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THERMAL CONTROL COATINGS FOR HIGH THERMAL CONDUCTIVITY (K) SUBSTRATES



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1.0 INTRODUCTION

The Air Force Wright Laboratory Materials Directorate awarded the program, "Thermal Control Coatings for High Thermal Conductivity (K) Substrates," contract number F33615-95-C-5028, to Lockheed Martin Vought Systems on September 10, 1995. The original program had a 40 month duration, ending in January, 1999. The program was modified on November 6, 1997 to include additional activities, which resulted in a 3-month extension to the duration. This made the program end April 30, 1999. The original contract monitor was Patrick Carlin, MLBT. Mr. Carlin was replaced by Dr. Jeff Sanders, as the concluding contract monitor. The purpose of the program was to investigate plasma sprayed thermal control coatings for space hardware and develop the coating production and application methods suitable for scale up to industry sources.

1.1 PROGRAM OBJECTIVES

The program had three primary objectives. The first objective was to develop reproducible processes to produce low solar absorptance ($\alpha_s \leq 0.15$) (end of life $\alpha_s \leq 0.20$) space stable ($\Delta \alpha_s$ (1000 ESH + 10¹⁵ electrons/cm²) ≤ 0.05), high hemispherical thermal emittance ($\epsilon_H \geq 0.80$), plasma sprayable thermal control coatings for high thermal conductivity substrates. The second objective was to demonstrate the suitability, cost effectiveness and reliability of using plasma sprayed thermal control coatings. The third objective was to investigate plasma sprayed thermal control coatings for space hardware and develop the coating production and application methods suitable for scale up to industry.

1.2 APPROACH

The technical approach to achieving the program objectives was organized into eight tasks. Task 1, "Transition Plan," presented the program plan which described the technical approach based upon proven thermal control coating process specification scale-up that resulted in the successful transition of plasma sprayed coatings for thermally advanced spacecraft structures. Task 2, "Material and Processes Optimization," screened coating materials and optimized applications which were economically and technically feasible for full scale manufacturing. Task 3, "Material and Characterization Qualification," characterized and qualified the selected coating systems from Task 2. Task 4, "Standard Operating Procedure," produced standard operating procedures (SOP)s for the qualified coating and the application. The SOP's were included as part of the material and application specifications. Task 5, "Materials Demonstration and Technology Transition," demonstrated uniform quality and properties of the coating produced to the approved SOP by a vendor and applied to the approved SOP by a vendor. Task 5 was modified to include fabrication and delivery of two C-C radiators for the Carbon-Carbon Space Radiator (CSRP) partnership. Task 6, "Presentations," provided view graphs and other presentation materials for periodic meetings. Task 7, "Management Briefing," provided for the semiannual meetings held alternately at the program monitor's facility and contractor's facility. Task 8, "Deliverables and Final Report," provided for delivering the space stability specimens, demonstration article and the final report to the contract monitor.

1.3 SUMMARY OF RESULTS

The program objectives of industry scale up of both the powder production and the plasma spraying were achieved and illustrated by procurement of powder and plasma spraying of the demonstration article to the applicable SOP. The Al2O3/ZrO2/Y2O3 powder production was found to be reproducible, repeatable, and economically feasible. The powder was successfully produced to the SOP by three vendors; Lockheed Martin Vought Systems (LMVS), Contract Materials Processing (CMP), and Praxair Speciality Powders-Seattle. Praxair Speciality Powders-Indianapolis also provided power produced to a similar SOP. The powder was also successfully plasma sprayed to the SOP by two vendors; LMVS and Praxair Thermal Systems. The space stability test results of the Al2O3/ZrO2/Y2O3 powders performed at

the Space Combined Effects Primary Test and Research (SCEPTRE) Facility at AFRL/ MLBT powders indicated similar performance of all of the powders. The beginning of life solar absorptance values made in situ were approximately: CMP 0.14, LMVS 0.16, Praxair-WA 0.19 and Praxair-Indianapolis 0.17. The end of life solar absorptance values made in situ at 1000ESH were: CMP 0.27, LMVS 0.27, Praxair-WA 0.28 and Praxair-Indianapolis 0.28. The end of life solar absorptance values made in situ at 2200 ESH were: CMP 0.30, LMVS 0.31, Praxair-WA 0.31, and Praxair-Indianapolis 0.30. The hemispherical emittance of the LMVS blended powder was 0.79. Samples from one source were measured and is typical for the powder composition. The ultra pure alumina hemispherical emittance was 0.80. An ultra pure alumina LMVS produced and plasma sprayed was also evaluated at the SCEPTRE facility. The beginning of life solar absorptance values made in situ were approximately 0.10 and the end of life values were 0.20 at 1.2 EUVS, 0.21at 2.2 EUVS, and 0.23 EUVS. The contract goal for solar absorptance was 0.15 beginning of life and 0.20 at 1000ESH. The plasma sprayed coating passed an acoustic qualification test conducted at 144 dB level for 23 minutes 50 seconds at the LMVS acoustics test facility.

The three C-C radiators were successfully built and delivered to NASA for the CSRP.

The overall objective of this task was coating material screening and application optimization in a manner that is feasible both economically and technically for full scale manufacturing.

2.1 SCREENING MATERIALS

The objective of this task included candidate plasma powder material selection, under coat material investigation, substrate specimen fabrication, coating trials, and screening test and evaluation.

2.1.1 Plasma Spray Powders

Candidate plasma spray powders were selected and procured or produced by LMVS. The candidate materials were: 1) baseline sol-gel derived oxide produced 86wt%Al2O3/13wt%ZrO2/1%wtY2O3 (ML-TCC-5), 2) ultra high purity Al2O3, 3) vendor produced 86wt%Al2O3/13wt%ZrO2/1%Y2O3, and 4) custom porcelain using zinc orthotitanate ZOT pigment in a ceria doped borosilicate glass matrix.

The baseline sol-gel derived oxide powder was furnished by the customer as GFE. This powder was produced on an earlier customer program and was chosen to be the primary material for scale up activities by the customer. One lot of material of various particle sizes was received and evaluated for material properties and plasma spraying applications. In order to make sufficient quanities to plasma spray, the powders were mixed together and spray dried to make a powder lot.

The high purity Al2O3 powder was obtained from three sources. One source was Alpha Aesar. The powder procured from Alpha Aesar was 99.99% pure with an approximate particle size of 1μ m. A second source was Chemat Technology. The powder procured from Chemat Technology was a sol gel derived alumina at 99.98% purity at a particle size of 35-100 μ m. The powder produced by CHEMAT was

dropped from evaluation due to powder flowing characteristics resulting from particle size and shape. A third source of powder was Praxair-Indianapolis. This powder was in plasma sprayable condition and was used directly in the plasma spraying evaluation.

The ultra high purity Al2O3 powder from Alpha Aesar was an off-the-shelf item, used primarily as an economical material to develop the processing and plasma spraying parameters. The procured Al2O3 powder was spray dried to obtain plasma spraying compatible characteristics and produce the required particle size. Figure 1 shows a typical macrograph of the Al2O3 powder. A non ash producing binder was used and all of the surfactant additions were completely removed. This was determined by scanning electron microscope analysis and crystallographic analysis method. The crystallographic analysis for the powder is shown in Figure 2. The crystallographic analysis was performed on the coating after plasma spraying. Figure 3 shows the XPS scan after plasma spraying. After the processing parameters were developed for the Al2O3, the information was used to reproduce a powder similar to the baseline sol-gel derived oxide powder from the customer.



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Figure 1 Typical Macrograph of Alumina Powder



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Figure 3 Composition of Thermal Control Coating After Plasma Spraying

LMVS successfully produced a blended plasma spray powder similar to the baseline sol-gel produced powder by using the processing parameters developed for the ultra high purity Al2O3 powder. LMVS produced the blended oxide powder by using the ultra high purity Al2O3 dry powder as a primary constituate. The ZrO2 and Y2O3 materials were blended into the primary constituate to produce the desired chemical composition. The ZrO2 and the Y2O3 materials were procured as chemically pure sols. LMVS prepared SOPs and a specification for blended powder production. The specification and the SOPs were forwarded to vendors for review and comments. The blended powders were procured from vendors in the Task 3 activities.

The custom porcelain glaze was produced by LMVS using Pilkington CMX borosilicate glass ribbon and zinc orthotitanate YB-71 pigment. The glass ribbon was received in sheet form and was processed into a frit. The processing included mechanical break up, rotary milling and mixing with the pigment and spray dried into a plasma powder.

2.1.2 Plasma Spraying

The plasma spraying parameters for the derived oxide powders were developed using the ultra pure alumina. This approach was used to preserve the existing GFE powders and to use a cost effective, similar material to identify the performance driving variables. The parameters were tested and transferred to the blended powder.

A similar approach was used to develop plasma spray parameters for the custom porcelain. The initial parameters were developed using a commercially available pyrex. The parameters were then transferred to the custom porcelain.

2.1.2.1 Taguchi Design of Experiment for Alumina Based Powders

A Taguchi Design of Experiment was used to optimize the plasma applications. The Taguchi method optimizes the multiple variable systems by using orthogonal arrays to minimize the number of experiments required to define the optimum parameter levels. The Taguchi experiments also developed robust processes which identified performance driving parameters and minimized environment caused variables. The initial Taguchi matrix (Table 1) consisted of a set of 12 plasma spray trials for 10 factors at 2 levels for each factor (Table 2). The gases pressure, gun speed, powder feed, power settings and powder gas pressure are equipment settings. The equipment used for all LMVS plasma spraying was a Plasmadyne SG-100 gun, rotary hopper feed system and 80 kW plasma system.

The Chemet material was dropped from the evaluation after failed attempts to spray dry the powder into a plasma sprayable material. The Taguchi experiment was adjusted to use only powder from Alpha Aesar (Table 3).

Table 1 Initial Plasma Coating Taguchi Matrix (L ₁₂)											
Run	Gun Angie	Supplier	Thickness mm (in)	Distance cm (in)	He Gas	Gun Speed	Argon Gas	Powder Feed (rpm)	Power (amp)	Powder Gas	
. 1	45	Alpha	0.38 (0.015)	15.25 (6)	35	4	50	23	700	15	
2	45	Alpha	0.38 (0.015)	15:25 (6)	35	6	65	2.8	800	25	
3	45	Alpha	0.43 (0.017)	20.32 (8)	50	4	50	2.3	800	25	
4	45	Chemet	0.38 (0.015)	20.32 (8)	50	4	65	2.8	700	15	
5	45	Chemet	0.43 (0.017)	15.25 (6)	50	6	50	2.8	700	25	
6	45	Chernet	0.43 (0.017)	20.32 (8)	35	···· 6	65	2.3	800	15	
7	90	Alpha	0.43 (0.017)	20.32 (8)	35	4	65	2.8	700	25	
8	90	Alpha	0.43 (0.017)	15.25 (6)	50	6	65	2.3	700	15	
9	90	Alpha	0.38 (0.015)	20.32 (8)	50	6	50	2.8	800	15	
. 10	90	Chemet	0.43 (0.017)	15.25 (6)	35	4		2.8	800	15	
11	90	Chemet	0.38 (0.015)	20.32 (8)	35	6	50	2.3	700	25	
12	90	Chemet	0.38 (0.015)	15.25 (6)	50	4	65	2.3	800	25	

The deletion of the supplier variable changed the factors matrix as shown in Table 4.

The Alpha material was procured from Alpha AESAR.

Para-meter	Factor	Units	Level 1	Level 2
A	Gun Angle	Degree	45	90
В	Supplier		Alpha	Chemet
C	Thickness	mm (in)	0.38 (0.015)	0.43 (0.017)
D	Distance	cm (in)	15.25 (6)	20.32 (8)
E	He Gas	psi	35	50
. F	Gun Speed	in/sec	4	6
G	Argon Gas	psi	50	65
н	Powder Feed	rpm	2.3	2.8
ſ	Power	amp	700	800
J	Powder Gas	psi	15	25

Table 2 Initial Plasma Coating TaguchiFactors Matrix

Table 3 Initial Plasma Coating Taguchi Matrix (L_{12})

	Gun	Sumplion	Thickness	Distance	He	Gun Speed	Argon	Powder Feed (rpm)	Power (amp)	Powder Gas
1 1	45	Aloha	0.38 (0.015)	15.25 (6)	35	4	50	2.3	700	15
2	45	Alpha	0.38 (0.015)	15.25 (6)	35	6	65	2.8	800	25
3	45	Alpha	0.43 (0.017)	20.32 (8)	50	4	50	2.3	800	25
4	45	Alpha	0.38 (0.015)	20.32 (8)	50	4	65	2.8	700	15
5	45	Alpha	0.43 (0.017)	15.25 (6)	50	6	50	2.8	700	25
6	45	Alpha	0.43 (0.017)	20.32 (8)	35	6	65	2.3	800	15
7	90	Alpha	0.43 (0.017)	20.32 (8)	35	4	65	2.8	700	25
8	90	Alpha	0.43 (0.017)	15.25 (6)	50	6	65	2.3	700	15
9	90	Alpha	0.38 (0.015)	20.32 (8)	50	6	50	2.8	800	15
10	90	Alpha	0.43 (0.017)	15.25 (6)	35	4	50	2.8	800	15
11	90	Alpha	0.38 (0.015)	20.32 (8)	35	6	50	2.3	700	25
12	90	Alpha	0.38 (0.015)	15.25 (6)	50	4	65	2.3	800	25

The Alpha material was procured from Alpha Aesar

Table 4 Plasma Coating TaguchiFactors Matrix

Para-meter	Factor	Units	Level 1	Level 2
A	Gun Angle	Degree	45	90
c	Thickness	mm (in)	0,38 (0.015)	0.43 (0.017)
D	Distance	cm (in)	15.25 (6)	20.32 (8)
E	He Gas	psi	35	50
F	Gun Speed	in/sec	4	6
G	Argon Gas	psi	50	65
н	Powder Feed	rpm	2.3	2.8
	Power	amp	700	800
J	Powder Gas	psi	15	25

The plasma spraying experiments were conducted using 15.24 cm X 15.24 cm carbon-carbon substrates.

The plasma spraying parameters were customized to optimize the solar absorptance, emittance, number of coats, and density. The initial alumina plasma spray Taguchi matrix results are shown in Table 5. The optimum solar absorptance and normal emittance was measured to be 0.116 and 0.829 in the experiment.

L ₁₂ Array															
	A	В	¢	D	E	F	G	Н	1	J	ĸ	Results	Results	Results	Results
Run	1	2	3	4	5	6	7	8	9	10	11	ε _n	a 🖉	No. of Coats	Density
ેલાં	1	1	1	1	1	T	4	1	1	1	1	0.841	0.152	14	2.878
2	11	1	1	1	1	2	2	2	2	2	2	0.842	0.160	17	2.979
3	1	1	2	2	2	1 3	1	1	2	2	2	0.826	0.136	22	2.844
4	1	2	1	2	2	1	2	2	1	1	2	0.836	0.125	-16	2.646
5	1	2	2	1	2	2	1	2	1	2	1	0.834	0.152	27	2.889
6	1	2	2	2	1	2	2	1	2	1	1	0.829	0.116	42	2.711
7	2	13	2	2	1		2	2	1	2	1	0.830	0.119	18	2.610
8	2	1	2	1	2	2	2	1)	1	1	2	0.839	0.153	26	2.813
9	2	1	1	2	2	2		2	2	1	1	0.821	0.142	19	2.777
10	2	2	2	1	1	1	S	2	2	1	2	0.840	0,171	15	2.956
11	2	2	1	2		2	J	્યું	1 0	2	2	0.828	0.129	26	2:572
12	2	2	1	1	2	1	2	1	2	2	1	0.848	0.145	13	2.899

Table 5 Alumina Plasma Spray Taguchi Matrix Results

Notes:

The array is 11 factors of 2 levels each.

C1 = Sum of results for all experiments containing C1 = Sum of run #1 + 2 + 4 + 9 + 11 + 12 for the number coats that = 105. C2 = Sum of results for all experiments containing C2 = Sum of run #3 + 5 + 6 + 7 + 8 + 10 for the number coats that = 150.

However, the economic considerations of the number of required coats to obtain these optical properties indicate that similar results are obtainable with fewer coats. These parameters were used as the primary factors in the second Taguchi experiment.

The test results were organized into different formats which were used to identify the main factors influencing a particular property. The magnitude of the factor line indicates the strength of that variable on that property. Figure 4 shows that the main factors for minimum solar absorptance are a distance from the plasma spray gun to the work piece, argon gas pressure, powder feed rate and power. Figure 5 shows





that the main factors for normal emittance to be a distance from the plasma spray gun to the work piece, gun speed and argon gas pressure. Figure 6 shows that the main factors for density to be a distance from the plasma spray gun, power, gun angle, and argon gas pressure. Figures 7 through 18 show the solar absorptance measurements.



