE PERSHING PROGRAM

<u>Manufacturer and</u>	<u>Designation</u>	<u>Filler Materials</u>	<u>Maximum Erosion Depth per Firing (inches)</u>
Epoxies			
Magnolia Chem	3906-A-B	Alumina particles	0.200
Magnolia Chem	3906-1A-1B	Fused silica particles	0.310
Dyna-Therm	F-700	Asbestos	Completely eroded
General Tire	7242-VI-50A	Unknown	Completely eroded
Butadiene Acrylonitrile Rubbers			
General Tire	372-V52R1	Unknown	0.330
General Tire	481-V50R1	Unknown	0.360
Neoprene Rubber			
Raybestos-Manhattan	Not specified	Asbestos inconel fabric	Completely eroded
Silicone Rubbers			
Aerojet	MMB-4	Fiberglass cloth	0.270
Fiberite	MX-473C	Fiberglass cloth	0.360
Dow Corning	DC-2048	Fiberglass cloth	0.440
General Electric	SE-5004	Fiberglass cloth	0.440
Dow Corning	DC-651	Fiberglass cloth	0.450
Aerojet	MMB-26	Fiberglass cloth	Completely eroded

TABLE II (Continued)

<u>Manufacturer and Designation</u>	<u>Filler Materials</u>	<u>Maximum Erosion Depth per Firing (inches)</u>
Phenolics		
Martin Co.	Not specified Fused silica particles	0.100
Martin Co.	Not specified Silicon carbide and zirconium oxide particles	0.140
Fiberite	Not specified Quartz fibers	0.140
3M Company	XP220F Pluton fabric	0.150
Fiberite	2625 Silica fabric	0.150
Trevarno	4P051-8 Silica roving	0.160
Raybestos-Manhattan	41 RPD Asbestos	0.160
Martin Co.	Not specified Fused silica particles	0.170
Hooker Chem	Durez 21833 Black Durez	0.170
Thompson	XM 12 Refrasil and rubber	0.270
Miscellaneous		
4130 steel with CrNi diffused coating		0.060

SECTION 3

COATING DESIGN REQUIREMENTS

The environmental design criteria defined for the Chaparral Weapon System operating under worldwide extreme conditions (Reference 19) were utilized for specifying environments in the coating evaluation test program. The environmental conditions selected from the criteria as being significant in the evaluation of any coating material are:

- (1) Temperature extremes.
- (2) Temperature shock.
- (3) Humidity.
- (4) Precipitation.
- (5) Fluid exposure (hydraulic oil, fuel, hydrochloric and sulphuric acids).
- (6) Vibration.

The design loads on the Chaparral Fire Unit and the XM-730 vehicles are presented in References 20 and 21, respectively. The information presented in these documents was used to formulate screening tests for potential coating systems. The loading conditions that the selected coating systems must successfully pass are the following:

- (1) Missile jet blast.
- (2) Mechanical shock (slamming doors and tools being dropped on the deck).

The detailed test conditions and test procedures are presented in Appendix A.

In addition to the firm requirements stated above, a number of soft requirements were imposed on the coating materials. These soft requirements are:

- (1) The coating shall be repairable in the field under fair weather conditions.
- (2) The required coating application and cure time should be kept to a minimum.
- (3) The protective coating should be applied by means of either a trowel or a spray technique.
- (4) The coating should have skid resistant characteristics.
- (5) The developed coating should either be a drab color or be capable of being colored by the addition of nonoily type, drab pigment.

SECTION 4

EXPERIMENTAL INVESTIGATION

An experimental investigation was conducted to determine the effect of environmental and design loads on the structural integrity of selected protective coating materials. The test program involved two phases of work. In Phase I, a total of fifteen coating materials were investigated. The most promising coatings from the results of the jet erosion testing on the Mauler and Pershing Programs were included in the fifteen selected materials. In Phase II, a total of nineteen trowelable or sprayable coatings were evaluated. The nineteen candidate coatings included trowelable and sprayable coatings which did not pass the physical testing in Phase I, but were subsequently modified to help them overcome these deficiencies.

4.1 GENERAL DESCRIPTION OF THE TEST PROGRAM

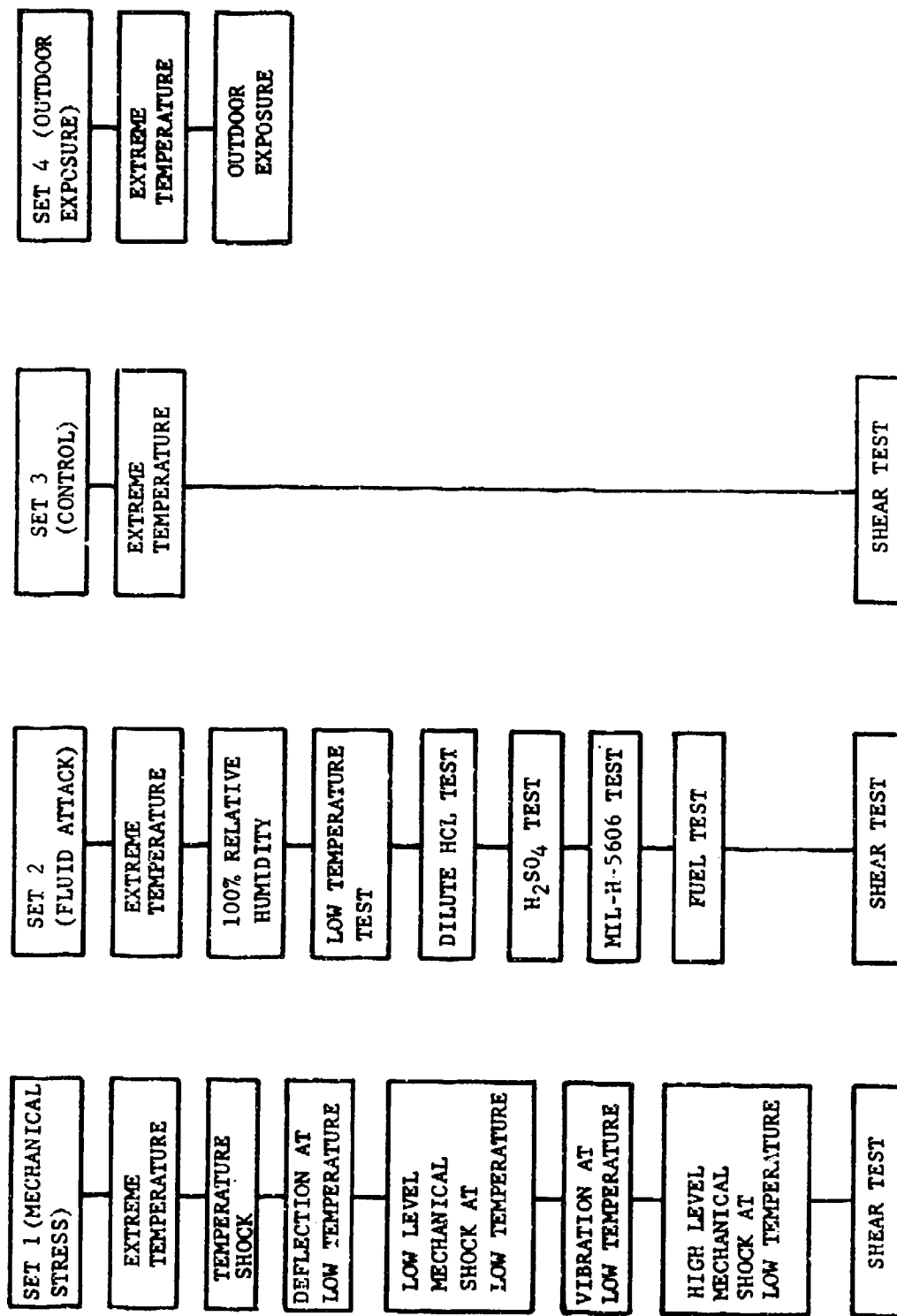
The protective coating test program involved two phases of work. The test trees for each phase are presented in Figures 1 and 2. For each test phase, four complete sets of the candidate coating materials were fabricated.

4.1.1 PHASE I TESTING

All the specimens were subjected to extreme high and low temperatures.

The specimens in Set One were then subjected to the mechanical stress series of tests in the order indicated on the test tree. This series consisted of:

- (1) Temperature shock
- (2) Mechanical deflection at low temperature
- (3) Low level mechanical shock at low temperature



F07420 U

FIGURE 1. PROTECTIVE COATING TEST TREE FOR PHASE I

- (4) Vibration at low temperature
- (5) High level mechanical shock at low temperature
- (6) Shear test.

The specimens in Set Two were subjected to a fluid attack series of tests after the extreme temperature test. This series consisted of exposing the coatings to:

- (1) 100 percent relative humidity
- (2) Low temperature
- (3) Dilute hydrochloric acid
- (4) Sulfuric acid
- (5) Hydraulic oil
- (6) Gasoline
- (7) Shear test.

The specimens in Set Three were retained as a control for comparison with the condition of the coatings as they progressed through the test program. This set was cut up for the shear test only after all other tests were completed. Set Four was placed on the roof of Aeronutronic Applied Research Laboratories building at Newport Beach to determine the combined effects of atmospheric conditions on the integrity of the coatings.

4.1.2 PHASE II TESTING

The testing in Phase II was similar to that performed in Phase I except that the vibration test was deleted and heel, air blast, and jet erosion tests were added to the program. The vibration test was deleted after Phase I because the bondline stress levels are low, less than 2 psi, and even the partially bonded coatings in Phase I passed this test.

The purpose of the heel test was to determine what detrimental effects a 200 pound man walking on the Fire Unit might have, should the coating be at 190°F temperature.

The air blast test was designed to investigate the ability of the coating edges to withstand the high fluid pressure levels resulting from direct impingement of the Sidewinder missile exhaust. It was noted from the results of the shear test in Phase I that some of the specimens had voids and/or passages between the coating and the metal substrate. This condition was

more prevalent in the pre-cured sheet specimens. The air pressure level used in this test is the maximum value that the Sidewinder motor exhaust would provide after impingement.

The purpose of the jet erosion test was to determine the coating's resistance to the heating and erosion rates which will be experienced when exposed to the Sidewinder missile blast environment.

The jet erosion and shear test will be completed in the near future and the results reported at that time.

4.1.3 COATING FAILURE CRITERIA

In establishing testing philosophy at the beginning of the program, it was decided that the complete failure of any candidate coating within either Sets One or Two would eliminate that candidate from all subsequent testing. The complete failure of a coating was defined as:

- (1) Thirty percent or more loss of bond.
- (2) Cracking, shattering, or delaminating of the coating to a point whereby its ability to protect the aluminum substructure from detrimental effects of the jet blast was questionable.
- (3) Significant changes in the coating thickness or hardness which could constitute a serious degrading physical or chemical change within the coating.

The coating thickness was measured immediately after each test. Also, following each test the specimens were visually inspected for cracking or separation of the coating from the substrate.

4.2 TEST CONDITIONS AND PROCEDURES

The test conditions and procedures utilized in each test for Sets One and Two are presented in Appendix A. The test conditions were derived mainly from the Chaparral Weapon System Environmental Criteria (Reference 19), the Integration/Support Structure Design Criteria (Reference 20), and the Chaparral Weapon System Vehicle/Pallet Interface Specification (Reference 21).

4.3 TEST SPECIMEN

Each candidate coating was applied 3/16-inch thick in the center 5.5 by 5.5 inch portion of an aluminum substrate. The substrates were machined from

a bare 1/2-inch thick aluminum plate into 9-1/2 by 9-1/2 inch pieces. Detailed information on the preparation of each coating is given in Appendix B.

4.4 SELECTION OF CANDIDATE COATINGS

4.4.1 PHASE I COATING SELECTION

Because so little information is available on the environmental behavior of erodable coating materials, primary emphasis at the beginning of the program was placed on selecting materials with only low erosion rates. At the same time, consideration was also given to obtaining a representative cross section of all feasible material classes. No refractories were included because of their inherent brittleness and difficulties in attachment and field repair.

A total of 24 suppliers of high temperature protective materials were contacted for recommendations. Included within the 24 were the suppliers of the 3 top material choices recommended by the Army GSEL (Reference 1).

The 15 coatings finally selected for evaluation were composed of 4 epoxies, 4 silicone rubber materials, 2 urethanes, 2 reinforced phenolics and 3 modified acrylonitrile butadiene rubber materials. All of these coatings, together with their adhesive systems, are described in Table III.

4.4.2 PHASE II COATING SELECTION

As a result of the high installation costs involved in using the preformed sheet coating materials which were the only successful candidates to complete the Phase I environmental test series, a second coating investigation phase was launched. Primary emphasis during this second phase was on developing new coatings and modifications to the more promising ones from Phase I, in order that a successful coating could be found to be applied, either trowel or spray equipment.

In an effort to overcome the differential contraction failures of the epoxy coatings during Phase I, semi-flexible primer materials were used between the epoxy coatings and their aluminum substrates. In addition, low temperature epoxies using nylon and urethane as fillers were formulated, none being available from outside sources.

Two new silicone rubber materials were also included in the Phase II selection. Both of these materials were evaluated with and within the hydrocarbon resistant coverings referred to as "top coat." The addition of the top coat coverings was an attempt to reduce the sensitivity of the silicone rubber coatings when exposed to gasoline and hydraulic oil.

Table IV describes the coating systems evaluated during Phase II.

TABLE III

COATINGS AND ADHESIVE SYSTEMS SELECTED FOR EVALUATION IN PHASE I

Coating Number	Manufacturer and Material Designation	Description	Adhesive System	
			Aluminum Primer	Adhesive
1.	International Coatings Co., Encoat Thermo-shield 13 H-26.	Modified polysulfide epoxy, semi-flexible.	None	None
2.	Products Techniques, Inc., PT 2090 (4764-27).	Modified epoxy, rigid	None	None
3.	Whittaker Corp., Narmco 4056.	Modified epoxy-phenolic, rigid.	None	None
4.	Dyna-Therm Corp., E-310F	Modified polyamide epoxy flexible.	None	None
5.	General Electric Co., TBS-758.	Modified silicone rubber.	General Electric Co., SS-4155.	None
6.	Dyna-Therm Corp., D-65, Cable Wrap.	Modified polyurethane flexible sheet, reinforced with glass scrim cloth.	None	Dyna-Therm Corp., D-65 modified polyurethane liquid.
7.	General Tire & Rubber Co., V-52.	Modified acrylonitrile butadiene rubber, asbestos and silica filled, flexible sheet.	None	General Tire & Rubber Co., V-61 modified polysulfide epoxy.
8.	Fiberite Corp., MX-2625.	Chopped 1/2 in. by 1/2 in. silica fabric reinforced modified phenolic, rigid molding.	Dow Corning Corp., 1200.	Dow Corning Corp., 93-046 modified silicone rubber.
9.	Raybestos-Manhattan, Inc., RPD 150.	Asbestos fiber reinforced phenolic, rigid molding.	Dow Corning Corp., 1200.	Dow Corning Corp., 93-046 modified silicone rubber.
10.	Dow-Corning Corp., 93-019.	Modified silicone rubber, flexible.	Dow Corning Corp., 1200.	None
11.	General Electric Co., 2365-2-2083.	Modified silicone rubber, heat vulcanizable flexible molding.	General Electric Co., SS-4155.	General Electric Co., RTV-630 modified silicone rubber.
12.	Magna Coatings & Chemical Corp., Metalox.	Modified polyurethane, flexible.	None	None
13.	Dow-Corning Corp., 93-046.	Modified silicone rubber, flexible.	Dow-Corning Corp., 1200.	None
14.	Corning Corp., Corco Division, CB6505.	Chopped 1/2 in. by 1/2 in. glass woven roving reinforced acrylonitrile butadiene rubber modified phenolic, semi-flexible molding.	None	General Tire & Rubber Co., V-61 modified polysulfide epoxy.
15.	Rockhill Rubber Co., 3,0-C-1051.	Low temperature resistant, modified acrylonitrile butadiene rubber, flexible sheet.	None	General Tire & Rubber Co., V-61 modified polysulfide epoxy.