Figure 6 Factor Effect on Density



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Figure 7 Solar Absorptance Measurements of Panel 1



Figure 8 Solar Absorptance Measurements of Panel 2



Figure 9 Solar Absorptance Measurements of Panel 3



Figure 10 Solar Absorptance Measurements of Panel 4



Figure 11 Solar Absorptance Measurements of Panel 5





Figure 12 Solar Absorptance Measurements of Panel 6







Figure 14 Solar Absorptance Measurements of Panel 8



Figure 15 Solar Absorptance Measurements of Panel 9



Figure 16 Solar Absorptance Measurements of Panel 10



Figure 17 Solar Absorptance Measurements of Panel 11



1/6/99 - PPS-903213-015.PPT Figure 18 Solar Absorptance Measurements of Panel 12

The initial Taguchi experiments indicated that a solar absorptance of 0.120 at 0.38mm (.0015 in) is achievable and that a predication of a solar absorptance of .112 at a density of 2.625 g/cm³ (34% porosity) can be made. Figure 19 shows the solar absorptance plotted as a function of density.



Volumetric Density Effect on Absorptance

Figure 19 Solar Absorptance Versus Density

A second Taguchi experiment was performed to optimize the plasma spraying parameters developed in the initial Taguchi activities. The second Taguchi matrix consisted of a set of 4 plasma spray trials for 3 factors at 2 levels for each factor (Table 6). The factors optimized were power, distance from the plasma spray gun to the work piece and argon gas pressure.

Run	Power (watts)	Distance cm (in)	Argon Gas (psi)
1	700	20.32 (8)	65
2	700	22.86 (9)	80
3	650	20.32 (8)	80 -
.4	650	22.86 (9)	65

Table 6 Primary Plasma Spray Taguchi Matrix (L₄)

The second alumina plasma spray Taguchi matrix results are shown in Table 7. The solar absorptance and normal emittance values ranged from 0.112 to 0.119 and 0.824 to 0.832 in the experiment.

Run	A	В	C.	a	Number of	Coats	Panel Wt	Gain(g)	Thicknessm	m (in)	Density
1	1	1	1	0.119	19		21.0	369	0.37 (0.01	47)	2.4258
2		2	2	0.113	24		22.1	205	0.39 (0.01	51)	2.4832
3	2	1	2	0.116	22		23.0	580	0.39 (0.01	54)	2.5380
4	2	2	1	0.112	40		21.2	647	0.39 (0.01	55)	2.3255

Table 7 Alumina Plasma Spray Taguchi Matrix Results

Notes:

Factors are as follows: A=Power, B=Distance, C=Argon Gas pressure.

The factor effect of the settings optimized for the minimum number of coats indicated that main driver was the distance from the plasma gun to the work piece. However, the difference in the magnitude of the power, argon gas pressure, and the distance was minor. The main driver for the settings optimized for the minimum coating density was the argon gas pressure. The distance was medium effect and the power settings were a minor effect. The main driver for the settings optimized for the solar absorptance was the distance. The power and argon gas pressure were minor effects. Figures 20-22 show the relative strength of the parameters optimized for each factor. The solar absorptance plots were similar to those in Figures 7-19 and are not repeated here.

A final Taguchi experiment was performed to verify that the process parameters were improved to optimization. The power, distance from the plasma spray gun to the work piece, and argon gas pressure were evaluated at two variable parameters and optimized for the minimum values. The power setting of 650 watts was found to provide the lowest solar absorptance and density, while the 700 watts setting provided the fewest number of coats. Similar results were observed with distance from the plasma spray gun to the work piece. The distance of 22.86 cm (9 inches) provided the lowest solar absorptance and density while 20.32 cm (8 inches) resulted in the lowest number of coats. Table 8 shows the optimized settings from the final Taguchi experiment.







Figure 21 Factor Level Effect on Coating Density



Figure 22 Factor Level Effect on Solar Absorptance

Factor	Setting	Solar Absorptance	Number of Coats	Density
Power	700 watts	0.114	21.5	2.455 (g)
	650 watts	0.116	31.0	2.432 (g)
Distance	20.32 cm (8in)	0.118	20.5	2.482 (g)
	22.86 cm (9in)	0.113	32.0	2.404 (g)
Argon Gas	65 psi	0.116	29.5	2.376 (g)
	80 psi	0.115	23.0	2.511 (g)

Table 8 Alumina Plasma Spray Taguchi Verification Matrix

The differences between the verification results and the factors indicate the relative influence of that factor on a result. For example, the difference in the distance on the number of coats is 11.5 which indicates that 20.32 cm is significantly more efficient that at 22.86 cm. Figure 23 shows the strengths of the factors and the settings.



The settings were optimized for the minimum solar absorptance and were used to plasma spray the samples used for the screening testing and as the basis for the Standard Operating Procedure (SOP) draft for both plasma spraying the alumina and the blended powders. Table 9 lists the optimized plasma spray settings. The settings were made for coating thickness of 0.35 mm (0.0015 in).

Power	650 watts
Distance	22.86cm (9in)
Argon Gas	80 psi
Gun Angle	90 degrees
Helium Gas	35 psi
Gun Speed	4 in/sec
Powder Feed	2.3 rpm
Powder Gas	25 psi

Table 9 Plasma Spray Settings

2.1.2.2 Custom Porcelain

Plasma spraying experiments were initially made using a commercially available borosilicate frit (pyrex) to develop the plasma spraying parameters. The results indicated that the frit could be successfully applied to carbon-carbon substrates. The plasma spray parameters were transferred to the custom porcelain. The ZOT pigment encapsulated into the borosilicate glass matrix was successfully applied to the carbon-carbon substrates. The coating had a yellow tint and had poor optical properties. The solar absorptance was 0.436 and the emittance was 0.898. The custom porcelain was eliminated with customer concurrence from the potential thermal control coatings evaluation.

2.2 SUBSTRATE MATERIALS

Candidate materials for substrates were based on materials which are representative of current and projected spacecraft uses. The current design of most large radiators are aluminum and this material has served as a benchmark for composites in the thermal conductivity regard. However, the advanced radiator designs incorporate the use of the high thermal conductivity composite materials. The three candidates selected were 6061-T6 aluminum, organic matrix composite (K1100/ cynate ester) and carbon-carbon (K1100/C).

The C-C substrates used in the initial coating trials and screening tests were made using a lower cost fiber T300 and K640 resin system.

2.3 BARRIER COATINGS

Several considerations were given for the coating/interface. These considerations included thermal expansion mismatch, barrier to evolved gases and atomic oxygen protection, surface preparation for

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adequate adherence, and reflective undercoat. The plasma spray industry commonly uses adherence layers to provide a transition between materials which have large differences in thermal coefficient of expansion. Tailoring of these layers can allow the mechanical properties of the stresses to be minimized. Additional benefits are possible for carbon-carbon substrates in the form of oxidation protection. The candidates evaluated were bare substrate with and without a light grit blast, manually applied sodium silicate, plasma sprayed nickel encapsulated silicon carbide, and plasma sprayed aluminum. A finite element model (FEM) was created to analyze the stresses in the bond line and test coupons were prepared to evaluate each candidate.

A benefit from a plasma sprayed reflective undercoat was noted. A K1100 cynate ester substrate was plasma sprayed with a 0.0017 inch aluminum barrier coating followed by a 0.0064 inch alumina. The solar absorptance of this sample was 0.128 and emittance of 0.841.

2.3.1 FEM Activities

The FEM computed stresses for the temperature range of -200°F to +100°F which were based on the International Space Station operating temperature range. The assumption was made that the coating interface was in the stress free state at 100°F. The FEM evaluated the shear stresses at the coating interface, tension stresses in the coating cross section, and the peel stress perpendicular to coating interface. The FEM was constructed with 1952 nodal elements. The cases analyzed were 1.01 mm (0.040 in) thickness C-C K640 and K1100 C-C substrates with 90° layup. Barrier coatings of bare and .02mm (0.001in) nickel encapsulated silicon carbide were included in the analysis. The thermal control coatings analyzed included 0.30 mm (0.012 in), 0.36 mm (0.014 in), and 0.41 mm (0.016 in) alumina and custom porcelain. The alumina was the only oxide used in the FEM due to property similarity to the blended oxide powder. The results indicated that the tension stresses were not effected by a barrier coating and the stresses were approximately 4000 psi for the alumina and 2000 psi for the custom porcelain. The shear

stresses were approximately 2000 psi for the alumina and 1000 psi for custom porcelain without a barrier coat. However, a 50 % reduction in stresses was noted with a barrier coating. The shear stresses were considered minor for this application. The peel stress at the coating interface was approximately 1100 psi for the alumina and 600 psi for the custom porcelain without a barrier coating. Based on comparative literature, values of fracture stress for the porous plasma sprayed alumina are approximately 10000-11000 psi. This indicates that a tension failure in the coating is unlikely to occur for the conditions analyzed. The structural analysis report for the coatings and substrates is attached in Appendix A.

2.3.2 Screening Tests

The screening tests were organized into Levels 1 and 2 activities. The Level 1 testing was performed with substrates coated with each of the oxide powders, barrier coatings, and application techniques. The most promising candidate combination of barrier and thermal control coatings was carried into Level 2 screening. The Level 1 screening included thermal shock, solar absorptance, normal emittance, appearance and uniformity, plasma spraying ability, coating weight and thickness evaluation, and microscopic evaluation of the coatings and interface. The Level 2 screening included space stability testing, atomic oxygen resistance evaluation, flexure, vibration, thermal cycling, adhesion, outgassing, and moisture compatibility evaluation.

2.3.2.1 Level 1 Screening

The coating integrity was evaluated using a thermal shock test and pneumatic adhesion tensile testing. Three specimens were heated in an oven to 200°F and quenched in liquid nitrogen (320°F). No spalling or flaking was observed on any specimen. The pneumatic adhesion tensile testing was conducted per ASTM D4541. A ¹/₄ inch diameter shank was bonded on the coating surface and pulled in a tensile direction to provide a direct measurement of adhesion strength. The results were compared to the direct adhesion of Z-93P values. Table 10 shows the comparisons of the adhesion values used in the screening tests.

Coating	Sub	strate	💭 Value (psi	
Z-93P	Alun	ninum	40-150	
Al ₂ O ₃	¢	×	75	
Al ₂ O ₃	Alun	ninum	121	

Table 10 Coating Adhesion Evaluation

The solar absorptance, normal emittance, coating appearance and uniformity, coating weight, and thickness evaluations were recorded for each plasma spray candidate, spray trial and were part of the Taguchi experiments conducted for process optimization. The results of the plasma spraying trials were shown in Tables 5, 7 and 8.

The ability of the powders to plasma spray was evaluated by characterizing the size, phase, shape, particle size, surface area. The particle size was evaluated to determine the ability of the powder to fit through the plasma spraying equipment and the size of the plasma sprayed particle or splat. The splat size and shape influence the ability of the thermal control coating to reflect and scatter light. The particle has to be large enough to plasma spray, adhere to the substrate, and not be lost in the over spray. The particle also must be small enough to enter into the plasma chamber of the gun and properly feed into the plasma gas for the trajectory action onto the substrate. The powders which met this criteria were materials supplied by Praxair, customer, and the blended oxide powders made by LMVS and the Alpha Aesar alumina. The particle sizes are shown in Table 11.

Powder		Particle Size (um)
Praxair		2.5-3.0	
Customer		4.8	
Blended		2.6	
Alpha Aesar		1.8	

Table 11 Particle Size
The crystallographic phases of the powders were analyzed by X-ray diffraction. The alumina powders from Praxair and Alpha Aesar were identified to be alpha phase.

The ratio of intensity of the alumina (003) peak to the zirconia (111) peak represents the relative ratio of compounds of the blended powder. The ratio was 0.79. The yttria peak was not used in the characterization and identification of the powder blend due small amount of the material present.

The shape of the powders was determined by Scanning Electron Microscopy. The shape of the plasma spray powder was analyzed to determine the ability of the powder to flow through the feed system into the plasma spray gun. The desired shape of the powder is primarily spherical. Figure 24 shows the powder shapes of the material supplied by Alpha Aesar, Praxair, customer, and LMVS blended powder.



1/6/99 - PPS-903213-021.PPT Figure 24 Plasma Spray Powder Macrographs

The surface area of the customer furnished and the LMVS blended powders was evaluated for comparison. The surface area of the particle effects the reflectance of the coating by the splat configuration and the ability of the powder to flow. The surface area of the customer furnished powder was 5.210 m^2 /g and while the LMVS blended powder was 3.128 m^2 /g. The smaller particles produced the most uniform thermal control coating. Figure 25 is an SEM photomicrograph of the customer furnished powder.



1/26/99 - PPS-903213-022.PPT

Figure 25 Top and Through Views of Plasma Sprayed Coating on C-C Substrate



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Figure 26 Top and Through Views of Plasma Sprayed Coating on C-C Substrate

2.3.2.2 Level 2 Screening

The Level 2 screening included space stability testing, atomic oxygen resistance evaluation, flexure, vibration, thermal cycling, adhesion, outgassing, panel backside temperature measurement and moisture compatibility evaluation. The combined radiation environment testing was conducted at the Space Combined Environments Primary Test and Research (SCEPTRE) facility at Wright Laboratory, Materials Directorate in Dayton Ohio. The samples tested were LMVS plasma sprayed ultrahigh purity alumina, customer furnished blended oxide powder, LMVS produced and plasma sprayed blended oxide powder, and Z-93 reference samples. The testing was conducted in a 10^{-7} to 10^{-8} torr. The solar simulation intensity ranged from 1.2 to 2.8 suns exposure across the samples. The electron exposure was a total fluence of approximately 1 X 10^{16} electrons/cm². The 1 KeV electron flux was at 3 X 10^9 e/cm²/s and the 10 KeV electron flux was at 6 X 10^9 e/cm²/s. All reflectance measurements were made in situ. The beginning of life (BOL) solar absorptance was approximately 0.10 for the alumina. The samples degraded in a uniform rate relative to the exposure level. At approximately 250 equivalent sun hours (ESH), the rate of degradation was reduced. At 1000 ESH, the solar absorptance ranged from approximately 0.21 to 0.23. The Z-93 solar absorptance values increased from approximately 0.14 at BOL to 0.16 at 1000 ESH. Figure 27 shows the SCEPTRE test results for the alumina.

The atomic oxygen resistance of the alumina and the blended powders plasma sprayed onto C-C substrates was evaluated using the plasma asher at the NASA Lewis Research facility. The samples were exposed to an accelerated flux of 3 X 10^{15} atoms/cm²-sec and a total fluence of 1 X 10^{21} atoms/cm². No erosion of the plasma coating or substrate under the coating was observed. Approximately 0.0025-0.005mm (0.001-0.002 in) depth of erosion was observed on the unprotected edges of the samples. The alumina samples showed optical property degradation similar to the SCEPTRE results shown in Figure 27. The BOL was 0.157 and the EOL was 0.174.



Figure 27 Sceptre Test Alumina Results

The blended powder samples showed negligible optical property change. The BOL was 0.168 and the EOL was 0.169. The normal emittance also showed negligible changes (BOL=.788, EOL=.789). The material loss, upon atomic oxygen plasma exposure for the alumina and blended powders, is shown in Figures 28 and 29. The mass loss was primarily due to the erosion at the unprotected edges.



Figure 28 Material Loss Upon Atomic Oxygen Plasma Exposure



1/12/99 - PPS-903213-029.PPT Figure 29 Material Loss Upon Atomic Oxygen Plasma Exposure

The thermal cycling response of the candidates was determined by thermally shocking 2.54 cm X 15.24 cm (1 in X 6 in) specimens using an exposure of 20 minutes at 200°F, followed by a 5 minute exposure in liquid nitrogen (-320°F). The specimens were visually inspected before and after the thermal shock testing. No apparent damage to the bond interface was observed. These specimens were then used for the flexure, vibration, outgassing, and moisture compatibility evaluations.

The coating flexure was evaluated by bending a specimen around a 2.54 cm (1in) diameter rod. Both thermal cycled and non-thermal cycled specimens were tested. All failures either occurred in the substrate or in the coating/substrate interface. All of the thermal control coatings, barrier coatings and nonmetallic substrates were tested. Table 12 shows the flexure test results.

Panel	TCC/Barrier/Substrate	Pre Ti	nermal and a second	Post Thermal		
		Substrate Failure	Coating Failure	Substrate Failure	Coating Failure	
12	Al ₂ O ₃ , No Barrier, C-C	15	15	12	12	
36	Al ₂ O ₃ , Al, K1100	13	19	8	14	
44	Al ₂ O ₃ Ni-SiC, C-C	15	15	12	12	
45	AF, NI-SIC, C-C	12	12	13	13	
46	Blended, Ni-SiC,C-C	15	15	13	13	
50	Al ₂ O ₃ , Ni-SiC, K1100	7	9	7	7	
56	AF, No Barrier, C-C	13	13	14	-14	
62	Blended, No Barrier, C-C	14	14	12	12	
76	Blended, Ni-SiC, K1100	11	11	13	12	
78	AF, AI, K1100	9	12	7	10	
80	AF, Ni-SiC, K1100	12	12	7	10	
82	Blended, Al, K1100	12	12	10	16	
86	AF, AI, C-C	11	11	14	11	
94	Al ₂ O ₃ Al, C-C	•	*	14	14	
95	Blended, Al, C-C	14	14	10	10	
Z-93	AJ		38			

Table 12 Flexure Test Results

NOTES:

AF= Customer supplied Al₂ O₃/ZrO₂/Y₂ O₃. Blended = LMVS produced Al₂ O₃/ZrO₂/Y₂ O₃. Al= Aluminum. Ni-SiC= Nickel/Silicon Carbide. C-C= Carbon-Carbon *= Specimen broke before reading could taken.