TABLE IV
COATINGS AND ADHESIVE SYSTEMS SELECTED FOR EVALUATION IN PHASE II

<u>Coating Number</u>	<u>Manufacturer and Material Designation</u>	<u>Description</u>	<u>Aluminum Primer</u>
16	Gen Tire and Rubber V61	Modified polysulfide epoxy, semi rigid	None
17	Dyna-Therm Corp X-62-23	Modified epoxy silicone, semi rigid	None
18	Product Techniques PT2090-4763-27	Modified epoxy amine, rigid	Narmco 7344 catilized with Narmco 7119
19	Dyna-Therm Corp. E-310 F	Modified epoxy polyamide, semi rigid	Narmco 3170 catilized with Narmco 7133
20	Same as 19		Narmco 7344 catilized with Narmco 7119
21	General Electric 239-127-795	RTV silicone rubber, flexible	General Electric SS-4145
22	Same as 21 but with G.E. Traffic Coat		General Electric SS-4145
23	Dow Corning 93-063	RTV silicone rubber, flexible	Dow Corning 1200
24	Same as 23 but with DC94-024 Top Coat		Dow Corning 1200
25	Same as 19		B.F. Goodrich 934 with Narmco 3170 catilized with Narmco 7133 over the 934
26	Same as 19		B.F. Goodrich 934 with Narmco 7344 catilized with Narmco 7119 over the 934

TABLE IV (Continued)

<u>Coating Number</u>	<u>Manufacturer and Material Designation</u>	<u>Descript.</u>	<u>Aluminum Primer</u>
27	Aeronutronic 522-84-A	Modified epoxy urethane, rigid	B.F. Goodrich 934
28	Aeronutronic 522-84-B	Modified epoxy urethane, rigid	B.F. Goodrich 934
29	Same as 28		None
30	Aeronutronic 522-85-D	Modified epoxy urethane, rigid	B.F. Goodrich 934
31	Aeronutronic 522-86-A	Modified epoxy nylon, rigid	B.F. Goodrich 934
32	Aeronutronic 522-86-B	Modified epoxy nylon, rigid	B.F. Goodrich 934
33	Same as 30		3M Comp. y EC-2126
34	Same as 30		Dow Corning 4014

SECTION 5

DISCUSSION OF TEST RESULTS

5.1 PHASE I TEST RESULTS

Out of fifteen candidate coatings evaluated during Phase I, only four could be considered for additional evaluation in their "as tested" form. The four successful candidates were coatings 7, 8, 9 and 14; all preformed sheet stock.

A summary of the laboratory environmental test results is presented in Table V. Individual performance of each candidate is discussed in the following paragraphs.

5.1.1 LABORATORY ENVIRONMENTAL TEST RESULTS

Coating 1. Permanent softening of this coating following the extreme high temperature test indicated a physical change had taken place within the coating. The effect of this softening on the coating bond strength could not be determined immediately so the candidate was allowed to continue in the program. Following the humidity test it was noted that 30 to 50 percent of the specimens had become unbonded. This failure was probably due to differential contraction between the coating and substrate during the previous extreme low temperature test. Coating 1 was eliminated from consideration at this point.

Coatings 2, 3 and 4. Complete separation between these coatings and their substrates occurred after the extreme low temperature test. As in the case of Coating 1, differential contraction during low temperature exposure was larger than these rigid epoxies could tolerate.

TABLE V

SUMMARY OF PHASE I ENVIRONMENTAL TEST RESULTS

Coating Number	Type	Manufacturer and Designation	Test Results											
			Temp +160	Temp -65	Temp Shock	Deflect	Mech Shock	Vibrate	Humidity	Low Temp	Effluents HCl	H ₂ SO ₄	Gas	Hydrostatic Oil
1	Epoxy, Polysulfide	Int Coatings 13-M-26	D	--	--				F					
2	Epoxy	Product Techniques PT2090-4764-27	--	F										
3	Epoxy, Phenolic	Maraco 4056	--	F										
4	Epoxy	Dyna-Therm E310F	--	F										
5	Silicone Rubber	General Electric TBS-758	--	--	--	--	--	--	--	--	--	--	F	F
6	Urethane	Dyna-Therm D-65	--	D	D				F					
7	Acrylonitrile Butadiene Rubber	General Tire V-52	--	--	--	--	--	--	D	D	--	--	--	--
8	Phenolic, Chopped Silica	Fiberite MX2625	--	--	--	--	--	--	--	--	--	--	--	--
9	Phenolic, Asbestos	Raybestos RPD 150	--	--	--	--	--	①	--	--	--	--	--	--
10	Silicone Rubber	Dow Corning 93-046	--	--	--	--	D	--	--	--	D	--	F	F
11	Silicone Rubber	Gen Electric 2365-2-2083	--	--	--	--	D	①	--	--	--	--	F	F
12	Urethane	Magna Coatings Metalox	F											
13	Silicone Rubber	Dow Corning 93-046	--	--	--	--	D	--	--	--	--	--	F	F
14	Same as 7 with Phenolic and Fiberglass	Ferro Corp. WB 6502	--	--	--	--	--	①	D	--	--	--	--	--
15	Same as 7	Kirkhill 370-3-1051	--	--	--	F			--	--	--			

Legend: ① Test not performed due to lack of time.

D Coating experienced some degradation the long term effect of which was not determined.

F Coating failed to pass test.

-- Coating completed test with no apparent effects.

Blank Coating not tested due to elimination from program because of failure in previous test.

Coating 5. This particular silicone experienced loss of bond on three corners of one specimen during the extreme temperature tests. The other three specimens maintained adherence throughout their respective test series which included the same extreme temperature tests. During the gasoline and hydraulic oil tests, this silicone experienced no change in hardness and approximately 50 percent of the swelling that the other three silicone coatings (10, 11 and 13) demonstrated.

Coating 5 was the only silicone tested during Phase I which was not punctured by the impacting ball portion of the mechanical shock test. In addition, Coating 5 required on the average 250 percent of the shear load to fail the coating test coupons as compared with the other silicone coatings. Higher puncture resistance and shear strength are characteristic of the higher durometer rubber.

Since this coating had a cure procedure which requires temperatures ranging between 250 and 300°F, it is doubtful whether it would be practical from a field repair standpoint.

Coating 6. This coating material proved to be moisture sensitive as evidenced by its complete loss of bond and melting during the humidity test. No further testing of this candidate was performed.

Coating 7. Following the humidity test it was noted that some softening of the polysulfide epoxy used as the adhesive for this coating was in evidence on one of the test specimens. Some loss of bond on the same specimen was also noted after the low temperature test. It is possible that an adhesive more suitable for the application of this coating material could overcome this problem.

Coating 8. Some lack of bond was noted at two corners of all specimens prior to the start of any testing. Because Coating 8 was in the form of a highly rigid tile, it was difficult to detect if the bond had been affected further at these two corners as a result of environmental tests.

After the shear check was performed, it became obvious that a considerable portion of Coating 8 was without adhesive. The adhesive used was the same as Coating 13, a silicone rubber. Due to the high viscosity of the rubber, it did not completely flow throughout the bond line prior to curing.

Coating 9. The comments concerning the behavior of Coating 8 also apply to Coating 9 except that Coating 9 was without adhesive at two corners of only one test specimen, prior to the start of the tests.

Coating 10. Some unbonding of the corners of all specimens of Coating 10 occurred after both the extreme high and low temperature tests, likely due in part to insufficient cure time prior to the start of testing. The manufacturer recommended a seven day cure, but because this long cure cycle

would impose a severe restriction on the field use of Coating 10, it was decided to reduce the cure time to 30 hours.

As a result of the dilute hydrochloric acid tests, the edges of Coating 10 became friable, indicating some degree of adverse reaction with the hydrochloric acid.

Both the gasoline and hydraulic oil immersion tests caused enough swelling and deterioration of Coating 10 to constitute a failure. This type of behavior is quite common for silicone rubber materials.

The ball impact portion of the mechanical shock test caused Coating 10 to puncture.

Coating 11. Some unbonding of corners was experienced by this coating both prior to and after the first environmental test, extreme high temperature test. Vendor recommended application procedures were followed while applying Coating 11; therefore it must be concluded that the procedure is inadequate.

Both the gasoline and hydraulic oil tests caused sufficient swelling to eliminate this material from further consideration.

The ball impact portion of the mechanical shock test caused Coating 11 to puncture.

Coating 12. Complete loss of bond plus considerable warping characterized the failure of this coating as a result of the extreme high temperature test. Coating 12 was subsequently rejected from further consideration.

Coating 13. Of all four silicone rubber coatings, this coating material was the most seriously affected by the gas and oil. This was evidenced by the greatest decrease in hardness and the longest burn period during the flame test, indicating more gas and oil absorption.

The ball impact portion of the mechanical shock test caused Coating 13 to puncture.

Coating 13 was the only coating which indicated any decrease in shear strength between the control and test specimens. The shearing load to failure for coupons taken from both specimens from Sets One and Two was on the average only 65 percent of the load required to fail the coupons cut from the control specimen.

Coating 14. As in the case of Coating 7, softening of the same polysulfide epoxy adhesive was noticed following the humidity test. However, no loss of bond was detectable throughout the remainder of the test program, and Coating 14 was deemed as successfully completing the environmental tests.

Coating 15. This coating material experienced some loss of bond on two corners of the specimens from Sets Four and One after the extreme temperature and hydrochloric acid test, respectively. The adhesive used on this coating was the same as that used on Coatings 7 and 14. During low temperature deflection, Coating 15 cracked and was rejected from further consideration.

5.1.2 OUTDOOR EXPOSURE TEST RESULTS

In order to obtain additional information concerning the resistance of the various candidate coatings to weathering, the specimens remaining in the program were placed outdoors here at Newport Reach. After 103 days of exposure each specimen was inspected visually. Coatings 7, 8, 9 and 14 had no observable degradation as a result of the outdoor exposure period.

5.2 PHASE II TEST RESULTS

Out of 19 candidate materials tested during Phase II, only six remained at the conclusion of the environmental test series, Coatings 16, 23, 27, 30, 31, and 33. Of these six successful candidates, only three were without evidence of degradation, Coatings 16, 31 and 33.

A summary of the laboratory environmental test results is presented in Table VI. Individual performance of each candidate material is discussed in the following paragraphs.

5.2.1 LABORATORY ENVIRONMENTAL TEST RESULTS

Coating 16. The only derogatory comment which can be made against this candidate concerns its inability to maintain adhesion to the sand placed on its pre-cured surface to increase its anti-skid properties. No specific test, however, was included to measure skid resistance; therefore, it has not been established whether the sand was necessary for minimum skid resistance.

Coating 17. This coating lost adhesion to its substrate during the humidity test and was eliminated from the program.

Coatings 18, 19 and 20. Complete loss of adhesion characterized the failure of these materials as a result of the extreme low temperature test. The identical reaction of these materials had been observed during Phase I, but it was thought that the addition of a more flexible primer might overcome the problem of excessive differential contraction, previously concluded as cause of bond loss.

Coating 21. Significant loss of cohesive strength characterized the failure of this coating as a result of the extreme high temperature test, evidenced by the excessive crumbling and shredding experienced during the heel test.

TABLE VI
SUMMARY OF PHASE II ENVIRONMENTAL TEST RESULTS

Coating Number	Type	Manufacturer and Designation	Heel	Temperature +190	Temperature -65	Temperature Shock	Deflect	Mechanical Shock	Air Blast	Test Results		Low Temperature	Dilute HCl	H ₂ SO ₄	Hydraulic Oil	Gas
										Humidity	Temperature					
16	Epoxy, Polyurethane	General Tire V 61	--	--	--	--	--	--	--	--	--	--	--	--	--	--
17	Epoxy, Silicone	Dyna-Therm 1-62-23	--	--	--	--	--	--	F	F	--	--	--	--	--	--
18	Epoxy *	Product Techniques PT2090-4764-27	--	--	F	--	--	--	--	--	--	--	--	--	--	--
19	Epoxy *	Dyna-Therm E310F	--	--	F	--	--	--	--	--	--	--	--	--	--	--
20	Epoxy *	Dyna-Therm E310F	--	--	F	--	--	--	--	--	--	--	--	--	--	--
21	Silicone Rubber	General Electric 239-127-795	D	F	--	--	--	--	--	--	--	--	--	--	--	--
22	Silicone Rubber**	General Electric 239-127-795	--	D	F	D	--	--	F	--	--	--	--	--	--	--
23	Silicone Rubber	Dow Corning 93-063	--	D	D	D	--	--	D	--	--	--	--	--	D	D
24	Silicone Rubber**	Dow Corning 93-063	D	F	--	--	--	--	--	--	--	--	--	--	--	--
25	Epoxy *	Dyna-Therm E310F	--	--	--	--	--	F	--	D	--	--	--	--	--	--
26	Epoxy *	Dyna-Therm E310F	--	--	F	--	--	--	--	F	--	--	--	--	--	--
27	Epoxy, urethane*	Aerofutronic 502-84-A	--	--	D	--	--	--	--	--	--	--	--	--	--	--
28	Epoxy, urethane*	Aerofutronic 502-84-B	--	--	F	--	--	--	--	--	--	--	--	--	--	--
29	Epoxy, urethane*	Aerofutronic 502-84-B	--	--	F	--	--	--	--	--	--	--	--	--	--	--
30	Epoxy, urethane*	Aerofutronic 502-85-D	--	--	D	--	--	--	--	--	--	--	--	--	--	--
31	Epoxy, nylon *	Aerofutronic 502-86-A	--	--	--	--	--	--	--	--	--	--	--	--	--	--
32	Epoxy, nylon *	Aerofutronic 502-86-B	--	--	F	--	--	--	--	--	--	--	--	--	--	--
33	Epoxy, urethane*	Aerofutronic 502-85-D	--	--	--	--	--	--	--	--	--	--	--	--	--	--
34	Epoxy, urethane*	Aerofutronic 502-85-D	--	--	F	--	--	--	--	--	--	--	--	--	--	--

See Table V for legend

* This coating had a special primer material to reduce differential expansion effects between it and the substrate.

** This coating had an external top coat applied after cure to reduce the effects of exposure to hydraulic oil and gasoline.

Coating 22. This candidate represented an unsuccessful attempt to overcoat Coating 21 with a highly gasoline/oil resistant covering. This covering was loosened during the extreme temperature tests and partially removed during the air blast test. Consequently, the specimen was rejected from further consideration.

Coating 23. As a result of the extreme high temperature tests, some embrittlement of this coating was noted. Following the extreme low temperature test the specimens assumed a weak, crumbly texture and it was possible to remove small pieces from the specimen edges. Slight losses of bond were evidenced at the corners of the specimen when exposed to 160°F during the mechanical shock test. Both the gasoline and hydraulic oil exposure caused an adverse reaction within the coating. Consequently, it is doubtful whether this candidate would be capable of resisting the environments imposed on the Chaparral Fire Unit.

Coating 24. Again, as in the case of Coating 21, Coating 24 was an unsuccessful attempt to apply a topcoat covering to a silicone rubber. Those portions of the topcoat which did adhere prior to the start of environmental testing lost adhesion during the extreme high temperature test and the specimens were eliminated from further consideration.

Coating 25. This coating lost some adhesion at the corners of all test specimens during the extreme low temperature test and was completely destroyed as a result of the mechanical shock test.

Coating 26. Three out of four specimens exhibited some loss of adhesion at several corners following the extreme low temperature test. The fourth specimen lost adhesion at all four corners as a result of the humidity test. No additional testing of this candidate was performed.

Coating 27. Other than one corner of one specimen becoming unbonded during the extreme low temperature test, no adverse reaction to any of the environmental tests was noted.

Coatings 28 and 29. Complete separation of both of these candidates was experienced during the extreme low temperature tests, resulting in their elimination from the program.

Coating 30. All four corners of one of the candidate specimens lost a slight amount of adhesion during the extreme low temperature test. No additional degradation was noted throughout the remainder of the tests.

Coating 31. This coating successfully completed all tests without any adverse effects.

Coating 32. Complete separation of this coating from its substrate was experienced during the extreme low temperature test, resulting in the elimination of the coating from further consideration.

Coating 33. As with Coating 31, this candidate successfully passed all environmental tests.

Coating 34. The behavior of this coating was identical to Coating 32.

5.2.2 OUTDOOR EXPOSURE TEST RESULTS

Following 35 days of exposure to the exterior environments at Newport Beach, no visible change had occurred in the candidates which had successfully completed the environmental test program (Coatings 16, 23, 27, 30, 31 and 33).