The vibration testing was conducted at the same levels as the Heat Rejection Subsystem (HRS) for the Space Station. The testing was conducted in 180 second durations at 135, 138, 141, and 144 dB Over All

(OA) sound pressure levels. The final phase was conducted at 1430 seconds at the 144 dB-OA level. The specimens were visually examined after each testing level. No delaminations were observed from the testing. The microphone data confirmed that the test environments were equivalent to those proposed for the tests and was within the allowable tolerances. The testing details are given in the Appendix herein.

The adhesion of the coating candidates was evaluated by using ASTM D4541, "Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers." The test determined the extent to which the coating, barrier, and substrate type effected the pull-off strength of the coating. The overall highest pulloff strength (242 psi) of the candidates was customer supplied blended powder/aluminum barrier coating/K1100 organic matrix system. The highest pulloff strength (170 psi) on a C-C substrate was the ultra pure alumina/nickel silicon carbide system. The adhesion test results are shown in Table 13.

Panel	Coatings	Average Pulloff Strength (psi)	Disbonding Areas
12	Al ₂ O ₃ , C-C	60	Substrate/TCC Interface
44	Al ₂ O ₃ , Ni-SiC, C-C	170	Substrate Failure
94	Al ₂ O ₃ , Al, C-C	91	Substrate/TCC Interface
50	Al ₂ O ₃ , Ni-SiC, K1100	221	Substrate Failure
36	Al ₂ O ₃ , Al, K1100	128	TCC Failure
56	AF, C-C	108	Substrate/TCC Interface
45	AF, Ni-Si,C-C	149	Substrate Failure
86	AF, AL, C-C	84	Substrate/TCC Interface
80	AF, Ni-SiC, K1100	148	Substrate Failure
	AF, AI, K1100	242	Substrate/TCC Interface
62	Blended, C-C	82	Substrate/TCC Interface
46	Blended, Ni-SiC, C-C	134	Substrate Failure
95	Blended, Al, C-C	99	Substrate/TCC Interface
76	Blended, Ni-SiC, K1100	150	Substrate Failure
82	Biended, Al, K1100	162	Substrate/TCC Interface

Table 13 Coating Adhesion Test Results

NOTES:

The coating systems are listed as thermal control coating, barrier coating, and substrate. The coating systems are the same as listed in Table 12.

The moisture compatibility of the coatings was evaluated in accordance with MIL-C-48497. The testing included exposure at 120°F with 95-100% relative humidity. The testing also included moderate abrasion with a clean dry cheese cloth, acetone immersion and wiped with cheese cloth, ethyl alcohol wiped with cheese cloth, severe abrasion with an eraser, saline solution immersion, and distilled water

immersion. The only cracking or spalling was observed during the last five tests on the alumina/ aluminum/C-C candidate system. All other systems were unaffected by the moisture compatibility testing. Figure 30 shows the cracked specimen.

The outgassing evaluation was conducted by visually examining the K1100 organic matrix material during plasma spraying for resin bleed through. No bleed through was observed during the plasma spraying operation.

The backface temperature of the panel was measured to determine bondline temperature capability. Eight "j" thermocouples were bonded to the backface of a C-C panel. Five coats of powder were plasma sprayed on the panel. The measured temperature was determined to be approximately 344°F, which makes the operation compatible with 350°F panel bond line requirements.



Figure 30 Cracked Alumina/Aluminum/C-C Specimen

2.4 ADDITIONAL COATINGS

Additional coatings were evaluated as part of a contract change. The coatings included very low solar absorptance materials, gallium oxide and a gallium/gadolinium oxide and additional alumina powders produced to the LMVS SOP from additional sources. Attempts were made to plasma spray these powders in both the as-received and the spray dried conditions. The materials in either condition plasma sprayed poorly and produced gray colored coatings. The solar absorptance of the gallium oxide was 0.588 and the emittance was 0.875. The gadolinium oxide optical properties were not measured due to visual similarity to the gallium oxide. With customer concurrence, the additional activities for the gallium oxide and the gallium/gadolinium oxide powders were not pursued and the powders were dropped from evaluation. The alumina powders from additional sources were included in the Task 3 evaluations.

3.0 TASK 3. MATERIALS CHARACTERIZATION/QUALIFICATION

The overall objective of this task was to characterize and qualify the selected coating system on the substrates from Task 2. These activities included procurement of powders from commercial sources. The characterization and qualification testing were performed on the specimens. The testing included thermo-optical properties, materials durability, space compatibility, contamination potential evaluations and an optical performance as a function of coating thickness study.

3.1 THERMO-OPTICAL PROPERTIES

The hemispherical emittance for the LMVS produced blended powder, ultra pure alumina, and a Z-93P reference sample was measured by AZ Technologies in Huntsville, Alabama. The instrument used for the measurements was a TEMP 2000. The hemispherical emittance for the specimens is shown in Table 14.

Powder		Normal Emittance	Hemispheri	cal Emittance
LMVS Blender	đ	0.829	0.	792
Alumina		0.833	0.	800
Z-93P		0.950	0.	915

Table 14 Hemispherical Emittance Measurements

3.2 MATERIALS DURABILITY

The materials durability evaluations included flexure, vibration, thermal cycling, adhesion and moisture compatibility. These tests were conducted concurrently with Task 2 activities and the results are reported in the applicable sections.

3.3 SPACE COMPATIBILITY

The space compatibility of the candidate coatings were evaluated in the combined atomic oxygen and vacuum ultraviolet (VUV) radiation testing facility at NASA Lewis Research Center and the VUV radiation testing SCEPTRE Facility at AFRL/MLBT. The samples were exposed to a directed beam of atomic oxygen at an accelerated flux level of 8.6×10^{14} atoms/cm²-sec. The total effective fluence was 1 $\times 10^{21}$ atoms/cm². The VUV exposure was approximately 3.5 suns for half of the exposure and 5.6 suns for the remainder. The 5.6 suns exposure was due to a filter not being installed. The additional exposure did not result in a visible darking of the samples. The test results indicate that the plasma sprayed blended powder performed similar to Z-93P. The plasma sprayed alumina loss roughly twice as much mass as did the plasma sprayed blended powder samples. All of the plasma sprayed samples were applied to C-C substrates. No significant changes in optical properties were observed. Figure 31 shows the combined environment test results from NASA Lewis.

The blended powders were also tested in the SCEPTRE facility. The testing included two LMVS produced and plasma sprayed blended samples (LMVS AZ), two customer furnished powder which LMVS plasma sprayed samples (LMVS GA-AZ), one customer furnished powder from a previous contract sample (GA-AZ), and one Z-93 reference. The LMVS AZ and the LMVS GA-AZ performed similarity in the test and were within a normal data scatter. The GA-AZ had the highest BOL (.21) and EOL (.31). The LMVS AA had the lowest BOL (0.10) and an EOL at 0.25. The LMVS AA performance was similar to those reported in Figure 27. The LMVS plasma sprayed blended powders indicated that the process was repeatable and consistent. Figure 32 shows the SCEPTRE test results.



1/12/99 - PPS-903213-031.PPT Figure 31 Atomic Oxygen and VUV Combined Radiation Test Results



1/12/99 - PPS-903213-032.PPT Figure 32 Blended Powder Sceptre Test Results

3.4 CONTAMINATION EVALUATION

The contamination evaluation included Total Mass Loss (TML) and Collected Volatile Condensible Materials (CVCM). The testing was conducted in accordance with ASTM E595 at NuSil Technologies. Samples were plasma sprayed and collected on a cold steel plate. The samples were the coating candidates customer furnished GA-AZH10, ultra pure alumina, and LMVS blended powder. NuSil Technologies reported 0.00% TML and CVCM. No contamination potential from the plasma sprayed coatings would be anticipated.

3.5 OPTICAL PERFORMANCE AS FUNCTION OF COATING THICKNESS

The LMVS blended powder was plasma sprayed onto two 15.24 cm X 15.24 cm (6in X 6in) K1100 C-C panels. One panel was bare and the other was coated with nickel encapsulated silicon carbide. The solar absorptance and emittance were measured after each applied layer to a thickness goal of 0.254mm (0.010 in). The emittance remained fairly constant at 0.78 while the contract goal of 0.15 solar absorptance for both the bare and nickel silicon carbide coated panels, was achieved at a coating thickness of approximately 0.381mm (0.015 in). Table 15 gives the optical properties and number of coats correlation. Figures 33 and 34 show the data charts.

Coating Number	Ni-SiC C	pated	Bare		
	Solar Absorptance	Emittance	Solar Absorptance	Emittance	
1	0.647	0,780	0.665	0.778	
2	0.476	0.785	0.492	0.786	
3	0.376	0.785	0.338	0.797	
4	0.316	0.783	0.322	0.786	
5	0.277	0.782	0.284	0.783	
6	0.243	0,780	0.241	0.783	
7	0.214	0.782	0.208	0.783	
8	0.195	0.778	0.193	0.781	
9	0.183	0.781	0.182	0.781	
10	0.171	0.780	0.169	0.783	
11	0.163	0.783	0.162	0.779	
12	0.157	0.779	0.156	0.779	
13	0.151	0,779	0,151	0.780	
14	0.146	0.777	0,145	0.776	
15	0.142	0.776	0.143	0.776	
16	0.138	0.779	0.142	0.779	
17	0.134	0.777	0.134	0.778	
18	0.131	0.777	0.132	0.777	
19	0.129	0.775	0.126	0 776	

Table 15 Optical Properties Vs Coating Thickness



1/12/99 - PPS-903213-033.PPT Figure 33 Emittance Vs. Coating Thickness



Figure 34 Solar Absorptance Vs. Coating Thickness

3.6 LARGE LOT PROCUREMENTS

Two large lots of the blended powder were procured to the specification/SOP number 507-18-411 from different vendors. Sixty-eight kilograms (150 pounds) were procured from Praxair Surface Technologies and 15 kilograms (31 pounds) were procured from Contract Materials Processing. The purpose of the large lot procurements was to demonstrate the scale up potential of the production process from multiple vendors. The powders were characterized for particle size distribution, surface area, chemistry, crystallography, and thermo-optical properties.

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3.6.1 Powder Properties

The powder specific surface area, median particle size, trace elements, and crystallographic phase ratio was analyzed from each vendor. The specific surface areas of the powders were similar. However, the median particle size varied by 15.9µm. The Praxair powder contained approximately 0.4% sodium as a trace element impurity, whereas the Contract Materials Processing powder contained a trace element below the detectable limits. The crystallographic phase ratios also varied by a factor of approximately 2.6. The measured powder properties of the powders and the initial specification requirements are shown Table 16.

Powder Property	Specification Requirements	Praxair Supplied Data	LMVS Measured Properties for Praxair	Contract Materials Processing
Specific Surface Area (m²/gm)	3.08±0.05	3,36	3.35	3.44
Median Particle Size (µm)	14.3±0.9	37.4	27.2	11.3
Trace Element Impurities (%)	<0,1	0.44%	0.4% (Na)	NA
Crystallographic Phase latomine (113)/Idmonta (111)	1.37±0.06	NA	3.55	0.95

Table 16 Measured Powder Properties

NOTES: Contract Materials Processing powder properties were measured by LMVS.

The particle difference between the two powders is shown in Figure 35. The particle size and shape influence the powder flow characteristics, plasma gun feed rates, and powder deposit efficiencies. The larger, spherical particles tend to produce more efficient powder usage. However, coating uniformity and optical properties tend to be produced by the smaller particle size.



1/12/99 - PPS-903213-035.PPT Figure 35 Powder Micrographs

3.6.2 Chemical Composition

The gross chemical compositions for the powders from each vendor were determined by wet chemical analysis. The chemical analysis is shown in Table 17.

Element	Praxair	Contract Materials Processing
Oxygen	55.46	54.76
Aluminum	37.31	34.69
Zirconium	6.28	9.29
Yttrium	0.55	1.21
Other	0.4	0.05
Total	100	100

Table 17 Gross Chemical Composition (wt%)

3.6.3 Thermo-Optical Properties

The solar absorptance and emittance of the plasma sprayed powders from each vendor were measured at LMVS. The Praxair powder was slightly less white than the Contract Materials Processing powder. The room temperature optical properties for the two powders are shown in Table 18.

Optical Property	Specification Requirem	nent Praxair	Contract Materials Processing
Solar Absorptance	0.17 maximum	0.18	0.15
Emittance	0.78 minimum	0.81	0.79

Table 18 Plasma Sprayed Vendor Powders Optical Properties

The space stability testing of the procured powders was conducted at the SCEPTRE facility. At approximately 2200 equivalent sun hours, the Contract Materials Processing had the lowest solar absorptance at 0.290. The other powders were within the data scatter of the testing. Praxair-Indianapolis, Praxair-Seattle and LMVS produced the other powders. At approximately 1000 ESH, all of the powders ranged from 0.265-0.281. The CMP and LMVS powders were 0.265 and 0.267 respectively. The contract goal was 0.20 at 1000ESH. The degradation of the solar absorptance appeared to become less at approximately 106 ESH. A second change of degradation of the solar absorptance appeared at approximately 1000 ESH. The test results are shown in Table 19 and Figure 36.

Exposure Time (actual hrs)	ESH (hrs)	Praxair-IN (average)	Praxair-WA (average)	CMP (average)	LMVS (average)
0	0	0.174	0.186	0.141	0.154
22	48.4	0.195	0.195	0,152	0.177
48	105.6	0.205	0.210	0.169	0.195
115	253	0.223	0.220	0.189	0.212
200	440	0.241	0.237	0.207	0.225
301	662.2	0.260	0.260	0.232	0.248
451	992.2	0.281	0.280	0.265	0.267
600	1320	0.285	0.294	0.270	0.280
672	1478.4	0.286	0.298	0.273	0.284
810	1782	0.289	0.301	0.281	0.288
1000	2200	0.301	0.314	0,297	0,306

Table 19 Space Stability Testing



Figure 36 Sceptre Test Results

Standard operating procedures were developed for production of the ultrahigh purity powder, blended alumina/zirconia/yttria powder, and the plasma spraying operations. The SOPs for the alumina/zirconia/ yttria powder production and the plasma spraying were integrated into the material specification 507-18-411 and process specification 508-17-30. The specifications are attached in the appendix herein. The SOP for the ultrahigh purity powder is also attached in the appendix herein.

5.0 TASK 5. MATERIALS DEMONSTRATION/TECHNOLOGY TRANSITION AND EARTH ORBITER-1 CARBON-CARBON RADIATOR DEMONSTRATION

The overall objective of this task was to demonstrate uniform quality and properties of the coating applied by the approved SOP and to fabricate a carbon-carbon radiator panel for the Earth Orbiter-1 (EO-1) spacecraft. The task included the demonstration of the plasma coating using powders purchased from Contract Materials Processing and Praxair-Surface Technologies and applied by Praxair-Thermal Systems using robotic equipment. Both the powders purchased and the plasma spraying were conducted following the requirements and guidelines from the approved SOPs.

5.1 MATERIALS DEMONSTRATION/TECHNOLOGY TRANSITION

5.1.1 Transition Plasma Powder Production

The SOP for the powder production was submitted to five vendors for potential production. The four vendors were: Contract Materials Processing, Nyacol Inc. Praxair Surface Technologies, and Praxair Specialty Powders. All of the vendors were interested in participating in the program, except for Nyacol Inc. Nyacol Inc. declined to submit a bid. Powders were procured from the remaining four and were evaluated in the TASK 3 activities. An economic review of the delivered powder costs indicated that the powder produced by Praxair-Surface Technologies was the lowest cost per kilogram or pound and can be traced to larger scale lot production capabilities. The powder produced by Contract Materials Processing appeared to have been made by the methods following closest to the SOP and had the best optical properties and space stability performance. The cost was more than the Praxair Surface Technologies and could be traced to the capability of the equipment limitations of smaller lot sizes. The powder costs are shown in Table 20. LMVS produced powder is included in the table for comparisons.

Venuor see a second second second	Cost/Pound
Contract Materials Processing	\$406
Praxair Surface Technologies	\$55
Praxair Specialty Powders	\$450
LMVS	\$250

Table 20 Procured Powder Costs

5.1.2 Transition Plasma Spray Process

A 0.6m X 0.6m (24in X 24in) radiator panel with K1100 C-C face sheets was coated with powder from Contract Materials Processing on one side and Praxair Surface Technologies on the other at Praxair Thermal Systems. A schematic of the radiator panel is shown in Figure 37 and the actual article is shown in Figure 38.



1/27/99 - PPS-903213-037.PPT Figure 37 Demonstration Article Schematic

The activity demonstrated the plasma spraying SOP and the transition to a commercially available automated plasma spraying system. The plasma spray system used a SG-100 plasma gun attached to a robotic manipulation arm. Figure 39 shows the tooling, fixturing, and radiator panel. The Praxair Surface Technologies powder was applied to an approximate thickness of 0.017inch in 4 passes. The coating had a non-uniform appearance. The Contract Materials Processing powder was applied to an approximate thickness of 0.0155 inch in 14 pass. This coating had a uniform smooth appearance. Both coatings had a porosity of 21-28%. Figure 40 shows the coating microstructure.



1/14/99 - PPS-903213-040.PPT Figure 38 Demonstration Article



1/14/99 - PPS-903213-038.PPT Figure 39 Fixturing, Tooling, and Coating of Demonstration Article



Figure 40 Coating Cross Section X 250

5.1.3 Demonstration Article Acoustic and Optical Properties Testing

5.1.3.1 Acoustic Testing Results

The demonstration article was attached to a picture frame fixture using "Z" stringer restraints. The test levels and duration were the same as those used in Tasks 2 and 3. Those levels and duration were: 135 dB, 180 sec (Acceptance –3bD); 138bD 180 sec (Acceptance level); 141 dB, 180 sec (Maximum flight level); 144 dB, 180 sec (Qualification level); 144 dB, 23 minutes, 50 seconds (Qualification-Endurance). The demonstration article was visually examined after each test level and duration. No delaminations or coating cracking from the acoustics testing were observed. Additional details of the acoustics test are contained in Appendix H.

5.1.3.2 Optical Properties Testing

The optical properties were measured for the demonstration article by plasma spraying 15.24 cm X 15.24 cm (6 inch X 6 inch) C-C panels and 2.54 cm (1 inch diameter) C-C discs. Powders from CMP and Praxair Surface Technologies were used for the optical properties evaluation. The average solar

absorptance of the CMP samples was 0.162 and emittance was 0.811 at a coating thickness 0.38 mm (0.015 inch). The solar absorptance and emittance were 0.136 and 0.804 respectively at 0.53 mm (0.021 inch) coating thickness. The average solar absorptance and emittance of the Praxair Surface Technologies samples were 0.180 and 0.811 at a coating thickness of 0.38mm (0.15 inch). The discs were submitted for space stability testing at the SCEPTRE facility.

5.1.4 Cleaning Methods and Vendor Plasma Sprayed Samples Space Stability Testing

The vendor plasma sprayed and soiled and cleaned discs were submitted for space stability testing at the SCEPTRE facility. The testing was conducted for 100 ESH at similar radiation levels as performed during previous evaluations. Eleven combinations of soils and cleaning methods were evaluated. The most effective cleaning method for cleaning dusty fingerprints was an air/grit blast combination. The resulting increase in solar absorption was 0.03 after 100 ESH exposure. The least effective cleaning method was a grit blast/methyl ethyl ketone (MEK)/isopropyl alcohol (IPA) wipe. The resulting increase in solar absorption was 0.103 after 100 ESH exposure. The solvent cleaning methods appeared to disburse the soils into the coating porosity. The remaining soils/cleaning combinations appeared to follow a similar degradation pattern as did the plasma sprayed control specimens. The S-13G/LO and Z-93 did not appear to degrade significantly at 100 ESH exposure. The soils and cleaning methods are listed in Table 21 and the space stability testing results are shown in Figure 41.

	A rading opicjou ounproo			
Specimen Marking	Specimen Identification			
Z-93	Z-93			
S13G/LO	S13G/LO			
CMP	Alumina/Zirconia/Yttria from CMP			
ALO	Alumina/Zirconia/Yttria from Praxair			
GBR/CAB	Grit Blast Residue/Compressed Air Blast			
GBR/MEK/IPA	Grit Blast Residue/MEK & IPA Wipe			
GBB/MEK& IPA/GB	Grit Blast Residue/MEK & IPA Wipe/Grit Blast			
GBR/CAB/GB	Grit Blast Residue/Compressed Air Blast/Grit Blast			
DF/CAB/GB	Dusty Fingerprints/Compressed Air Blast/Grit Blast			
DEMEK & IPA	Dusty Fingerprints/MEK & IPA Wipe			
DE/Alconex	Dusty Fingerprints/Alconox Wash			
DF/Alconox/GB	Dusty Fingerprints/Alconox Wash/Grit Blast			
PI /MEK & IPA/GB	Pencil Lead/MEK & IPA Wipe/Grit Blast			
PI MEK & IPA	Pencil Lead/MEK & IPA Wipe			
PL/MEK & IPA/GB	Pencil Lead/MEK & IPA Wipe/Grit Blast			

Table 21 Cleaning and Vendor Plasma Sprayed Samples



5/20/99 – PPS-903213-054 Figure 41 SCEPTRE Testing Results for Cleaned and Vendor Plasma Sprayed Specimens

5.2 EO-1 CARBON-CARBON RADIATOR

The EO-1 spacecraft is the first in a series of earth orbiting missions for the NASA New Millennium Program. The EO-1 mission will validate a number of revolutionary technologies that will provide Landsat follow on instrumentation with increased performance at lower cost. In support of the New Millennium effort the Carbon-Carbon Space Radiator Partnership (CSRP); a consortium consisting of six Government and four industry participants, offered to supply a structural C-C radiator for integration into the NASA EO-1 spacecraft being fabricated by Swales Aerospace. A contract modification was negotiated for the design, fabrication, and testing of the structural EO-1 bay four radiator panel for the EO-1 spacecraft shown in Figure 42.



1/26/99 - PPS-903213-041.PPT Figure 42 Layout of EO-1 Satellite, Highlighting Bay Four

LMVS used CSRP and Swales requirements to design and build three C-C radiators consisting of GFE facesheets and inserts on aluminum honeycomb core and conducting subcomponent level mechanical, thermal, and electrical tests. Two C-C radiators were delivered to NASA for test and integration and the third one was used for subcomponent level testing.

5.2.1 Radiator Design/Analysis

Swales "EO-1 Spacecraft to Carbon-Carbon Radiator Interface Control Document (ICD)", SAI-ICD-028 that defined interface, configuration, mass, and mechanical requirements for the radiator panel. These requirements were used in the structural and dynamics analysis for defining edge inserts, insert installation, honeycomb type and density, and adhesives for the radiator fabrication. Figure 43 shows the radiator panel layout.



Figure 43 C-C Radiator Panel Layout of Boxes, Thermistors, and Inserts

The general configuration of the radiator is typical aluminum honeycomb sandwich panels and as such the design/analysis approach employed was characteristic of the same with the exception of the carboncarbon facesheets. The radiator consists of two 0.635 mm thick C-C facesheets bonded to 32.03 kilograms/m³ aluminum honeycomb. The panel is approximately 71 cm on a side and 2.54 cm in thickness. The radiator serves as the attachment platform for two electronic packages, PSE and LEISA. These packages are affixed to the panel by means of fasteners common to through holes in the packages and threaded inserts potted into the radiator.

The radiator is attached to the spacecraft by means of 18 fasteners located at the perimeter of the panel. The holes common to the panels were required to be through holes with the attached fasteners treading into attached hardware common to the spacecraft proper. Because of the nature of honeycomb

core, potted inserts are required at these locations; said inserts to function as the mechanism of load transfer from the spacecraft attach fasteners to radiator.

A radiator panel finite Element Model (NASTRAN) was created for mechanical design and performance safety factor analysis. All the hardware was designed and analyzed to the applicable safety factors specified in the ICD and indicated a positive margin of safety. The panel was designed to withstand quasi-static limit loads of $\pm 15g$ in any direction. The panel was designed to support a PSE mass of 20 kg with a CG offset of 13.5 cm and a LEISA Electronics mass of 5 kg with a CG offset of 11.5 cm from the panel surface. The panel also needed to sustain the following spacecraft loads while constrained at the attachment points.

Shear Loads of 16,100 N/m Edge Normal load of 19,500 N/m Panel Normal load of 1,850 N/m

The pertinent output from the analysis may be found in Appendix J.

The analysis of the edge inserts resulted in the selection of a NAS18334 type insert potted into the honeycomb using EA934NA potting compound. Proper insert installation required the potting material to extend beyond the insert diameter by approximately 2.54 cm. This approach resulted in a reduction of the loads induced by differential thermal expansion between the spacecraft and the C-C panel to 339.3 kg inplane and 77.5 kg normal. The design also maintained first mode frequency above 100 Hz.

Hand and F.E.M. analysis were used to derive stresses present in the potting compound employed to affix the inserts to the radiator. A margin of safety was calculated for both types of analysis and the lesser of the two used as the valid result. The ultimate margin of safety = +0.14 (ultimate). A margin of safety

for the potting compound/facesheet interface shear analysis, assuming the stress distributed over an area equal to the contact area of the potting compound to a single facesheet resulted in a margin of safety = +2.76 (ultimate). The analysis for the normal component of load for these inserts local to the electronic packages was performed as a simple shear out hand analysis utilizing potting compound shear allowable and resulted in a margin of safety = +Large (ultimate).

Analysis of the threaded inserts local to affixed electronic packages was performed. Data from the loads analysis (Appendix J) indicated that two locations local to the PSE packages have peek loads. The resultant load, due to inertia, resulted in a value of a 39.0 kg force in the in-plane direction and 127.0 kg force normal. The normal component is the peek value for all these locations. This analysis was performed as a simple shear out hand analysis and potting compound shear allowables. Utilizing the appropriate factor of safety, the ultimate margin of safety for this method and condition is equal to +LARGE (ultimate). The honeycomb core adjacent to the location in question was analyzed for shear out. Utilizing the appropriate factor of safety, the ultimate margin of safety for this method and condition is equal to +.00 (ultimate). The normal load induces a 'resisting shear' of form VQ/I between the carbon-carbon facesheets and the honeycomb core. Utilizing the appropriate factor of safety for this method and condition is equal to +.02 (ultimate). Because of very large relative difference in magnitudes of applied and allowable shear stresses the ultimate margin of safety is considered to be a positive large value for the core/facesheet adhesive system.

Analysis of the threaded inserts local to GSE electronic package was performed. In plane loads were minimal compared to that of other locations. Loads normal to the radiator at these locations have a maximum value of 69.8 kg. As previous analysis has shown that similar geometry and greater load has a positive margin of safety for potting compound allowables, no analysis was required. However, this load will be subsequently used for core analysis.

The honeycomb core adjacent to the location in question was analyzed for shear-out. The applied normal load was carried over an area subtended by a cylinder of radius indicated on the interface radiator drawing and a height equal to the core thickness. Utilizing the appropriate factor of safety, the ultimate margin of safety for this method and condition is equal to +.48 (ultimate). The normal load induces a 'resisting shear' of form VQ/I between the carbon-carbon facesheets and the honeycomb core. Utilizing the appropriate factor of safety, the ultimate margin of safety for this method safety, the ultimate margin of safety for this method and condition is equal to +.52 (ultimate).

5.2.2 Radiator Fabrication

Design engineering prepared E-size drawing of the EO-1 C-C radiator panels to be fabricated. Shown in Figure 44 is the assembly flow, materials, specification, and inspection operations used in the fabrication of the two radiator panels and test panel. Figures 45 and 46 are reproductions of the design drawings used to manufacture the radiator panels. A complete materials and specifications list for EO-1 radiator fabrication is shown in Tables 22 and 23, respectively.



1/14/99 - PPS-903213-043.PPT

Figure 44 EO-1 Radiator Assembly Flow Chart



1/14/99 - PPS-903213-044.PPT Figure 45 EO-1 Configuration Drawing



1/14/99 - PPS-903213-045.PPT Figure 46 EO-1 Configuration Drawing

.

Description	Part No.	Qty
0.025 C-C Face Sheet	Skin	2/Panel
Aluminum Honeycomb 2lb/ft ³	Honeycomb	AR
Blank Insert	EO-103	18
0.250-28 THD Insert	406HE428-14	8
#10-32 THD Insert	406HE428-14	14
Primer	EA9205	AB
Film Adhesive	EA9689	AR
Potting Compound	EA934NA	AR
Silver Teflon Tape	507-9-428 Type VI	AR

Table 22 EO-1 Radiator Fabrication Materials List

T	ab	le	23	EO-1	I Ra	idiator	Fa	brication	S	pecifica	tion	List

Specification	Туре
508-9-134	Process Specification
508-8-42	Process Specification
508-8-260	Process Specification
MMM-A-132	MATL Spec
507-8-447	MATL Spec
BFGHT2-LC	MATL Spec
MIL-A-7438	MATL Spec

The carbon-carbon panels were fabricated using the materials and specifications listed above. Fabrication operations and quality checks were performed and verified by the use of a laboratory traveler. These travelers defined each process step and were delivered as part of the quality package to NASA. Figure 47 shows the GFE supplied face sheets used to fabricate the panels in this task. Shown in Figure 48 is the tooling used to prepare the core material for insert installation and the tool used to install inserts into the panels. Test coupon panel and an EO-1 radiator panel are shown in Figures 49 and 50, respectively.



1/14/99 - PPS-903213-046.PPT Figure 47 GFE Supplied C-C Facesheets



1/14/99 - PPS-903213-047.PPT Figure 48 Radiator Fabrication Tooling


1/14/99 - PPS-903213-048.PPT Figure 49 C-C Radiator Test Coupon Panel



1/14/99 - PPS-903213-049.PPT Figure 50 EO-1 Carbon-Carbon Radiator

5.2.3 Radiator Panel Tests

Tests were performed on a panel fabricated like the EO-1 radiator to verify design parameters. The tests include insert pullout, insert bearing, flatwise tension and core shear. Results of the insert specimen tests (Table 24) indicate that the typical pullout and bearing strength values are either equivalent or higher than the anticipated values, thus providing adequate margin of safety in the operational environment. Table 22 also lists the test results of the thermal conductivity of P30X/C facesheet test specimens which was provided by NASA LARC.

Sandwich Flatwise Tension	
- Average Ultimate Strength	62.2 kPa (>43.5 kPa)
- Standard Deviation	8.3 kPa
• 2.0 pcf Core Shear	
- Parallel to Core Ribb on	
- Shear Strength	13.1 kPa (11.6 kPa/min)
- Shear Modulus	4.12 mPa (3.9 mPa)
- Transverse to Core Ribbon	
- Shear Strength	0.45 mPa (6.6 mPa/min)
- Shear Modulus	1.9 mPa (2.0 mPa)
Sandwich Insert (#10-32) Pullout	
- Average Max. Load	386.9 kg (104.3 kg)
- Standard Deviation	153.9 kg
Insert Shear Bearing	
- Average Bearing Stress	3.8 mPa (≅2.0 mPa)
- Standard Deviation	0.81 mPa
Thermal Conductivity	
- 00 Direction	214 W/m -K
- 900 Direction	213 W/m -K

Table 24. Radiator Panel Test Summary (Analytically Predicted Values)

P30X/C facesheet specimens were provided by NASA LARC for in-plane thermal conductivity tests. Results of these conductivity tests are also included here for completeness. While the test results are discussed in the following paragraphs, the specific test data and results are included in Appendix K.

Three types of inserts were used to fabricate the panel: a 0.250-28 THD insert, a #10-32 THD insert and a blank insert. The blank insert was drilled through to provide holes for the attachment bolts that secure the radiator panel to the frame of the spacecraft. Figure 51 shows the radiator test panel after installation of the inserts.

Lockheed Martin Astronautics, Denver, CO conducted the following mechanical tests of the EO-1 test panel. These tests included insert pullout, flatwise tension, pin bearing-blind inserts, and facesheet/core shear. Figure 52 shows the location of the test specimens from the panel. The flatwise tension and facesheet/core shear test specimens were cut from the clear areas (inserts-free) of the panel.



1/14/99 - PPS-903213-050.PPT Figure 51 Test Panel with Inserts



^{1/14/99 -} PPS-903213-051.PPT Figure 52 Layout of Test Specimens from the Radiator Panel for Destructive Testing

Table 25 lists the test results of the insert (dia. 1.4 cm.) pullout tests. The minimum pullout load was 249.5 kg and the maximum was 578.8 kg. Even the weakest specimen had a factor of 10 over capacity in the normal direction. Figure 53 shows a photo of specimen after failure. The large region of material pulled out with the insert is clearly visible.

Specimen ID	Specim	en Dimensions	s. cm	Maximum Load, kg	Comm	nents
	Thickness	Width	Length			
inPul-1 inPul-2 inPul-3 inPul-4	2.53 2.51 2.52 253	10.15 10.15 10.15 10.15	10.16 11.16 10.13 10.17	249.5 578.8 275.3 444.5	1-in. dia. plug r >1-in dia. plug >1-in dia. plug >1-in dia. plug	egion region region region

Table 25 Results of the Sandwich Insert Pullout Test

Torque test were performed to determine the torque required to disbond the threaded fasteners installed in the radiator panel. Three samples, approximately 10 cm by 10 cm with a 10/32 fastener installed in the center of the panel with Hysol 934 adhesive were tested. The panels were clamped in a vise and had a hex head bolt threaded into the fastener. The measured torque at which an audible crack was heard (signifying disbond of the adhesive) was recorded. The lowest torque at which the adhesive



1/14/99 - PPS-903213-052.PPT Figure 53 Post-Test Photo of Insert Pullout Test Specimen InPul-4

disbonded was 7.43 joules ± 0.127 joules. The average disbond torque was 7.61 joules ± 0.127 joules. Two fasteners held in the panel after disbonding until the hex head bolts sheared off at 8.58 joules.

Flatwise tensile tests were conducted on the honeycomb sandwich using ASTM Standard C29794. The results of the test are given in Table 26. The average measured strength was 194.6 kg with a standard deviation of 12.25 kg. The failures were in the bond line at the aluminum honeycomb core and C-C interface.