SECTION 6

CONCLUSIONS AND RECOMMENDATIONS

✓ The results of the environmental and design loads testing on 34 potential coating systems have indicated that seven of these materials can pass the Chaparral requirements. It should be noted that these seven coatings have yet to pass the jet erosion test. The successful coatings ~~were~~ included; follows:

	<u>Coating No.</u>	<u>Material Designation</u>	<u>Material Classification</u>	<u>Application Technique</u>
Phase I	7	General Tire V-52	Acrylonitrile-butadiene rubber, abestos and silica filled	Precured bonded sheet
	8	Fiberite Corp MX-2625	Phenolic, chopped silica fabric	Precured bonded sheet
	9	Raybestos-Manhattan RPD-150	Phenolic, abestos fiber	Precured bonded sheet
	14	Ferro Corp WB 6505	Acrylonitrile-butadiene rubber, chopped fiberglass	Precured bonded sheet
Phase II	16	General-Tire V-61	Epoxy-polysulfide	Trowelable
	31	Aeronutronic 522-86-A	Epoxy-nylon	Trowelable
	33	Aeronutronic 522-85-D	Epoxy-urethane	Trowelable

phenolics, butadiene acrylonitrile rubber, epoxy -

polysulfide, epoxy-nylon and epoxy-urethane formulations.

The three generic types of material represented by the successful candidates have demonstrated acceptable jet erosion characteristics from the results of the Mauler and Pershing Programs. Since the Mauler and Chaparral missile launch environments are reasonably similar, the test results shown in Table 1 will provide direction on the performance of these materials in a Chaparral launch environment. The prototype firings at NOTS on the Chaparral development program indicated that Product Techniques, PT 2090 (epoxy) would erode approximately 0.06 inch per firing. The Mauler results showed an average erosion depth of 0.045 inch per firing. For this reason, it is expected that the epoxy base materials from Phase II will demonstrate similar erosion characteristics.

The Pershing jet erosion test results shown in Table II are for a material exposure time of 1.2 seconds. These erosion results can provide a first order indication of the performance of these materials in a Chaparral launch environment by dividing the jet erosion depth values by a factor of ten. The material exposure time is about one-eighth second on the Chaparral system.

As a result of the experimental investigation on this program, it is apparent that high blast erosion resistance is not the only performance criterion which must be closely examined to select a protective coating for the Chaparral System. In view of the fact that it was possible to find three trowelable coatings that could pass the Chaparral environmental requirements, it is recommended that these three coatings be given primary consideration for application to the engineering model. Single and multiple sub-scale motor firing data should be obtained on these three coatings. The jet erosion data along with other performance parameters (i.e., potting time, curing time, application cost etc.), should be included in the evaluation to select one coating out of the three for application to Engineering Models 1 through 5.

It is recommended that future protective coating work be directed to the following areas:

- (1) Study field repairability techniques. In the field it would be desirable to repair a damaged area by troweling after cleaning.
- (2) Determine if the selected coating will adhere to a chem-film treated surface. All of the testing performed to-date concerned coatings applied to bare aluminum surfaces.

- (3) Study production application techniques. This study would involve the examination of trowel or spray techniques and their associated costs.
- (4) Investigate new coating materials which have the potential of providing significant reduction in coating weight and cost.

REFERENCES

1. Wade, J. R., and Lyons, C. E., Final Report on Finish and Coating Development for Mauler Weapon Pod, RL-TM-65-61 U. S. Army Missile Command, Redstone Arsenal, Alabama, July 1965.
2. Galmish, M., et al., Solid Propellant Rocket Exhaust Effects Study Phase I Report, NASA-CR-63-27, Martin Company, Denver, Colorado, July 1963.
3. Pershing Blast Deflector Materials Development Program, OR 3172, Martin Company, Marietta, Georgia, March 1963.
4. Quinville, J. A., Diffuser Type Jet Deflector for the Hawk Launcher, Bumblebee Aerodynamics Panel, Minutes of 39th Meeting held 30 September and 1 October 1958 at University of Texas; TG 14-36, Johns Hopkins University, APL, Silver Spring, Maryland.
5. Lays, E., Design Handbook for Protection of Launch Complexes from Solid Propellant Exhaust, CR-66-11, Martin Company, Denver, Colorado, March 1966.
6. Evans, R. L. and Sparks, O. L., Launch Deflector Design Criteria and Their Application to the Saturn C-1 Deflector, TND-1275, National Aeronautics and Space Administration, Washington, March 1963.
7. Design Criteria for Launchers and Deflectors, Vitro 2331-1-60, Vitro Engineering Company, Redstone Arsenal, Alabama, March 1960.
8. Ellis, H. B., et al., Design Information for Uncooled Flame Deflectors, Report 1286, Aerojet General Corporation, Azusa, California, July 1957.

REFERENCES (Continued)

9. Grinberg, I. M., Determination of Analytical Techniques Available to Determine Material Suitability for Turning of Hot Gases, NAVWEPS 8746, U. S. Naval Ordnance Test Station, China Lake, California, May 1965.
10. Viles, J. M., Ablation of Extreme Temperature Resisting Materials in Rocket Exhausts, Report S-81, Rohm and Haas Company, Redstone Arsenal, Huntsville, Alabama, November 1965.
11. Baker, R. E., Development of Aircraft Protective Coating Against Rocket Motor Exhausts, NAVWEPS 8479, U. S. Naval Ordnance Test Station, China Lake, California, June 1964.
12. Locke, S. R., et al., Development of Materials for Solid Propellant Rocket Blast Deflectors, Paper 579C, SAE, National Aerospace Engineering and Manufacturing Meeting held October 1962.
13. Batchelor, J. D., Ablative Plastics in Solid Propellant Motor Exhausts, TR 65-315, USAF Materials Laboratory, July 1965.
14. Kremith, R. D., Thermal Erosion of Ablative Materials, TDR-64-67, USAF Materials Laboratory, June 1964.
15. Ablative Plastics for Rocket Nozzles, TDR-63-56, USAF Materials Laboratory, January 1964.
16. Thermal Erosion of Ablative Materials, TDR-63-254, USAF Materials Laboratory, July 1963.
17. Robbins, D. L. and Epstein, G., Thermal Erosion of Ablative Materials, TR 60-193, USAF Materials Laboratory, July 1961.
18. Auger, W. L., et al., Critical Literature Survey of Missile Blast Resistant Materials, Report 6-1, Department of Ceramic Engineering, University of Illinois, August 1965.
19. Bryan, T. G., et al., Chaparral Weapon System Environmental Criteria, AS109606B, Aeronutronic Division, Philco Corporation, April 1966.
20. Mar, P., Integration/Support Structure Design Criteria, AS109574A, Aeronutronic Division, Philco Corporation, April 1966.
21. Bryan, T. G., Chaparral Weapon System Vehicle/Pallet Interface Specification, AS109544B, Aeronutronic Division, Philco Corporation, July 1966.

APPENDIX A

TEST PROCEDURES

This appendix serves to define what each individual environmental test was designed to accomplish as well as the parameters which were controlled during each test.

A.1 PHASE I TEST PROCEDURES

The test title and purpose are stated first followed by a chronological listing of the detailed test steps.

A.1.1 EXTREME TEMPERATURE TEST

The purpose of this test is to assess what effect extreme high and low temperatures will have on the integrity and adhesive strength of each coating. A secondary purpose of the extreme high temperature test is to accelerate aging phenomena within the coating.

- (1) See note 1 prior to starting test.
- (2) Place all specimens in a temperature chamber adjusted to room temperature.
- (3) Adjust temperature control to $160 \pm 5^{\circ}\text{F}$ and soak all specimens for a minimum of 7 days.
- (4) Reduce temperature to room temperature.
- (5) Remove specimens 30 minutes after the chamber has attained room temperature and inspect visually.

- (6) Place all specimens in a chamber at room temperature and reduce to -65°F .
- (7) Soak at $-65 \pm 5^{\circ}\text{F}$ for 16 hours.
- (8) Increase chamber temperature to room temperature.
- (9) Remove specimens and inspect 30 minutes after the chamber has attained room temperature.

A.1.2 TEMPERATURE SHOCK TEST

The purpose of this test is to determine if the coating has sufficient adhesive strength to remain intact while experiencing rapid changes of environmental temperature.

- (1) See note 2.
- (2) Allow the specimens to stabilize at room temperature for a minimum of 3 hours.
- (3) Place the specimens in a chamber precooled to $-65 \pm 5^{\circ}\text{F}$ for 3 hours. Insure that the chamber temperature does not rise more than 7°F at any time.
- (4) Increase chamber temperature linearly with time to 120°F in 20 ± 5 minutes.
- (5) Maintain the chamber at $120 \pm 5^{\circ}\text{F}$ for 30 minutes.
- (6) Withdraw specimens and check visually.

A.1.3 DEFLECTION AT LOW TEMPERATURE TEST

The purpose of this test is to evaluate the ability of the coating to adhere to aluminum structure while undergoing large deflections at low temperature.

- (1) See note 1.
- (2) Place all specimens in a chamber at room temperature and adjust the temperature to -65°F .
- (3) Soak the specimens at $-65^{\circ}\text{F} \pm 5^{\circ}\text{F}$ for 30 minutes.
- (4) Each specimen will be removed from the chamber individually, deflected, and returned to the -65°F chamber within 5 minutes. Each specimen shall be cantilevered at one edge, and deflected with a uniformly distributed load at the

opposing edge such that the substrate is deflected in the direction of the coating. The magnitude of the load shall cause a maximum bending moment of 9600 ± 320 inch pound in that portion of the substrate containing the coating. After all the specimens have been tested once repeat this step.

- (5) Increase the chamber temperature to room temperature.
- (6) Soak the specimens at room temperature for 30 minutes.
- (7) Remove specimens and inspect visually.

A.1.4 LOW LEVEL MECHANICAL SHOCK TEST

The purpose of this test is to determine if the coating will survive mechanical shocks while at extreme low temperature.

- (1) See note 1.
- (2) Place all specimens in a chamber at room temperature and lower to -65°F .
- (3) Soak at $-65 \pm 5^{\circ}\text{F}$ for a minimum of 30 minutes.
- (4) Individually remove each specimen from the temperature chamber, drop freely on flat metal surface from a height of 12 inches with the coating side up, and return immediately to the temperature chamber. Do not keep any specimen outside of the temperature chamber more than 5 minutes during either steps 4 or 5.
- (5) Individually withdraw all the specimens from the chamber and drop a weight faced with a $5/8$ inch diameter ball from a height such that the product of the weight and height equals 2 foot pound. The weight shall be dropped against the coating 3 successive times and the specimen returned to the temperature chamber.
- (6) Raise chamber temperature to room temperature.
- (7) Allow the specimens to soak for 30 minutes at room temperature.
- (8) Inspect all specimens.

A.1.5 VIBRATION TEST AT LOW TEMPERATURE

The objective of this test is to determine whether the coating adhesion is affected by vibrational fatigue.

- (1) See note 1.
- (2) Place all specimens in a chamber at room temperature and adjust to -65°F .
- (3) Thirty minutes after the chamber has reached -65°F , remove each specimen and mount within the $-65 \pm 5^{\circ}\text{F}$ environmental chamber surrounding the vibration table.
- (4) Sinusoidal vibration of each specimen shall proceed for one hour as follows:

<u>Frequency Interval, Hz</u>	<u>Time Duration Min.</u>	<u>Sweep Rate Octaves/Min.</u>	<u>Level of Vibration</u>
130-320	0.4	7	.01 in. double amp
320-450	7.7	1	.01 in. double amp
450-575	22.3	1	90 G
575-700	18.9	1	90 G
700-830	8.6	1	90 G
830-950	1.7	1	90 G
950-1300	0.4	1	90 G

During vibration, each substrate is to be simply supported on two opposing edges and clamped on the remaining two edges.

- (5) After all specimens have been vibrated, raise the chamber temperature to room temperature and remove the specimens for a visual inspection.

A.1.6 HIGH LEVEL MECHANICAL SHOCK TEST

The purpose of this test is to determine if the candidate coating will be able to survive shocks in a low temperature environment.

- (1) See note 1.
- (2) Place all specimens in a chamber at room temperature and lower the air temperature to -65°F .
- (3) Soak at $-65 \pm 5^{\circ}\text{F}$ for a minimum of 30 minutes.
- (4) Perform steps 4 and 5 in test A.1.4 using 20 inch and 5 foot pound, respectively.
- (5) Raise chamber temperature to room temperature.
- (6) Allow the specimens to soak 30 minutes at room temperature.
- (7) Inspect all specimens.

A.1.7 SHEAR TEST

The purpose of this test is to determine the relative degradation of coating bond shear strength due to the environmental tests previously run.

- (1) Cut up each specimen into one-inch square coupons.
- (2) Inspect the remaining portions of each specimen along with coupons and note any loss of bond between coating and substrate.
- (3) Place all coupons in a vented oven at room temperature and adjust the temperature to 150°F .
- (4) Maintain the oven at $150 \pm 10^{\circ}\text{F}$ for a minimum of 4 hours.
- (5) Reduce the temperature to room temperature and remove the coupons after 30 minutes.
- (6) Individually load each coupon in a shear fixture such that the load is applied to one edge of the coating only.

- (7) Load at a rate of 1/2 in./min ± 10 percent until the shear head travels at least 1/4 inch from the point of contact.

- (8) Inspect the fracture.

A.1.8 100 PERCENT RELATIVE HUMIDITY TEST

The object of this test is to determine what effect extended periods of rain or high humidity have on the specimen coatings.

- (1) Remove .01 inch of coating from all the specimens using sandpaper or a wire brush.
- (2) Record the thickness and take a Barcol or shore hardness measurement at the center of the coating.
- (3) Place all the specimens in a chamber maintained at $120 \pm 5^\circ\text{F}$ with a relative humidity of 95 ± 5 percent for 6 days.
- (4) Measure the thickness and take Barcol or shore hardness measurement at the same location as in step 2.
- (5) Inspect each specimen for loss of bond or other degrading effects and immediately place back into the humidity chamber.
- (6) Maintain the specimens at $120 \pm 5^\circ\text{F}$ and 95 ± 5 percent humidity until the start of test A.1.9.

A.1.9 LOW TEMPERATURE TEST

The purpose of this test is to determine the effect caused by freezing any moisture contained within the coating on the coating itself or its bond with the substrate.

- (1) See note 1.
- (2) The specimens from test A.1.8 will have their thickness recorded and then left in the previous test chamber or placed in a chamber at room temperature and the temperature reduced to 0°F .
- (3) Maintain the chamber at $0 \pm 5^\circ\text{F}$ for 30 minutes.

- (4) Increase the temperature to room temperature.
- (5) Withdraw specimens and inspect.

A.1.10 DILUTE HYDROCHLORIC ACID TEST

The purpose of this test is to determine the ability of the coating to withstand exposure to dilute hydrochloric acid generated from a mixture of missile exhaust products and atmospheric moisture.

- (1) All specimens will be placed in a vented oven at room temperature and the temperature will be raised to 150°F.
- (2) Maintain the oven at 150 ±10°F for a minimum of 4 hours.
- (3) Reduce the temperature to room temperature and withdraw the specimens.
- (4) Measure thickness at the center of each specimen, and hardness at all 4 corners using a Barcol or shore hardness test.
- (5) Prepare a one percent by volume solution of hydrochloric acid.
- (6) Totally immerse the specimens in the dilute acid for 16 hours.
- (7) Remove the specimens, wipe them off, and allow them to air dry until dry to the touch.
- (8) Take thickness measurements at the center of each specimen, and Barcol or shore hardness measurements at all four corners.

A.1.11 SULFURIC ACID TEST

The object of this test is to determine the effect of accidental spillage of battery acid on the coatings.

- (1) Place the specimens in a vented oven at room temperature and raise the temperature to 150°F.
- (2) Maintain the oven at 150 ±10°F for a minimum of 4 hours.

- (3) Reduce the temperature to room temperature and withdraw the specimens.
- (4) Place the specimens flat on a table and meter ten drops of concentrated, room temperature sulfuric acid, having a specific gravity of $1.280 \pm .005$, onto each coating at one corner. The acid should be placed so that about half of it runs over the edge into the corner between the coating and the aluminum substrate.
- (5) Allow the acid to stand for 3 hours.
- (6) Wash the acid off with tap water and dry the specimens in air until they feel dry to the touch.
- (7) Measure the thickness at the center of the coating and the Barcol or shore hardness at all four corners of each coating.
- (8) Inspect the specimens visually.

A.1.12 MIL-H-5606 OIL TEST

The purpose of this test is to determine the effects of accidental spillage of hydraulic oil on the coating.

- (1) Place the specimens vertically on edge in a pan so that the impression stamp is on the uppermost edge.
- (2) Fill the pan with MIL-H-5606 hydraulic oil to a point where half the coating is immersed and soak for 16 hours.
- (3) Withdraw the specimens and wipe off excess oil.
- (4) Measure the thickness at the center (in an area which was immersed) and the Barcol or shore hardness at the two corners which were immersed.
- (5) Individually expose a single spot on the side of each coating which was immersed, to the tip of the inner blue flame of a single orifice bunsen burner for 5 seconds.