Specimen ID	Thickness, cm	Thickness, Length, cm cm		Max, Load. Kg	Ultimate Strength, kPa
	2.51	3.81	3.82	449.9	63.9
FI TEN-2	2.51	3.81	3.81	428.6	60.9
FLTEN-3	2.51	3.81	3.81	454.0	64.6
FLTEN-4	2.51	3.80	3.82	439.9	62.5
FLTEN-5	2.51	3.82	3.80	444.1	62.9
FLTEN-6	2.52	3.82	3.81	379.2	53.7
ELTEN-7	2.51	3.82	3.82	445.8	63.1
ELTEN-8	2.51	3.82	3.82	470.4	66.6
Average				439.0	62.2
Std. Dev				26.7	3.91
%CV				6.1%	6.1%

Table 26 Results of the Flatwise Tensile Testing

Insert bearing tests were conducted using the configuration shown schematically in Figure 54. Each specimen was prepared with the e/d (edge distance to insert diameter) ratio of 1.0 (insert diameter 0.1.27 cm, specimen width 2.54 cm). The average bearing load was 1522.5 kg with the standard deviation of 27.1 kg. The large scatter was primarily due to the core fill diameter around each insert varying from 0.20 to 2.54 cm. The failure was tensile in nature as the insert with the core fill acted as a large effective insert to an applied tensile load. The calculated average failure stress value of approximately 2.46 mPa is quite comparable with the tensile strength of the 0.508 mm P30X/C laminate.

Thermal conductivity test specimens were cut from sub-panel A. Six 0.635 cm x 15.24 cm x 0.55 cm specimens were cut in both the 0' and 90' directions. One half the specimens were sent to Lockheed Martin Astronautics and forwarded to TPRL and one half to NASA GSFC for testing.



Figure 54 Schematic of Pin Bearing Test Configuration

The samples submitted to TPRL were tested using the Kohlrausch method. The Kohlrausch method involves the determination of the product of the thermal conductivity " λ " and the electrical resistivity " ρ ". Since the electrical resistivity is measured at the same time as the product of resistivity and conductivity, λ can be calculated. The method involves passing constant direct current through the specimen to heat the sample while the ends are kept at constant temperature. An external heater whose center temperatures are maintained at the sample's midpoint temperatures and whose ends are also cooled by water or liquid nitrogen minimizes radial heat losses. Thermal conductivity values accurate to within ±5% are obtained by the Kohlrausch method and all measured quantities are directly traceable to NIST standards.

TPRL tested two of the six specimens. The results for sample TC-5 (90° direction) are given in Table 27 and the results for sample TC-11 (0° direction) are listed in Table 28. The conductivity of the TC-11 (0°) sample is about 2% greater than the conductivity of the TC-5 (90°) sample. The resistivity of the TC-5 (90°) sample is around 1.5 % greater than the resistivity of the TC-11 (0°) sample. Complete results are listed in Appendix K.

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Table 27 Sample TC-5 (90° Direction) Thermal Conductivity

Table 28 Sample TC-11 (0° Direction) Thermal Conductivity

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temperature, c	Conductivity, W/m-K	Resistivity, microhms-cm
51.4 72.4 95.9 119.3 144.6 168.5 193,0 216.8	21.5 213 209 202 197 195 190 190 185	546 532 519 507 496 487 479 472

6.0 CONCLUSIONS

The powder production and the plasma spray application methods were shown to be repeatable and reproducible on both a laboratory and industry scale. The powder and the plasma spraying operations were shown to be applicable to multiple substrates of aluminum, C-C, and organic matrix. The use of barrier coatings was also demonstrated. The powder was found to be economically feasible to produce on an industrial basis from multiple vendors. The Standard Operating Procedures were successfully developed, demonstrated and transitioned to industry. The blended powder was shown to be more space stable than the pure alumina for the test conditions described herein. However, the pure alumina was found to have the lowest BOL solar absorptance of the powders tested and slightly exceeded the contract goal of 0.20 at 1000 ESH. The coatings were successfully acoustically qualified to Space Shuttle launch loads. The coating was found to be compatible with a variety of substrates.

Alternate oxide powders were also successfully plasma sprayed.

The advanced technology C-C radiator for the EO-1 satellite was successfully designed, fabricated, and delivered for the New Millennium program.

Some areas which showed potential for additional development would include development of a cost effective thin transparent topcoat to enhance the emittance of the thermal control coating; additional development of reflective undercoats to provide a lowest solar absorptance with an optimized plasma sprayed thickness of the thermal control coating; and additional blends of thermally applied oxide coatings. Investigate additional technical areas for reducing powder production cost. Investigate larger scale (3m X2.4m) commercial demonstration of plasma spraying application.

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Appendix A Thermal Control Coating

DESIGN INFORMATION REQUEST - RELEASE

LOCKHEED MARTIN VOUGHT SYSTEMS

PROGRAMMODEL				DER NO.		REV	DATE		PACE	
Thermal Control Coatings		÷		3-47300/7DII	R-053		9 June 19	97	1 OF	4
PREPARED	DATE	GROUP	DATE		REASON FOR REVISION				REV DA	TR
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1.0 PURPOSE

The stress analysis on the coating layer of the space radiator/structure was conducted to evaluate the adhesion strength of the coating layer using the plasma spray technique. This is an initial screening effort to evaluate the material system including coating and barrier coating viability being used on the space radiator.

2.0 DESIGN CONFIGURATION

The baseline configuration for the study is a honeycomb sandwich structure as in the proposal, shown in Figure 1 and Reference (1). The design is to have the face sheet plasma sprayed with the reflective/thermal coating. The stress analysis is to study the face sheet response to assure the structural integrity of the coating so that its function as thermal barrier is not degraded.

3.0 MATERIAL SELECTIONS AND PROPERTIES

The analysis was conducted according to the parametric matrix shown in Table 1. Geometrical variations and material selection are the two variables for the parametric arrangement. The thickness of the substrate is fixed at 0.040 inch and that of barrier coating at 0.001 inch. The coating thickness ranged in three thicknesses: 0.012, 0.014, 0.016 inch. The geometry of the coating layer arrangement is shown in Figure 4.

The candidate face sheet materials (or called substrates) are aluminum 6061-T6, carbon/carbon K640, carbon/carbon K1100 and K1100 composite. For the initial screening, only carbon/carbon K640 and carbon/carbon K1100 are selected for analysis evaluation. The candidate coating materials are Al_2O_3 , $Al_2O_3 + ZrO+Y_2O_3$, and custom porcelain. Only Al_2O_3 (called type 1 coating in the analysis) and custom porcelain (called type 3 coating) were selected for evaluation. On barrier coating, the analyses used either NiSiC or no barrier coat between substrate and coating layer. The honeycomb is flexible relative to the substrate materials and is not included in the analysis model.

Material properties of the coating system are briefly evaluated. The carbon/carbon substrate materials have been obtained from the literature report in Reference (2).

Coating materials property selection are based on the literature in References (3), (4), and (5). It becomes obvious from these literatures that the coating porosity has great influence on the mechanical properties of the coating. In turn, the coating porosity is effected by the spraying technique and equipment used. Figure 2 is technical data that indicates the significance of the porosity effect on modulus and strength of the coating system. Due to the relative new experience on the coating spray of this program, it is necessary to make reasonable assumptions on the coating materials properties for the analysis study. A summary of the key properties referenced for analysis is shown in Table 2. For this initial study, the temperature effect on the mechanical properties of these materials are considered small for the temperature of -200°F to 100°F.

4.0 COATING ANALYSIS

Finite element analysis model shown in Figure 3 was built on NASTRAN QUAD4 elements and represents a 2.1 inch segment of the face sheet structure with unit width. The analysis results are summarized in Figures 4 through 6 for the carbon/carbon K640 substrate and Figure 7 through 9 for the carbon/carbon K1100 substrate. Tension Stress is related to the surface tension or surface cracking potential. Peel stress and shear stresses are relevant to the coating layer adhesion to the substrates.

5.0 CONCLUSIONS

- 1. The coating thickness range evaluated has little or no influence on the adhesion capability.
- 2. Type 1 coating (AL_2O_3) has significantly more stresses on the coating layers as compared to that of the type 3 coating (porcelain).
- 3. Peel stress of the interface region is lower for the K640 substrate than that of the K1100 substrate.
- 4. Barrier coat NiSiC plays only a minor role in influencing the stress state of the adhesion.

6.0 REFERENCES

- 1.0 LTV Report No. 3047300/R9501, Technical Proposal, "Thermal Control Coating for High Thermal Conductivity Substrates", June 12, 1995.
- 2.0 ICI Fiberite Literature, Technical Conference at Anaheim Marriott Hotel, April 12, 1994.
- 3.0 T. Ho, E. C. Matza, J. Medford, and S. Watabe, "Design Concept Study for NASP Control Surface", NASA-CR 181713, October, 1988.
- 4.0 H. Carrerot, J. Rieu, P. Girardin, G. Bousquet, A. Rambert, "Mechanical Properties of Porous Plasma Sprayed Coatings On Metal Stems for Joint Prostheses", in Ceramics in Substitutive and Reconstructive Surgery, Elservier Science 1991.
- 5.0 S. Kuroda, T. Fukushima, and S. Kitahara, "Significance of Quenching Stress in the Cohesion and Adhesion of Thermally Sprayed Coatings", Journal of Spray Technology, vol. 1 (4), December 1992.

LAYER	ER CANDIDATE MAT'L YOUNG'S MOD SHEAR MOD (Msi)		SHEAR MOD (Msi)	POISSON'S Batio	CTE (10 ⁻⁶ in/in/°F)		
		=	۲.	Ģ	Natio √	=	Ŧ
Substrate	 AI 6061-T6 C/C-K640 C/C-K1100 K1100 comp. 	9.90 16.80 41.00 41.00	9.90 0.50 0.50 0.80	2 3 5	0.33 0.04 0.04 0.04	12.8 0 -0.33 -0.33 -0.33	12.8 3.0 3.0 20.1
Barrier Coat	 Bare NiSiC Sodium-Silicate Al 	 2.70 2.70 0.43	2.70 2.70 0.43	 	 0.3 0.3 0.3	 2.8 2.8 12.8	 2.8 2.8 2.8
Coatings	 Al₂O₃ Al₂O₃+ZrO₂ + Y₂O₃ Custom Porcelain (glassy) 	2.9 2.7 2.7	2.9 2.7 2.7		0.3 0.3 0.3	5.7 5.7 2.8	5.7 5.7 2.8

MECHANICAL PROPERTIES OF SPACE COATING SYSTEM

NOTE: Eporus = $0.043 E_0$

3-47300/7DIR-053 Page 4 of 4

NASTRAN FILE NAME	SUBSTRATE TYPE	COATING TYPE	COATING THICKNESS (INCH)	BARRIER COAT TYPE
CC 640 BA 112 CC 640 BA 114 CC 640 BA 116		Al ₂ O ₃	0.012 0.014 0.016	BARE
CC 640 BA 312 CC 640 BA 314 CC 640 BA 316	CARBON / CARBON K640	Porcelain	0.012 0.014 0.016	
CC 640 SS 112 CC 640 SS 114 CC 640 SS 116		Al ₂ O ₃	0.012 0.014 0.016	NiSiC
CC 640 SS 312 CC 640 SS 314 CC 640 SS 316		Porcelain	0.012 0.014 0.016	
CC K11 BA 112 CC K11 BA 114 CC K11 BA 116		Al ₂ O ₃	0.012 0.014 0.016	BARE
CC K11 BA 312 CC K11 BA 314 CC K11 BA 316	CARBON / CARBON K1100	Porcelain	0.012 0.014 0.016	
CC K11 SS 112 CC K11 SS 114 CC K11 SS 116		Al ₂ O ₃	0.012 0.014 0.016	NiSiC
CC K11 SS 312 CC K11 SS 314 CC K11 SS 316		Porcelain	0.012 0.014 0.016	

ANALYSIS MATRIX FOR COATING LAYER





Coating1 Stress Plot For K640



COATING THICKNESS (IN.)

1.00 820





(ISA) SSEALS

4-29-97-1111-2

COATING THICKNESS (IN.)



COATING THICKNESS (IN.)



Interface Peel Stress for K640

79

Coating1 Stress Plot for K1100



4-29-97-1111-4



4-29-97-1111-5

81





STRESS (PSI)

4-29-97-1111-6

COATING THICKNESS (IN.)

Appendix B Results of Acoustic Test on Thermal Coating Samples

LOCKHEED MARTIN VOUGHT SYSTEMS CORP

DESIGN INFORMATION REQUEST - RELEASE DRD #640, DR #55-E-601-10C4

PROGRAM/MODEL				DIR NO.			REV	DATE		PAGE	-
Results of Acoustic Tests on Thermal Coating Samples 3-47300/71				DIR- 0	64		9/30/97		1 01 1	3	
PREPARED C. S. Brown (20	date 9/30/97	GROUP	DATE 12	.3-97	REASON	FOR REVISIO	N			REV DA	TE
PROJECT C. K. Reed (1)	DATE	JE Hicz	fas	DATE 12/2/91	APPROVE	D		DATE	APPROVED		DATE
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то			GROU	P NO.	RELEASE	DTO			GROUP NO.		
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C. K. Reed, Joe Wright, R. I	L. Cox, T. I	Ho, C. S. Brown	1, D. F	R. Wright, .	J. B. Mi	niatas, J.	B. Hick	s, J. A. H	utchinson, S	5. E. Sr	nith
SOW PARA REF	······································	WBS R	EF			SYSTEMS S	EGMENT				

DESIGN INFORMATION -

1.0 INTRODUCTION AND SUMMARY

Two acoustic tests were conducted in succession on samples of different thermal coatings according to Test Request 3-56310/97TR-06 (Reference 1) and Test Request 3-56310/97TR-10 (Reference 2). Both of these tests used the same acoustic environments.

The purpose of these tests was to demonstrate the ability of the samples to withstand the prescribed vibro-acoustic environments. Several levels of acoustic excitation were used for the test to encompass the expected vibro-acoustic environments of a broad class of transportation vehicles. In particular, one environment used was the maximum that is expected to be experienced by hardware during transport in the space shuttle. Other environments included two at lower levels for initial evaluation and two at higher levels for more severe testing.

The tests were conducted in the Acoustics Laboratory at the Jefferson Street facility of the Lockheed Martin Vought Systems (LMVS) Environmental Test Laboratory (ETL).

The dates of the tests were August 13 and 14, 1997. Print-outs of data recorded during the tests were given to Engineering on August 19, 1997.

Pre-test inspections were conducted on each test specimen as well as post-test inspections after each phase of both tests by M & T T Engineering. No changes were reported on any of the samples as a result of the acoustic excitation experienced during any phase of either test.

The test data confirm that the articles did indeed experience the proposed acoustic environments during the tests. Post-test analysis of the data recorded during the tests is highlighted in this report, while a more detailed analysis is still in progress.

2.0 THE TEST ARTICLES

The test articles were designed so that several thermal control coating coupons could be tested simultaneously. However, the strains and resonant frequencies experienced by each coupon on the test articles during testing were to be representative of those typically experienced by space radiator hardware during lift-off in a transport vehicle.

Each test article was comprised of an aluminum panel with test coupons bonded to it. The test coupons were substrates, each coated with one of the test coatings. Descriptions of the test articles for the first test, Panels 1 and 2, are given in Table 1 and are further detailed in Reference 1. Similarly, a description of the test article for the second test, Panel 3, is given in the table and is further detailed in Reference 2. The references specify the substrate and coating materials that were used for each specimen and identify the location of each specimen on each of the panels.

Samples of the same test coupons were used on both Panels 1 and 2, but the positions of identical samples on the two panels were different. This scheme was used so that if any damage was incurred to a coupon during the test there would be another sample in a different location on the other panel for comparison.

Panel	Size	Sub	ostrates
ID	(ins)	Total	Size (ins)
1	36 x 24 x 0.08	20	6 x 1
2	36 x 24 x 0.08	20	6 x 1
3	36 x 24 x 0.08	2	6 x 6
		4	6 x 1

 Table 1: Description of Test Articles

For the first test, Panel 1 and Panel 2 were fastened to the test fixture for simultaneous testing. At the completion of this test, Panel 1 was removed from the test fixture and replaced by Panel 3 for the second test. Panel 2 was left attached to test fixture for the second test to preserve the setup of the acoustic environment in the chamber for the test, though it was not instrumented for data collection during this test.

Figure 2.1 shows the configuration of Panels 1 and 2. In addition to the one inch doublers shown in the figure on the upper surface of the panel, two more doublers of the same dimensions were used on the lower surface to allow the panel to attach to the test fixture. Figure 2.2 shows the configuration of Panel 3, which also used the additional doublers to attach to the test fixture. Figure 2.3 (a) shows two panels attached to the test fixture.

For both tests, the test fixture was suspended with two bungees, one on each end, to isolate it from the chamber walls in order to provide uniform pressure levels on all the panel surfaces. This setup is partially depicted in Figure 2.3 (b), which shows the backs of two test panels as they were suspended in the reverberant chamber for testing.

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(a) Attachment to the Test Fixture

(b) Placement in the Reverberant Chamber

Figure 2.3 Test Panels Attached to the Test Fixture

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3.0 TEST CONFIGURATION

Under ideal circumstances each test article should experience a uniform distribution of sound pressure levels on all of its surfaces during the tests. Thus the determination of the final test setup and the method to be used for continuous monitoring were made to provide as much as possible this ideal uniform distribution of the sound pressure levels during the tests.

Additional instrumentation was also used to measure selected responses of the test articles themselves.

3.1 Reverberant Chamber

Figure 3.1.1 is a diagram of the 400 cubic foot reverberant chamber used for these acoustic tests. Also shown in this figure is the approximate location of the test fixture with the test articles attached as it was positioned in the reverberant chamber. This orientation was determined prior to the testing so that the test articles were isolated as much as possible from the chamber itself and had no surface parallel to a wall of the chamber.



Figure 3.1.1 400 ft³ Reverberant Chamber of the Acoustics Laboratory (All dimensions are given in inches)

3.2 Acoustic Excitation

Acoustic excitation was introduced into the reverberant chamber through the opening shown in Figure 3.1.1 for the noise source. This excitation was supplied through the use of two Electro-Pneumatic Transducers (EPT-200s).

3.3 Data Acquisition

Various data were acquired during the tests. The sound pressure field within the reverberant chamber was monitored with microphones, while panel response measurements were obtained with accelerometers and strain gages.

Four microphones were used during the tests to monitor the sound pressure levels at various locations near the test panels. One microphone was positioned on each side of each panel, approximately eleven inches from the panel surfaces. Figure 3.3.1 shows the four microphones setup in the reverberant chamber. In Figure 2.3 (b) two of the microphones can be seen with two test panels in the chamber.

The test panels were instrumented with accelerometers and strain gages. Five accelerometers were used on each test panel, located in the same positions on each, to measure the acceleration environments. In addition four strain gages were used on Panels 1 and 2 and three were used on Panel 3 to measure strains. Figures 3.3.2 (a) and (b) show the accelerometers and strain gages as they were attached to Panels 1 and 2 and Panel 3, respectively. Figure 3.3.3 also presents a view of the panel instrumentation and two of the microphones.





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(a) Panels 1 and 2

(b) Panel 3





Figure 3.3.3 Instrumented Panels in the Reverberant Chamber

4.0 DESCRIPTION OF TESTS

Table 2 summarizes the test levels and durations used for the tests. The overall level and the corresponding spectrum for each phase were based upon typical requirements for hardware that is to be transported in the cargo bay of the space shuttle. Because of the extended duration of the fifth phase, this phase goes well beyond these requirements. This phase is equivalent to a 180 second test at 147 dB-OA, 3 dB up from the actual test level of 144 dB-OA (References 3 and 4). The duration required for Phase 5 was calculated using the slope of the resonant fatigue curves found in Reference 4.

Both Test Requests (References 1 and 2) give specific details of the acoustic environments for each phase of the tests as well as allowed tolerances.

Phase	Environment (Relative to Shuttle)	Overall Sound Pressure Level (dB)*	Duration (sec)
1	Very Low Level (-6 dB)	135	180
2	Low Level (-3 dB)	138	180
3	Shuttle	141	180
4	Severe (+3 dB)	144	180
5	Very Severe (+3 dB, extended duration)	144	1430

 Table 2:
 Test Level and Duration (Both Tests)

*SPL (dB) reference is $2 \times 10^{-3} (N/m^2)$

4.1 Pre-Test Spectrum Equalization

Pre-test spectrum equalizations were performed with an empty reverberant chamber. During this phase of testing the microphones were attached to the data acquisition system and their positioning scheme in the chamber was determined. Acoustic excitation was introduced into the chamber using the four overall pressure levels given in Table 2. At each level, the excitation and spectrum shaper were adjusted to achieve the prescribed spectrum that demonstrated that all of the one-third octave band sound pressure levels as well as overall sound pressure levels were within tolerance.

4.2 Test I (Panels 1 and 2)

After the pre-test spectrum equalizations were completed, the test proceeded to the test on Panels 1 and 2. These panels were connected to the test fixture, instrumented, and positioned in the test chamber. The instrumentation was then connected to the data acquisition system. A pre-test inspection of each thermal coating specimen was performed by M & T T Engineering.

The test then proceeded through the five phases required by the Test Request. At the completion of each phase a visual inspection was conducted by M & T T Engineering, using a 10X glass and lamp, before the test proceeded to the next phase. No delaminations or disbonds of the test specimens were reported from the inspections, and all phases were completed without unexpected time delays or adjustments to the test articles.

4.3 Test II (Panel 3)

The instrumentation on Panels 1 and 2 were disconnected and the test fixture and test articles were removed from the reverberant chamber. Panel 1 was replaced by Panel 3 on the test fixture and instrumented, and the test fixture was again positioned in the test chamber. The instrumentation on Panel 3 was connected to the data acquisition system. As discussed in Section 2.0, Panel 2 was left attached to the test fixture, but the instrumentation on this panel was not connected to the data acquisition system.

The main purpose for leaving the extra panel attached to the test fixture was to assure consistency of the sound pressure levels for both of the tests and to preserve the pre-test spectrum equalizations. However, it also provided an opportunity to subject at least one coupon of each sample to double testing.

A pre-test inspection of each thermal coating specimen on Panel 3 was performed by M & T T Engineering.

The second test then proceeded through the five phases required by the Test Request. At the completion of each phase, a visual inspection was conducted by M & T T Engineering, using a 10X glass and lamp, before the test proceeded to the next phase. No delaminations or disbonds of the test specimens were reported from the inspections, and all phases were completed without unexpected time delays or adjustments to the test articles.

5.0 TEST RESULTS

Data were continuously recorded during both of the acoustic tests. For select times of the tests, printouts were made of the recorded data. This section presents a summary of some of the data from these printouts after they were released to Engineering.

The primary focus of these tests was the screening of several thermal coatings as candidates for further research by subjecting the samples to severe vibro-acoustic environments. An analysis of the microphone data confirms that the proposed environments were present in the test chamber during the tests. Furthermore, data from the individual microphones show a high degree of uniformity of the SPLs recorded by each.

The remaining data were from panel responses obtained during the tests. The analysis of these data is still in progress and is somewhat beyond the scope of the original task. Thus only a portion of this part of the data will be presented, highlighting the results. The panel response data include accelerations and strains. Figures 5.1 and 5.2 designate the accelerometers and strain gages numbers that identify their respective positions on the test panels. Figure 3.3.2 illustrates two of the panels as they were instrumented.

Still to be completed is a comparison of the panel responses of the test articles with similar data of radiator panels currently being manufactured by LMVS. Since the test articles were designed to simulate radiator panels, this comparison would yield useful information for further research efforts.



Figure 5.1 Instrumentation of Panels 1 and 2 Accelerometers: A1-A5; Strain Gages: S1-S4



Figure 5.2 Instrumentation of Panel 3 Accelerometers: A1-A5; Strain Gages: S1-S3

5.1 Microphone Data

Appendix A presents microphone data for both tests. Pages A-1 through A-6 give results for the first test and pages A-7 through A-12 give results for the second test. These plots show that the Overall SPLs for the control microphone (average of the four microphones) were in tolerance for each phase of each test.

5.2 Accelerometer Data

Overall RMS acceleration measurements obtained during Phase 4 of both tests are given in Table 3. For each panel, the first two accelerometers were on the panel doublers and the last three were spread across the centers of the panels as shown in Figures 5.1 and 5.2. The Power Spectral Density (PSD) for each accelerometer is given in Appendix B. Pages B-1 through B-10 contain results for the first test, and B-11 through B-14, results for the second test.

As expected, the data show low responses from the accelerometers on the doublers and higher responses from those on the interior of the panels. The table shows somewhat higher responses on Panel 2 than on Panel 1. However, an examination of the individual PSDs for corresponding accelerometer locations on the two panels shows a high degree of correlation at most frequencies. Most of the differences were concentrated in the frequency range between 100 and 105 Hz. This concentration of differences would indicate some sort of external interference or fluctuations in the sound pressure levels in that particular frequency range causing artificial variations in the responses.

Acceler- ometer	Overall RMS Acceleration						
	Panel 1	Panel 2	Panel 3				
1	5.375	6.214	_*				
2	4.894	4.996	5.155				
3	27.194	30.036	21.463				
4	23.194	30.263	39.709				
5	24.842	29.214	28.271				

Table 3:Acoustic Tests, 144 dB-OA (Phase 4)Overall RMS Accelerations

*Measurement not available

5.3 Strain Gage Data

Strain measurements obtained during Phase 4 of both tests are given in Table 4. The PSDs for the strain gages are given in Appendix C. Pages C-1 through C-8 contain results for the first test, and C-9 through C-11, results for the second tests.

As with the accelerometer data, the table shows that Panel 2 responses overall were somewhat higher than those for Panel 1. It should also be pointed out that, although the first and the fourth strain gages on both Panel 1 and Panel 2 are in symmetric positions on the panels, these substrates are made of different materials. Thus this difference could account for some of the differences in the responses in these two locations.

Strain Gage	Panel 1	Panel 2	Panel 3 ·
1	17.143	15.874	7.720
2	6.202	8.259	11.996
3	4.436	6.685	22.406
4	9.480	10.453	

Table 4:Acoustic Tests, 144 dB-OA (Phase 4)Overall RMS Micro-Strain

6.0 CONCLUSION

The two acoustic tests for the screening of thermal coating samples were completed successfully. Microphone data confirmed that the test environments were equivalent to those proposed for the tests, within the allowable tolerances.

No negative effects were demonstrated by the coupon samples of the thermal coatings that were tested as a result of the tests. Even the severe environments of Phases 4 and 5 of each test caused no degradation or deterioration of the samples. The condition of the samples was monitored with pre-test and post-test inspections as well as inspections during the tests.

Additional data were acquired during the tests which can be utilitized for follow-up work. The test panels were modeled as radiator panels, and panel responses were monitored during the tests. These results were summarized to highlight the results and confirm that the panel response data are consistent with expected results. However, this analysis could be extended to compare the responses of these panels with similar production hardware currently being manufactured by LMVS.

7.0 REFERENCES

- Brown, C. S., "Acoustic Tests of Thermal Coating Samples", TR No. 3-56310/97TR-06, Lockheed Martin Vought Systems, Dallas, Texas, March 20, 1997.
- Brown, C. S., "Second Phase Acoustic Tests of Thermal Coating Samples", TR No. 3-56310/97TR-10, Lockheed Martin Vought Systems, Dallas, Texas, July 22, 1997.
- 3. Military Standard: "Environmental Test Methods and Engineering Guidelines:, Method 515.4, MIL-STD-810E, 14 July 1989.
- 4. Rudder, F. E., Plumblee, Jr., H. E., "Sonic Fatigue Guide for Military Aircraft", AFFDL-TR-74-112, Air Force Flight Dynamics Laboratory, Aero-Acoustics Branch, Wright-Patterson Air Force Base, Ohio, May 1975.

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Appendix A

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Microphone Data


000 Hz dBSPL 138.17 dBS STATUS: PAUSED RMS: 10 10 STOP: ×∵ ы J Pril: L RANGE: THERMAL COATNG 8-40 AUG MICS 1-Y BANDS HHS A: 1/3 OCT RUN#6 1384E RUN 3:00 26-13-97 400 F13 6.3 Hz LEVEL START: (X: RMS | Ţ >¢ 10 dB /DIV JBSPL 170 164 99

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Page A-2





















3-47300/7DIR- 064

Appendix B

Accelerometer Data









PFTA 08/13/97 11:59:18









FFTA 08/13/97 12:01:47





FFTA 08/13/97 12:07:14









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Appendix C

Strain Gage Data





Page C-2









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Page C-8


Page C-9



Page C-10



5-47500/7D1R-004 Page C-11

Appendix C Material Specification for Alumina/ Zirconia/Yttria Powder for Plasma Sprayed Thermal Control Coatings

	ENGINEERING DEPARTMENT	NO. 507-18-411A
LOCKHEED MARTIN	SPECIFICATION	PAGE 1 OF 8
VOUGHT SYSTEMS CORPORATION		DATE 10 June 1998
P.O. BOX 650003	CONTRACT NO ~	RLSE. AUTH. 11030.273
DALLAS, TX 75265-0003	DISTRIBUTION 500	CAGE NO. 64059
	PREPARED BY C.K. Reed	

MATERIAL SPECIFICATION

FOR

ALUMINA/ZIRCONIA/YTTRIA POWDER

FOR

PLASMA SPRAYED THERMAL CONTROL

COATINGS

	LOCK	HEE	D
(VO	UGHT	SY5	TEMS)
	OFF	ICIAL	/
\mathbf{X}	ENGIN	IEERIP	NG /
	REL	EASE	

APPROVALS

PREPARER	THE PROFIMER.	ENG. PROJ. MGR.	QUALITY	MFG. ENG.	TECH. DATA
CKReed	CKROOD	NA	NA	AA	Rebert Tietale
DATE 980609	DATE 960602	DATE	DATE	DATE	DATE 6-10-98

NO. <u>507-18-411A</u>

PAGE ____2

CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION

REVISION PAGE REV DATE **REVISED BY** PAGES AFFECTED REMARKS 07/21/97 ---Initial Issue. Coleman ---06/10/98 Hutchinson 4 -A-Incorporate redlines. VERTICAL BAR IN THE MARGIN INDICATES CHANGE

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NO. <u>507-18-411A</u>

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PAGE

CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION

1. SCOPE

1.1 <u>Scope</u>. This specification establishes the requirements for production of high purity alumina/zirconia/yttria plasma spray powder.

1.2 <u>Responsibilities</u>. The Purchaser shall be responsible for authorizing use of alternate processing materials and equipment.

2. APPLICABLE DOCUMENTS

2.1 <u>Government documents</u>. The following documents, of the issue in effect on date of invitation for bids, or request for proposal, form a part of this specification to the extent specified herein.

OTHER PUBLICATIONS

Regulations

29 CFR 1900 - 1910 Code of Federal Regulations, Part 1910 -Occupational Safety and Health Standards

2.2 <u>Non-Government documents</u>. The following documents, of the issue in effect on date of invitation for bids, or request for proposal, form a part of this specification to the extent specified herein.

STANDARDS

American Society for Testing Materials

ASTM	C573	Standard Method for Chemical Analysis of Fireclay and High Alumina Refractories
ASTM	C958	Standard Test Method Particle Size Distribution of Alumina or Quartz by X-ray Monitoring of Gravity Sedimentation
ASTM	C1069	Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption
ASTM	C1070	Standard Test Method for Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering

OTHER PUBLICATIONS

Lockheed Martin Vought Systems Corporation

3-56420-SOP-7-001	Standard Operating Procedure (SOP) for th	е
	Production of High Purity Alumina/Zirconi	a/
	Yttria Plasma Spray Powder	

4

CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION PAGE

3. REQUIREMENTS

3.1 General material requirements.

3.1.1 <u>Characteristics</u>. The alumina/zirconia/yttria powder shall be characterized as a high purity material in the form of a flowable powder of micron particle size.

3.1.2 <u>Material</u>. The plasma spray powder shall be a blend of 86 weight (wt) percent alumina, 13 wt percent zirconia, and 1 wt percent yttria. The constituent materials for synthesis of the powder shall be those listed in Table I. The powder shall be synthesized by the method described in SOP 3-56420-SOP-7-001.

	TABLE I.	MATERIALS	
Constituent Material	Product ID		Vendor
Aluminum oxide	39814		Alfa Aesar
Zirconium oxide	12732		Alfa Aesar
Yttrium oxide	36274		Alfa Aesar

3.1.3 <u>Quality control material</u>. When specified, the vendor shall supply purchaser with an adequate quantity of component materials for quality control checks.

3.2 Properties.

3.2.1 <u>Particle size distribution</u>. The blended powder shall have a particle distribution as referenced in Table II and be spherical in shape. The particle analysis shall be in accordance with an optical method agreed upon by purchaser and vendor.

3.2.2 <u>Plasma Spraying</u>. Blended powder shall produce acceptable plasma spray coatings. The plasma sprayed coatings shall have a solar absorptance 0.17 maximum and an emittance of 0.77 minimum.

3.3 <u>Quality</u>. The powder shall be thoroughly blended and meet the requirements in Table II. The blended powder shall be uniform in color and quality, dry, and free from foreign materials and imperfections detrimental to its plasma spraying qualities and function as a thermal control coating.

TABLE II. M	ATERIAL REQUIREMENTS	
PROPERTIES	REQUIREMENTS	TEST METHOD PARAGRAPH
Specific Surface Area: Brunauer, Emmett, Teller Method	3.00 m ² /gm maximum	4.3.1
Particle Size Distribution Method 1:	D_{50} equal to 30 μ m maximum	4.3.2
Particle Size Distribution Method 2:	D_{50} equal to 30 μ m maximum	4.3.3
Purity, Trace Chemical Analysis	Trace-element impurities collectively shall not exceed 0.1%	4.3.5
Crystallographic Phase:		
Alpha Aluminum Oxide Zirconiam Oxide	$I_{Alumina}/I_{Zirconiam} = 0.80$ ±0.05	4.3.4

NO. <u>507-18-411A</u>

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3.4 <u>Stability</u>. The material has an unlimited storage life from date of manufacture and shall meet all requirements herein after storage in an unopened sealed container.

3.5 <u>Toxic products and safety</u>. Toxic and hazardous substances listed in 29 CFR 1910.1000 including suspected carcinogenic agents, shall not be used in the formulation of the material. The usage instructions shall include safety and handling criteria.