- (6) Allow the specimens to burn freely. If still burning after 20 seconds extinguish by blowing or washing with water.
- (7) On the specimens which burn freely for more than 5 seconds, remove the charred portion by scraping or brushing and measure the thickness at the bottom of the resulting crater.

A.1.13 FUEL TEST

The purpose of this test is to determine the effects of accidental spillage of gasoline on the coating.

- (1) Place the specimens vertically on edge in a pan so that the impression stamp is on the lowermost edge.
- (2) Fill the pan with room temperature gasoline of 90 octane or less such that half the coating is immersed, and soak for 3 hours.
- (3) Withdraw the specimens and dry them in a forced air hood for 30 minutes.
- (4) Measure the specimen thickness at the center where immersion occurred. Also measure Barcol or shore hardness at the two immersed corners.
- (5) Expose a single spot of the immersed edge to the tip of the inner blue flame of a single orifice bunsen burner for 5 seconds.
- (6) Allow the coating to burn freely for up to 20 seconds, then extinguish by blowing or washing in water.
- (7) On the specimens which burn freely for more than 5 seconds, remove the charred portion by scraping or brushing and measure the thickness at the bottom of the resulting crater.

A.1.14 OUTDOOR EXPOSURE TEST

The purpose of this test is to assess the effect of the combination of natural environments on the coatings.

- (1) Place the specimens outdoors for the duration of the tests.
- (2) Remove and inspect as required.

A.2 PHASE II TEST PROCEDURES

Besides the addition and deletion of tests as was stated in Section 4.2, certain changes within each test carried over from Phase I were made in order to make the tests more realistic. These changes along with their specific reasons are stated below.

A.2.1 EXTREME TEMPERATURE TEST

The high temperature extreme was raised from 160 to 190°F to account for the fact that MPU cooling air will be contributing to the high operating temperature of the MPU compartment door. The minimum low temperature extreme exposure time was reduced from 16 to 12 hours as the last 4 hours were considered unnecessary.

A.2.2 HEEL TEST

After the first 2 days of exposure to the extreme high temperature, each specimen from set I was removed from the temperature chamber and the heel test performed. This consisted of placing a rubber heel, size 11 to 12, in contact with the coating but overlapping it by 1 1/2 inches. The heel was then forced down on the coating with a load of 200 ±10 pounds, rotated 45 ±10 degrees to the right, 90 degrees ±10 degrees to the left, 45 ±10 degrees to the right and then withdrawn.

A.2.3 MECHANICAL SHOCK TEST

During both the high and low level shock tests, the free drop and ball impact portions are also performed at +160°F. This change was made because low hardness elastomerics are more susceptible to penetration at high temperature.

A.2.4 100 PERCENT RELATIVE HUMIDITY TEST

The time duration within the relative humidity chamber was reduced from six to three days as the last three days were considered unnecessary.

A.2.5 MIL-H-5606 OIL TEST

The 16 hour exposure period was replaced by a 1 hour period because indications of coating degradation were obvious after the first hour of exposure.

A.2.6 FUEL TEST

The three hour exposure period was replaced by 5 cycles of a 5 minute immersion and 30 minute drying period in order to more closely approach a life test.

A.2.7 AIR BLAST TEST

Attach a fixture to the edge of the specimen which will allow 375 ± 20 psig air pressure to exist over a 2 inch length of the edge of the coating. No mechanical contact is to be made between the fixture and coating.

- NOTES: (1) During all periods that the air temperature within the chamber containing the test specimens is changing, the rate of change shall not exceed 2°F per minute.
- (2) When the specimens are placed within the temperature chamber for the temperature shock test, all of their aluminum surfaces shall be insulated with the minimum equivalent of $1/8$ in. thick plywood.

APPENDIX B

TEST SPECIMEN PREPARATION

The purpose of this appendix is to explain the methods used in fabricating the test specimens. Comments concerning the ease of handling and work life of the coatings are also included.

B.1 PHASE I SPECIMEN PREPARATION

All Phase I coatings were mounted on 9.5 inch x 9.5 inch x 0.5 inch plates of 6061-T6 bare aluminum alloy. The coatings themselves were applied approximately 0.19 inch thick and centrally located on one side of the aluminum substrate within a 5.5 inch x 5.5 inch area.

Basically 3 different methods were used to attach the coatings depending on their nature. The coatings with high viscosity were troweled on with the aid of a template while those with low viscosity were cast in place also with the help of a template. The third technique concerned the precured materials which were bonded in place using various adhesive systems. All aluminum and precured coating bonding surfaces were first sanded with 180-200 mesh silicon carbide grit paper and then cleaned with methyl ethyl ketone and allowed to air dry for 20 minutes minimum. The details peculiar to each coating application are listed in tables B.1 through B.4.

B.2 PHASE II SPECIMEN PREPARATION

The substrate alloy used for Phase II specimens was changed to 7075-T6 bare aluminum, the same material used to fabricate Chaparral deck plates.

Because all of the coatings selected for evaluation during Phase II were capable of being troweled, the only bonding procedure differences between

the various coatings concerned the primer systems used. Substrate cleaning was identical to the procedure described in Phase I.

Primer materials, when employed, were brush coated on 5 to 10 mils thick and allowed to air dry for a minimum of one hour. After the coatings were troweled in place, each specimen was allowed to cure at room temperature for a minimum of 6 days before any testing commenced.

The top coats used for the silicone rubber coatings were applied by brush 30 to 60 mils thick as soon as the silicone undercoating was tack free.

Table B.5 contains the detailed specimen preparation procedures for Phase II.

Eight of the candidate coating primer systems evaluated during this phase were formulated by Aeronutronic. The constituents used for each are listed in Table B.6.

TABLE B.1
PHASE I APPLICATION PROCEDURES - TROWELED COATINGS

<u>Coating Number</u>	<u>Aluminum Primer</u>	<u>Estimated Work Life (hours)</u>	<u>Recommended Cure</u>	<u>Comments</u>
1 (Incoat 13 H-26)	None	0.5	7 days at 80°F for full cure. Had 4-day cure before 160°F exposure.	1. Good troweling characteristics. 2. Needs a little more body.
2 (Product Techniques PT 2090-46C4-27)	None	3	7 days at 80°F for full cure. Had 4-day cure before 160°F exposure.	1. Good body and texture. 2. Irritating odor.
3 (Narmco 4056)	None	0.5	2-4 days at 75 ± 5°F for full cure. Had 4-day cure.	1. Difficult to mix. 2. Difficult to apply
4 (Dyna-Therm E 310F)	None	1	8 hours at 77°F for full cure. Had 4-day cure.	1. No difficulties.
5 (G.E. TBS 758)	Brush coat GE-SS4155 Primer and Air Dry 60 minutes.	4	10 minutes at 300°F or 60 minutes at 250°F. Had 60 minutes at 250-300°F.	1. Requires very careful mixing to avoid soft spots. 2. Good pot life. 3. Easy to trowel.