3.6 <u>Identification and marking</u>. Identification and marking of containers shall be in accordance with Section 5.

3.7 <u>Traceability</u>. Constituent materials used to synthesize the powder will have documentation available as required to provide traceability and demonstrate compliance with quality requirements.

4. QUALITY ASSURANCE PROVISIONS

4.1 <u>Responsibility for inspection</u>. Unless otherwise specified, the Supplier is responsible for the performance of all inspection and testing specified herein. The purchaser has the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure supplies and services conform to the prescribed requirements.

4.2 Acceptance requirements.

4.2.1 Acceptance testing. Acceptance testing shall consist of the tests in 4.3.

4.2.2 <u>Acceptance reports</u>. The Supplier shall furnish with each shipment three copies of a conformance report that shall include:

- (a) Vendor designation.
- (b) Vendor name.
- (c) Lot number.
- (d) Date of manufacture and shipment.
- (e) Net weight.
- (f) Purchase order number.
- (g) Certificate of analysis.

4.3 <u>Test methods</u>. Test methods as compared to physical and chemical property requirements are summarized in Table I. Alternate test methods require an agreement between the Purchaser and Supplier.

4.3.1 <u>Specific surface area</u>. Specific surface area shall be determined in accordance with ASIM C1069. The surface area shall be determined from a measure of gas absorbed on a solid surface as calculated using the Brunauer-Emmett-Teller equation for specific surface area.

4.3.2 <u>Particle size distribution Method 1</u>. Particle size distribution shall be determined in accordance with ASIM C1070 except using the Cilas 1064 Particle Size Analyzer or equivalent.

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4.3.3 <u>Particle size distribution Method 2</u>. Particle size distribution shall be determined in accordance with ASIM C958.

4.3.4 <u>Crystallographic phase</u>. The constituent phases intensity ratio shall be determined by direct comparison X-ray diffraction techniques for Alpha Alumina (113) 2.09 angstroms and Zirconium Oxide (111) 2.93 angstroms.

4.3.5 <u>Trace chemical analysis</u>. The powder shall be tested for trace-element impurities by the method described in ASIM C573 and/or approved spectrochemical techniques or equivalent.

5. PREPARATION FOR DELIVERY

5.1 <u>Preservation and packaging</u>. Preservation and packaging of the material shall be such as to prevent contamination.

5.2 <u>Marking</u>. Each container shall be marked in accordance with, but not be limited to the following:

- (a) Vendor designation.
- (b) Vendor's name.
- (c) Lot number.
- (d) Net weight.
- (e) Contract or purchase order number.
- (f) Specification number and revision letter.
- (g) Shipment date.

6. NOTES

6.1 <u>Intended use</u>. The material covered by this specification is intended to be used for production of plasma sprayable powders to used for thermal control coating on spacecraft.

6.2 <u>Ordering data</u>. Procurement documents should specify, but not be limited to, the following information:

- (a) Title, number, and date of this specification.
- (b) Quantity of material.
- (c) Place of delivery.
- (d) Responsibility for inspection (see 4.1).

6.3 Definitions.

- (a) <u>Storage life</u>. Storage life is the period of time during which packaged material can be stored under specific conditions and remain suitable for use.
- (b) <u>Lot</u>. A lot shall consist of all the material from one cross blend manufactured by the same conditions and processes using the same lots of raw materials.

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10. APPENDIX I

10.1 <u>3-56420-SOP-7-001</u>. The attached is a standard operating procedure for the production of high purity alumina/zirconia/yttria plasma spray powder.

NO. <u>507-18-411A</u>

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PAGE <u>8</u>

APPENDIX I

		SOP NUMBE	R:	REVISION:	DATE ISSUED:	
3-56420-SOP-7-001 5-15-97			5-15-97			
STANDARD C	PPERATING PROCEDURE	CONTRACT/	PROJECT:	<u></u>		
TITLE OF OF	PERATION / TEST:		REFERENCE(S)	:		
Production	Production of high purity					
alumina/zir	conia/yttria plasma spra	y powaer.				
PURPOSE:						
alumina/zir	conia/yttria powder for	plasma spra	ay application	ns.	lty	
NAME OF HAZ	ARDOUS ITEM / MATERIAL(S)	INVOLVED:	LOCATION	N OF WORK TO	BE PERFORMED:	
alumina zirconia yttria polyethyler poly (2-ethyl-2-	(CAS# 1344- (CAS# 1314- (CAS# 1314- (CAS# 1314- (CAS# 25322 (CAS# 25805- oxazoline)	28-1) 23-4) 36-9) -68-3) -17-8)		TBD		
	SEQUENCE	OF OPERATI	ONS (KEY STE	PS)		
(a)	Weigh 86 wt% alumina, 1 (dry weight basis) yttr milling media.	3 wt% (dry ia colloid	weight basis into an alum) zirconia co ina mill jar	olloid, and 1 wt% with alumina	
(d)	Add 2.5 wt% (dry weight wt% (dry weight basis) as Aquazol 50 to the al	basis) of of a poly(: umina/zirce	20M polyethy 2-ethyl-2-oxa onia/yttria p	lene glycol s zoline) binde owder mixture	solution and 0.5 er solution such e.	
(c)	Add D.I. water to bring	the solid	s content to	30 volume %.		
(đ)	Ball mill slurry for 3	hours.				
_ (e)	Spray dry slurry under properties given in 3.2	appropriate •	e conditions	to yield a po	owder with the	
(f)	Dry blend coarse and fi	ne powders	for a minimu	m of 1 hour.	:	
(g)	Sieve powder to less th	an 35 mesh	to remove la	rge agglomera	ites.	
(h)	Calcine powder at 1400°C	for 1 hou	ır in air.			
(i)	Sieve powder to less th	an 35 mesh	to remove la	rge agglomera	tes	
Air-purifyi Safety Data	Air-purifying respirators should be used when dust is present. See appropriate Material Safety Data Sheets (MSDS) for precautions to be taken when handling polyethylene glycol					
Sprav drver	EQUIPMENT / TOOLING REQUIREMENTS					
APPROVAL(S)	:			y meara, JJ II		
DISTRIBUTIC	IN -					
L					·····	

Appendix D Process Specification for Plasma Spraying Thermal Control Coatings

	ENGINEERING DEPARTMENT	NO. 508-17-30
LOCKHEED MARTIN	SPECIFICATION	PAGE 1 of 8
VOUGHT SYSTEMS		DATE: 2 October 1997
CORPORATION	CONTRACT NO. F33615-95-C-5028	RLSE. AUTH. 11030.252
P.O. BOX 650003	DISTRIBUTION 500	CAGE NO. 64059
DALLAS, TX 75265-0003	PREPARED BY C.K. Reed	

PROCESS SPECIFICATION

FOR

PLASMA SPRAYING

THERMAL CONTROL COATINGS

,

LOCKHEED MARTIN
(VOUGHT SYSTEMS)
OFFICIAL
ENGINEERING
RELEASE

APPRUVA	\L				
PREPARER	TECH. PROJ. MGR.	ENG. PROJ. MGR.	QUALITY	MFG. ENG.	TECH. DATA
CKReed	HIM.W.W	RZLor	74	14	Palet fietsely
DATE 9-24-91	DAGE 29.97	DATE 9/25- 51	DATE	DATE	DATE 10-2-97
<u> </u>		,,,,			

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CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION

REVISION PAGE							
REV.	DATE	REVISED	PAGES AFFECTED	REMARKS			
	10/02/97	Coleman		Initial issue.			
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VERTIC	AL BAR IN M	ARGIN INDICATE	S CHANGE.				

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1. SCOPE

1.1 <u>Scope</u>. This specification establishes the requirements for Plasma Spraying Thermal Control Coating for spacecraft applications.

- 1.2 <u>Responsibilities</u>. The Purchaser shall be responsible for:
 - (a) Maintaining and interpreting this specification.
 - (b) Authorizing use of alternate processing materials and equipment.

2. APPLICABLE DOCUMENTS

2.1 <u>Government documents</u>. Unless otherwise specified herein, the following documents, of the issue in effect at the time of use, form a part of this specification to the extent specified herein. In the event of conflict between these documents and this specification, the requirements of this specification shall govern.

SPECIFICATIONS

Federal

TT-I-735 Isopropyl Alcohol

2.2 Non-Government documents. Unless otherwise specified herein, the following documents, of the issue in effect at the time of use, form a part of this specification to the extent specified herein. In the event of conflict between these documents and this specification, the requirements of this specification shall govern.

SPECIFICATIONS

Lockheed Martin Vought Systems Corporation

507-18-411

Material Specification for Alumina/Zirconia/Yttria Powder for Plasma Sprayed Thermal Control Coatings

OTHER PUBLICATIONS

Compressed Gas Association

CGA	G-9.1	Helium
CGA	G-11.1	Argon

CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION PAGE.

3. REQUIREMENTS

3.1 Equipment. The following equipment, or equivalent as approved by the Purchaser, is required for the plasma spray process:

- (a) Torch which dissociates and ionizes a suitable plasma-forming gas when an electric arc is struck between an anode and cathode.
- (b) Any plasma spray power source capable of producing coatings that meet the requirements within.
- (c) Infrared reflectomer, Gier Dunkle, model DB-100.
- (d) Solar reflectomer, AZ Technologies, model LPSR-200-IR, or Gier Dunkle model MS251.

3.2 <u>Materials</u>. The following materials or equivalent as approved by the purchaser are required for the plasma spraying process:

- (a) Gas, argon, as specified in CGA G11.1, Grade C.
- (b) Gas, helium, as specified in CGA G-9.1-1986, Grade L.
- (c) Cloths, commercial Grade A.
- (d) Isopropyl Alcohol (IPA), as specified in TT-I-735.

WARNING

ISOPROPYL ALCOHOL IS FLAMMABLE. Keep away from heat and open flame. Keep container closed. Avoid prolonged breathing of vapors.

- (e) Alumina/zirconia/yttria thermal control plasma spray powder as specified in 507-18-411.
- (f) Aluminum oxide, size 30-60 grit, commercial.

3.3 Required procedures and operations.

3.3.1 Material control.

3.3.1.1 <u>Shielding and purging gases</u>. Shielding and purging gases shall be argon, helium, or a combination of the types per paragraph 3.2. The mixture accuracy shall be 10 percent of the minor component, which shall be verified by the gas distributor.

3.3.1.2 <u>Cleaning</u>. The substrate shall be lightly grit blasted with aluminum oxide at 40 pounds per square inch (psi) maximum and Isopropyl Alcohol (IPA) wiped. Fiber_damage from the grit blasting operation shall be avoided.

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3.3.2 Operator qualification. Operators, or other personnel performing manual plasma spray operations, shall be qualified to spray using each material and gas system designated. Qualification of the manual spray operator, or of fully mechanized equipment, shall be demonstrated by spraying test specimens as defined as follows:

- (a) Each operator is required to plasma spray a 6 inches X 6 inches sample to the thickness specified on the schedule.
- (b) The sample will be tested as per paragraph 4.6 and meet the requirements per paragraph 3.3.4.1

3.3.2.1 <u>Oualification of equipment. materials and processes</u>. The suitability of the equipment, plasma spraying processes, plasma spray powder, and any supplementary treatments selected shall be demonstrated through qualification testing of plasma sprayed specimens representative of production materials and configuration. The plasma spray control system shall provide traceability to the equipment, operator, and the inspector.

3.3.3 <u>Certification of plasma spray schedule and the standard operating</u> <u>procedure</u>. Process Engineering or the Purchaser shall sign for Engineering on the schedule and the SOP. See Figure 1 for a typical schedule. Standard Operating Procedure 3-56410-SOP-7-002 is shown in Figure 2. A schedule and Standard Operating Procedure (SOP) will be required for each powder type and substrate combination. A copy of the schedule and SOP shall be maintained at the equipment.

3.3.4. Certification test specimens.

3.3.4.1. <u>Test specimen requirements</u>. These test specimens shall be visually inspected, have the optical properties measured, have the coating adhesion verified, and be metallographically examined. The requirements per paragraph 4.6 shall be met. The specimen shall be cross sectioned to insure conformance to the engineering requirements. The schedule, SOP, operator or equipment is certified for plasma spraying after the aforementioned tests are successfully completed.

3.3.5 <u>Process control</u>. The plasma sprayed production hardware shall require one process control specimen of the same type, material, size and shape. These process control specimens shall be plasma sprayed prior to production plasma spraying. Process control specimens shall be tested per 4.6.

3.4 <u>Application</u>. Plasma spray the substrate using the parameters established in the schedule and the SOP.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Quality Assurance shall assure compliance with the requirements of this specification by performing the inspections and tests herein. Additionally, Quality Assurance shall maintain adequate surveillance over all facilities, materials, and processes to assure compliance with the requirements and procedures specified herein.

4.2 Monitoring procedures for equipment used in process. Equipment shall be checked and periodically calibrated as determined by Quality Assurance to ensure its accuracy.

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4.3 <u>Monitoring procedures for materials</u>. Materials shall be monitored by the Purchaser to assure Engineering Drawing compliance.

4.4 <u>Certification</u>. All personnel performing inspection operations specified herein shall be certified.

4.5 Inspection.

4.5.1 <u>Prespray and postspray inspection</u>. Sufficient visual inspection, by certified plasma spray inspector, shall be accomplished on parts and assemblies, prior to each plasma spraying operation, to ensure proper cleaning, fit-up, and the use of correct spray material.

4.6 Evaluations.

4.6.1 <u>Visual</u>. The plasma sprayed coating shall be uniform and opaque in appearance. No crazing, cracking or spalling is permitted.

4.6.2 <u>Solar Absorption</u>. The solar absorption of the plasma sprayed coating shall be 0.17 maximum when measured by an LPSR-200-IR.

4.6.3 <u>Emittance</u>. The normal emittance of the plasma sprayed coating shall be 0.77 minimum.

4.6.4 <u>Pull off strength</u>. The pull off strength shall meet the requirements of the Engineering Drawing.

4.6.5 <u>Metallography</u>. The microstructure of the plasma sprayed coating shall reveal uniform scattered porosity of 20-30 percent measured by optical or porosimetry methods.

5. <u>PREPARATION FOR DELIVERY</u>. This section is not applicable to this specification.

6. NOTES

6.1. <u>Operator certification</u>. Documentation of inspection, and test results establishing that an operator or automated machine has produced plasma sprayed coatings which meet the prescribed standards.

6.2. <u>Standard Operating Procedure (SOP)</u>. A document providing the required detailed variables for specific application to assure duplication by properly trained operators, or automated equipment.

		NO. <u>508-17-30</u>
CAGE NO. 64059 ENGINEERING DEPART	MENT SPECIFICATION	PAGE7
· · · · · · · · · · · · · · · · · · ·		
PLASMA SPRA	Y SCHEDULE	
PART NAME Radiator Panel PLASMA SPR	AY COMPANY NAME LMVS)
AREA TO BE COATED Panel exterior surface	۶ <u></u>	
PREPARATION		
Cleaning Light grit blast and IPA wipe		
Blasting Grit: Type <u>AL203</u> Size <u>54</u>	Air Pressure, ps	i (Mpa) <u>40</u>
EOUIPMENT	_	
Manufacture Type <u>Plasmadyne</u>	Plasma System <u>80KW</u>	
Gun Nozzle Electrode_		Powder Port
CONSOLE .		
Primary Gas Argon Console, p	osi (Mpa) <u>80</u> F	low,CFH
Secondary Gas <u>Helium</u> Console, p	osi (Mpa) <u>35</u> F	low,CFH
Amperage, D.C. Operating <u>650</u>		
Voltage D.C., Operating <u>28/34</u>	Voltage D.C.	,Open Circuit
Power Control Kilowatt Level: Start		Finish
Primary Gas Dew Point	Secondary Gas Dew P	Point
POWDER FEEDER		
Carrier Gas_Argon	Flow, CFH_25_psi	
Powder Feed Mechanism Hopper	Powder Feed, RPM (gm/	'hr) <u>2.3</u>
Vibrator:OnOffFeeder	Hose: Diameter	Length
Vibration Amplitude:		
COATING MATERIAL		
Material Identification 507-18-411	Lot	•
Manufacturer	Particle Size Bang	
Spray Rate .002inch/pass Spray	_ Distance 8 inch	,
COATING DATA		
Required Coating Thickness_015 inch	After Spraying	·
Part Dimension: Before Spraying		
Maximum Part Temp		
Spray Time (Per Cycle)	Cool Time (Per Cycl	.e)
Method of Cooling		
Position of Cooling	<u></u>	
WORK HANDLING EOUIPMENT		
Part	Gun_Plasmadyne_SG-1	00
Part Speed	Gun Speed <u>4 inch/se</u>	<u>:C</u>
OUALITY CONTROL		
Solar Absorptance Emittance	Metallography	
Pull Off Strength		
OPERATOR	CERTIFICATION NO.	
APPROVAL		
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NO	508-17-30	
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CAGE NO. 64059 ENGINEERING DEPARTMENT SPECIFICATION PAGE