TABLE B.1 (Continued)

<u>Coating Lumber</u>	<u>Aluminum Primer</u>	<u>Estimated Work Life (hours)</u>	<u>Recommended Cure</u>	<u>Comments</u>
10 (Dow Corning 93-019)	Brush coat DC-1200 primer and air dry 30 minutes.	2	7 days at 75 ±5°F for full cure. Had 3 days before 160°F exposure.	No difficulties
12 (Dow Corning 93-046)	Brush coat DC-1200 primer and air dry 30 minutes.	1	7 days at 75 ±5°F for full cure. Had 3 days before 160°F exposure.	No difficulties

TABLE B.2

PHASE I APPLICATION PROCEDURES - CASTABLE COATINGS

<u>Coating Number</u>	<u>Aluminum Primer</u>	<u>Recommended Cure</u>	<u>Comments</u>
12 (Metalox)	None	At 75 ± 5°F until tack free. Had 30 hours before 160°F exposure.	None

TABLE B.3

PHASE I APPLICATION PROCEDURES - BONDED PRECURED TILE COATINGS

<u>Coating Number</u>	<u>Tile Molding Procedure</u>	<u>Primer Preparation</u>	<u>Adhesive Mixing and Application</u>	<u>Recommended Cure</u>
7 (General Tire V 52)	Mold two 0.1 inch layers at 500 psi and $205 \pm 10^{\circ}\text{F}$ for 1.5 hour; $308 \pm 5^{\circ}\text{F}$ for 2.0 hours. Remove slab from mold to reduce its temperature to below 225°F within 60 minutes. Density 1.32 gm/cc.	None	1.0 parts by weight of V-61-1 to 1.0 parts by weight of V-61-2 Has 25 minutes pot life. Trowel 5-10 mils of adhesive onto both surfaces. Assemble and press firmly together.	2 days at $75 \pm 5^{\circ}\text{F}$ for full cure. Had 3 days before 160°F exposure.
8 (Fiberite MX 2625)	Preheat material 10 minutes at 200°F . Preheat mold to 300°F . Mold at 3000 psi and 300°F for 45 minutes. Density 1.75 gm/cc.	Brush coat DC-1200 primer to all surfaces and air dry 30 minutes.	93-04(-10 parts by weight catalyst to 100 parts by weight base. Mix 5 minutes by hand Pot life is 60 minutes. Trowel 5-10 mils of adhesive onto both surfaces. Assemble and press firmly together.	7 days at $75 \pm 5^{\circ}\text{F}$ for full cure. Had 3 days before 160°F exposure.

TABLE B.3 (Continued)

<u>Coating Number</u>	<u>Tile Molding Procedure</u>	<u>Primer Preparation</u>	<u>Adhesive Mixing and Application</u>	<u>Recommended Cure</u>
9 (Raybestos RPD 150)	Preheat material 10 minutes at 200°F. Preheat mold to 300°F. Mold at 3000 psi and 300°F for 45 minutes. Density 1.73 gm/cc.	Brush coat DC-1200 primer to all surfaces and air dry 30 minutes.	93-046-10 parts by weight catalyst to 100 parts by weight base. Mix 5 minutes by hand. Pot life is 60 minutes. Trowel 5-10 miles of adhesive onto both surfaces. Assemble and press firmly together.	7 days at 75 ± 5°F for full cure. Had 3 days exposure.
11 (GE2365-2-2083)	Freshen stock on rubber mill. Mold at 500 psi and 340 ± 10°F for 1/2 hour. Cool slab below 200°F before releasing pressure. Post cure 350°F for 60 minutes and 400°F for 120 minutes. Density 1.24 gm/cc.	Brush coat GE-SS-4155 primer on aluminum, only, and air dry for 60 minutes.	RTV630-10 parts by weight. A to 1 part by weight B. Working time is 120 minutes. Trowel 5-10 mills of adhesive onto both surfaces. Assemble and press firmly together.	48 hours at 75 ± 5°F for full cure. Had 3 days before 160°F exposure.

TABLE B.3 (Continued)

<u>Coating Number</u>	<u>Tile Molding Procedure</u>	<u>Primer Preparation</u>	<u>Adhesive Mixing and Application</u>	<u>Recommended Cure</u>
14 (Ferro Corp. WB 6505)	Mold at 225°F and 30 psi for 15 minutes; 275°F and 30 psi for 15 minutes; 325°F and 100 psi for 60 minutes. Density 1.58 gm/cc.	None	1.0 parts by weight of V-61-1 to 1.0 parts by weight of V-61-2. Had 3 days Has 25 minutes before 160°F pot life. Trowel exposure 5-10 mils of adhesive onto both surfaces. Assemble and press firmly together.	2 days at 75 ± 5°F for full cure. Had 3 days before 160°F exposure.
15 (Kirkhill 370-C-1051)	Mold at 500 psi and 205 ± 10°F for 1.5 hour; 308 ± 5°F for 2.0 hour. Remove slab from mold to reduce its temperature to below 225°F within 60 minutes. Density 1.21 gm/cc.	None	1.0 parts by weight of V-61-1 to 1.0 parts by weight of V-61-2. Had 3 days Has 25 minutes before 160°F pot life. Trowel exposure 5-10 mils of adhesive onto both surfaces. Assemble and press firmly together.	2 days at 75 ± 5°F for full cure. Had 3 days before 160°F exposure.

TABLE B.4

PHASE I APPLICATION PROCEDURES - BONDED LAMINATE/COATINGS

<u>Coating Number</u>	<u>Aluminum Primer</u>	<u>Laminating Procedure</u>	<u>Recommended Cure</u>	<u>Comments</u>
6 (Dyna-Therm D-65)	None	Apply 10-mil coat of D-65 liquid to alu- minum. Position first D-65, 32-mil sheet ply. Lay up 4 additional 32-mil, D-65 sheet plies with 5-mil coat- ing of D-65 liquid between each ply.	1 day required for full cure. Had 4-day cure before 160°F exposure.	1. Very easy to apply. 2. Would be easy to apply under difficult conditions.

TABLE B.5

PHASE II APPLICATION PROCEDURE INFORMATION

<u>Coating Number</u>	<u>Aluminum Primer</u>	<u>Estimated Work Life (hours)</u>	<u>Comments</u>
16 (Gen. Tire V 61)	None	0.3	Obnoxious odor; low flow
17 (Dyna-Therm X 62-23)	None	0.5	Good handling, med flow
18 (Product Techniques PT 2090-4764-27)	Narmco 7344/7119	3	Good handling, med flow
19 (Dyna-Therm E-310F)	Narmco 3170/7133	1	Difficult to blend; very viscous
20 (Dyna-Therm E-310F)	Narmco 7344/7119	1	Difficult to blend; very viscous
21 (G.E. 239-127-795)	G.E. SS-4145	0.5	High flow
22 (Same as 21 with G.E. Traffic Coat)	G.E. SS-4145	0.5	High flow
23 (Dow Corning 93-063)	Dow Corning 1200	0.3	High flow
24 (Same as 23 with D.C. 94-024 Top Coat)	Dow Corning 1200	0.3	High flow
25 (Dyna-Therm E-310F)	B.F. Goodrich 934 with Narmco 3170/7133 over 934	1	Difficult to blend; very viscous

TABLE B.5 (Continued)

<u>Coating Number</u>	<u>Aluminum Primer</u>	<u>Estimated Work Life (hours)</u>	<u>Comments</u>
26 (Same as 25)	B.F.Goodrich 934 with Narmco 7344/7119 over 934	1	Difficult to blend; very viscous
27 (Aeronutronic 522-84-A)	B.F.Goodrich 934	5	High flow; cures to handling condition in 3 days
28 (Aeronutronic 522-84-B)	B.F.Goodrich 934	5	Same as above
29 (Same as 28)	None	5	Same as above
30 (Aeronutronic 522-85-D)	B.F.Goodrich 934	5	No flow, cures to handling condition in 3 days
31 (Aeronutronic 522-86-A)	B.F.Goodrich 934	1.5	Low flow; cures to handling condition in 1 day
32 (Aeronutronic 522-86-B)	B.F.Goodrich 934	1.5	No flow; cures to handling condition in 1 day
33 (Aeronutronic 522-85-D)	3 M Company EC-2126	5	No flow; cures to handling condition in 3 days
34 (Aeronutronic 522-85-D)	Dow Corning 4014	5	Same as above

TABLE B.6

PHASE II AERONUTRONIC COATING FORMULATIONS

Filler Loading Parts per Hundred Parts Resin by Weight (PHR)

Coating Number	Base Resin System* (Narmco)	Superpax A (National Lead) Zr. Silicate	Antimony Trioxide	Arochar 1248 (Monsanto)	Phenolic Powder (200 mesh)	Asbestos	
						(Asbestos Corp.)	Floats Tam 390 (390 mesh silica) Sand (60 mesh)
27	7344/7119	50	25	10	0	15	0
28	7344/7119	25	25	10	25	30	0
29	7344/7119	25	25	10	25	30	0
30	7344/7119	0	15	30	0	35	150
31	3170/7133	30	0	60	0	60	150
32	3170/7133	20	0	30	30	36	80
33	7344/7119	0	15	30	0	35	150
34	7344/7119	0	15	30	0	35	150

*Narmco 7119 and 7133, both curing agents, are mixed 14 and 100 PHR, respectively

DISTRIBUTION

Headquarters
U. S. Army Missile Command
Building 4488, Room A-329
Redstone Arsenal, Alabama 35809

Attention: Mr. Marvin Snipes ANSMI-XGC

10 copies