-	SOP NUMBE	Rı	REVISION:	DATE ISSUED:	
	3-56410-S	OP-7-002		7-28-97	
STANDARD OPERATING PROCEDURE	CONTRACT/ PROJECT:				
TITLE OF OPERATION / TEST:		REFERENCE (S):		
Plasma Spray SOP		507-18-41	1		
PURPOSE :					
This Standard Operating Procedure of control coatings for high thermal of	describes t conductivit	he applicati y substrates	on of plasma : •	sprayed thermal	
NAME OF HAZARDOUS ITEM / MATERIAL (S)	LOCATION OF	WORK TO BE P	ERFORMED:	
Plasma spray powder per 507-18-411 aluminum oxide grit	, IPA,				
SEQUENCE OF OPERATIONS (KEY STEPS)	:	•			
 b. Plasma spray sample as specific. c. Evaluate sample as specific. Approve and certify plasma spray process schedule. Record test data on 4. Clean substrate by light grit i wipe. 5. Attach substrate to holding fix 6. Plasma spray thermal control conspecified in the approved plasma specified in the approved	cified on p ed in 4.6. ay schedule ss control the schedu blast with xture. oating to s ma spray sc	e and SOP. samples as s le. clean alumin substrate in chedule.	schedule. pecified in 3 um oxide # 30 accordance wi	.3.5 and approved -60 grit and IPA th the parameters	
SAFETY REQUIREMENTS:					
Air purifying respirators if manua solvent evaporation.	l operation	is are used.	Adequate ven	tilation for	
EQUIPMENT / TOOLING REQUIREMENTS:					
Plasma spray equipment, Argon and blast chamber.	Helium gas,	vacuum sour	ce, plasma sp	ray booth grit	
APPROVAL(S):					
DISTRIBUTION					
-					

Figure 2. SOP 356410-SOP-7-002

Appendix E Production of High Purity Alumina Plasma Spray Powder

	SOP NUMB	ER:	REVISION:	DATE ISSUED:
	3-56420-SO	P-7-002		
STANDARD OPERATING PROCEDURE	CONTRACT/ PROJECT:			
TITLE OF OPERATION / TEST:		REFERENCE(S)	:	
Production of high purity alumina plasma spray	powder.	3-56420-SOP	-7-001`	
PURPOSE:	du ation a	fhich purity alumi	na nowder for pl	asma sprav
This standard operating procedure describes th applications.	e production t	ngn puncy alum		
NAME OF HAZARDOUS ITEM / MATERIAL(S	5)	LOCATION OF	WORK TO BE P	ERFORMED:
alumina (CAS# 1344-28-1), polyethylene glycol 25322-68-3), poly(2-ethyl-2-oxazoline) (CAS# 2 ammonium polyacrylate solution (CAS# 9003-0 SEQUENCE OF OPERATIONS (KEY STEPS)	(CAS# 25805-17-8), 13-6) :	TBD		
a) Weigh alumina into an alumina mill jar with a	alumina milling	media.		
b) Add 2.5 wt% (dry weight basis) of 20M polya polyacrylate dispersant solution such as Dat binder solution such as Aquazol 50 to the al	ethylene glyco rvan 821A, an umina.	l solution, 2.0 wt% d 0.5 wt% (dry wei	(dry weight bas ght basis) of a p	is) of an ammonium oly(2-ethyl-2-oxazoline)
c) Add D.I. water to bring the solids content to	20 vol%.			
d) Ball mill slurry for 3 hours.				
e) Spray dry slurry under appropriate condition	ns to yield a po	wder with the prop	perties given in 3).2.
f) Dry blend coarse and fine powders for a min	imum of 1 hou	I Г .		
g) Sieve powder to less than 35 mesh to remo	ve large agglo	merates.		
h) Burn out powder at 500°C for 5 hours in air.				
i) Sieve powder to less than 35 mesh to remov	ve large agglor	nerates.		
SAFETY REQUIREMENTS:	dust is present	See appropriate	Material Safety	Data Sheets (MSDS) for
Air-purifying respirators should be used when precautions to be taken when handling polyeth	yiene glycol, a	mmonium polyacr	ylate solution, a	nd poly(2-ethyl-2-
EQUIPMENT / TOOLING REQUIREMENTS:				
Spray dryer, roller mill, alumina mill jar, alumina	a milling media	a, 35 mesh sieve.		
APPROVAL(S):				
DISTRIBUTION				

Appendix F Durability Testing of Plasma Sprayed Coatings

TEST INFORMATION RELEAS	E				
	1			WR NO.	TIR NO. (REV)
			1	97-56410-120	97-56410-042
	C.K. REED	WI-78		PAGE	DATE
DISTRIBUTION				1 OF 7	JUNE 30, 1997
	J.M. WRIGHT	SK-03			
				MODEL	
	DI WUNN	EM-18		TCC	
	D.L. HUNN	<u></u>	WORK COMPLETE IN	APPROVAL A A	
TESTED BY	F.R. MORENO	hm		(JShr	ley
TITLE OF TEST	T	DUR	RABILITY TESTING OF PLAS	SMA SPRAYED COATINGS	6 /
		1		1	

INTRODUCTION

The Thermal Control Coating Program requires durability testing for the plasma sprayed coatings.

Fifteen specimerswere prepared in the High Temperature Materials (HTM) Lab and delivered to the Materials and Processes (M&P) Lab for testing I.a.w. MIL-C-48497A. The specimens were fabricated using carbon-carbon or K1100 as a substrate. A barrier and coating were subsequently added to each specimen. Table I lists the specimens received in the M&P Lab.

OBJECTIVE

Perform various tests i.a.w. MIL-C-48497A, para. 3.4.1, 3.4.2 and 3.4.3. Determine if any flaking, peeling, cracking or blistering is evident. In addition, the coated surfaces should be free of stains, smears, discoloration, streaks, cloudiness, etc.

CONCLUSIONS

1. There were no visible changes to any specimen, except #1358-094.

2. Cracking of the coating on specimen #1358-094 occurred after immersion tests.

PROCEDURE

The specimens were subjected to the tests described in MIL-C-48497A para 3.4.1, 3.4.2, and 3.4.3, and listed in Table II.

RESULTS

1. There were no visible changes during tests 1, 2 and 3; however, when test 4 was performed, cracking of the coating of specimen #94 was visable. Spalling occurred during test 5. The results of the tests are presented in Table II.

- 2. Figures 1, 3, 5, 7 & 9 show the specimens prior to testing.
- 3. Figures 2, 4, 6, 8 & 10 show the specimens after all testing was completed

TABLE	L
-------	---

Specimen #	Substrate	Barrier	Coating
90-6-012	Carbon-Carbon		Al ₂ O ₃
1358-036	K1100	Al	Al ₂ O ₃
1358-044	Carbon-Carbon	Ni-SiC	Al ₂ O ₃
1358-045	Carbon-Carbon		AF
1358-046	Carbon-Carbon	NI-SIC	Blended
1358-050	K1100	Ni-SiC	Al ₂ O ₃
1358-056	Carbon-Carbon		AF
1358-062	Carbon-Carbon		Blended
1358-076	K1100	Ni-SiC	Blended
1358-078	K1100	AI	AF
1358-080	K1100	Ni-SIC	AF
1358-082	K1100	AI	Blended
1358-086	Carbon-Carbon	AI	AF
1358-094	Carbon-Carbon	AI	Al ₂ O ₃
1358-095	Carbon-Carbon	AI	Blended

TABLE II Results of MIL-C-48497A Tests

Specimen Number	120°F ± 4°F and 96-100% Relative Humbity (Test 1)	Moderate Abrasion w/Clean Dry Cheesecloff (Test 2)	-80°F (-82.2°C) and a 160°F (+71°C) for 2 hrs/at Each Temp.* (Test 3)	Imperator in Acetone Wipad w/Chessectorn (Test-4a)	Aconol Wiped W/Cheesecloth	Severe Abrasion w/Eraser***	Immersed in Saine Solution	Immessed in Distilled Water
90-6-012	ok	ok	ok	ok	ok	ok	ok	ok
1358-036	ok	ok	ok	ok	ok	ok	ok	ok
1358-044	ok	ok	ok	ok	ok	ok	ok	ok
1358-045	ok	ok	ok	ok	ok	ok	ok	ok
1358-046	ok	ok	ok	ok	ok	ck	ok	ok
1358-050	ok	ok	ok	ok	ok	ak	ok	ok
1358-056	ok	ok	ok	ok	ak	ok	ok	ok
1358-062	ok	ok	ok	ok	ok	ok	ok	ok
1358-076	ok	Ok	ok	ok	ok	ok	ok	ok
1358-078	ok	ok	ok	ok	ok	ok	ok	ok
1358-080	ok	ok	ok	ok	ok	ok	ok	ok
1358-082	ok	ok	ok	ok	ok	ok	ok	ak
1358-086	ok	ok	ck	ok	ok	ok	ok	ok
1358-094	ok	ok	ok	Some Cracking was	Some Cracking was	Spalling	Spalling	Spalling
1358-095	ok	ok	ok	Evident on the Coating ok	Evident on the Coating ok	occurred ok	occurred ok	occurred ok

*The rate of temperature change did not exceed 4°F per minute.

**Eraser conformed to MIL-E-12397



FIGURE 1. SPECIMENS NOS. 12: 36A & 44 AS RECEIVED.



FIGURE 2. SPECIMENS NOS. 12, 36A & 44 AFTER ALL TESTING WAS COMPLETED. NO VISIBLE SIGNS OF CHANGE.

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FIGURE 3. SPECIMENS NOS. 45; 46 & 50 AS RECEIVED.



FIGURE 4. SPECIMENS NOS. 45; 46 & 50 AFTER ALL TESTING WAS COMPLETED. NO VISIBLE SIGNS OF CHANGE.

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FIGURE 5. SPECIMENS NOS. 56, 62 & 76AS RECEIVED.



FIGURE 6 . SPECIMENS NOS. 56, 62 & 76 AFTER ALL TESTING WAS COMPLETED. NO VISIBLE SIGNS OF CHANGE.



FIGURE 7. SPECIMENS NOS. 78, 80 & 82 AS RECEIVED.



FIGURE 8. SPECIMENS NOS. 78, 80 & 82 AFTER ALL TESTING WAS COMPLETED. NO VISIBLE SIGNS OF CHANGE.

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FIGURE 9. SPECIMENS NOS. 86, 94 & 95 AS RECEIVED.



FIGURE 10. SPECIMENS NOS. 86. 94 & 95 AFTER ALL TESTING WAS COMPLETED. THERE WAS VISIBLE CRACKING SPECIMEN NO. 94 WHICH OCCURRED AFTER IMMERSION TESTS.



TINFORMATION RELEA	C.K. Reed WT-78	J.M. Wrig	ht SK-03	WR NO. None	TIR NO. (REV) 98-56420-007		
	D.L. Hunn EM-16		<u></u>	PAGE 1 OF 12	DATE 7 May 1998		
				PROGRAM TCC			
TESTED BY	D.R. Bryant SK-03	WORK		APPROVAL			
TITLE OF TEST		RE	REVISION OF SPECIFICATION 507-18-411				

INTRODUCTION

An analysis was conducted by High Temperature Materials Lab to verify compliance of alumina/zirconia/yttria powder for a plasma spraying application (TIR 98-56420-004). During this analysis, it was discovered that the powder property values used in the appropriate specification (507-18-411) were incorrect. The reason for the incorrect values, as well as the correct values, needed to be determined.

OBJECTIVE

Determine the cause of the incorrect powder property values in 507-18-411 and determine the correct values.

CONCLUSION

The incorrect powder property values were traced to assays done on the zirconia and yttria colloid constituent materials. The yield values from these assays were found to be in error, causing the relative amounts of constituent materials to be incorrect. The revised powder property values are given in Table 6.

PROCEDURE

- 1. The alumina/zirconia/yttria powder property data used to create 507-18-411 was analyzed, including the batching process and constituent materials used.
- 2. A batch of alumina/zirconia/yttria powder was prepared per SOP 3-56420-SOP-7-001.
- 3. The bulk density of the new powder batch was determined using helium pycnometry.
- 4. Particle size analysis was conducted on the powder per ASTM C958.
- 5. Specific surface area analysis was conducted on the powder.

No. 20 80

- Evaluation of the shapes of the powder particles was conducted using a scanning electron microscope (SEM). Gross chemical composition was determined by using energydispersive X-ray analysis (EDXA) in conjunction with the SEM.
- 7. Analysis of crystallographic phases was conducted with an X-ray diffractometer.
- 8. The powder was plasma sprayed onto 25 carbon/carbon 15/16" diameter discs. Plasma spraying was conducted per 508-17-30.
- 9. The optical properties of the plasma sprayed coatings were measured.

RESULTS AND DISCUSSION

Three batches of alumina/zirconia/yttria powder were used to develop specification 507-18-411. The three batches are labeled by their spray dry identification numbers, SD-189, SD-195, and SD-210. A summary of the powder property values specified by 507-18-411 is given in Table 1. Also included are the powder batches used to determine the powder property values. The powder batch that was used to determine the specific surface area value could not be determined due to lack of information. The constituent material and impurity weight percents were not determined using powder batches. These values were based on the alumina/zirconia/yttria material desired for plasma spraying.

In order to determine if the powder property data used to create 507-18-411 were valid, the batching process used to produce the slurry for spray drying was investigated for the three powder batches. The appropriate amounts of zirconia and yttria colloid were determined through the yield of the colloid. The yield is the weight percentage of solid zirconia or yttria present in the aqueous colloid solution. The zirconia and yttria yields used to calculate the batch amounts for SD-189 were taken from the colloid vendor's information. However, an assay was later done on the colloids and it was discovered that the actual yields were much higher than the data given by the vendors. The yields determined by the assay were used to prepare the batch amounts for SD-195.

New lots of zirconia and yttria colloids were used to produce SD-210. Assays were done on the new colloid lots prior to batching the powder. After SD-210 was produced, another assay was conducted on the zirconia and yttria colloids. It was discovered that the original yield values determined through assaying the colloids did not match the new measured yields. It is believed that the original assays were incorrect because the colloids were not mixed thoroughly prior to a sample of colloid being taken from the lot. The new assays were taken on colloid samples that had been mixed thoroughly. This discovery invalidated the previous assays, causing the relative amounts of material present in the three powder batches to be incorrect. Table 2 lists the colloid yields used to batch the three powder batches, and the correct yield values. The assay done on the lot of colloid used to batch SD-195 cannot be confirmed because there is no colloid left to test. Table 3 lists the actual amounts of constituent materials present in the three powder batches, based on the correct assays.

Due to the incorrect assay values, the powder property values for the three powder batches (SD-189, SD-195, and SD-210) could not be used to revise the specification. In order to determine the correct powder property values, a new batch of alumina/zirconia/yttria was produced.

The new batch of powder was produced per SOP 3-56420-SOP-7-001. This batch is labeled SD-244. The colloids were mixed throroughly with a magnetic stirrer for 1 hour prior to use. Samples of the stirred colloids were taken and assays conducted on them to ensure that the yields used to calculate the batch amounts were correct. These assay values were within experimental error of the yields used to calculate the batch amounts.

The bulk density of SD-244 was determined to be 4.45 g/cm³. This value was used as the powder density in the particle size analysis. The median particle size (D₅₀) was determined to be 13.8 μ m. This value was compared to the median particle sizes of SD-195 and SD-210 (14.8 and 14.3 μ m, respectively), and the average of the three values was taken. The average of the three values is 14.3 μ m. Because the actual amounts of constituent materials present in SD-189 were significantly different than the desired amounts, the median particle size of 15.3 μ m was not used in the determination of the desired particle size for 507-18-411. Although the constituent material amounts in SD-195 and SD-210 are incorrect, the level of error is small enough that median particle size should not be affected significantly. The error range of the median particle size was chosen as \pm 0.9 μ m. This range is based on the variance of median particle size produced by the spray drying process. The revised median particle size for 507-18-411 is therefore 14.3 \pm 0.9 μ m.

The measured specific surface area of SD-244 is 3.08 m²/gm. Since there is not enough surface area data for the alumina/zirconia/yttria powder to choose a statistically determined error range, a range of \pm 0.05 m²/gm was chosen. This range was based on half of the difference between the surface areas of SD-195 and SD-210 (3.09 and 2.09 m²/gm, respectively). The revised specific surface area for 507-18-411 is therefore 3.08 \pm 0.05 m²/gm.

The particles of SD-244 were determined to be spherical and of good quality. Figures 1 and 2 are SEM photographs of the powder. The gross chemical composition of SD-244 is given in Table 4. The EDXA spectra are given in Figures 3 and 4.

The XRD pattern for SD-244 is given in Figure 5. The ratio of the alumina (113) peak to the zirconia (111) peak is 1.37. In order to determine the appropriate error range, a "scattering factor" was calculated to relate the ratio of the alumina/zirconia weight percents to the ratio of the alumina (113) and zirconia (111) peaks. Because the actual weight percents of SD-189, SD-210, and SD-244, as well as the crystallographic ratios, are known, the data from these powder batches were used to determine the scattering factor. An average scattering factor of 5.16 was calculated from the ratios of the weight percent ratios divided by the crystallographic ratios. For example, SD-244 has a weight percent ratio of 6.62 (86.0/13.0), and a crystallographic ratio of 1.37. The ratio of these values gives a scattering factor of 4.83. A weight percent error of \pm 0.5% was chosen, and the corresponding maximum and minimum alumina/zirconia weight percent ratios were determined (i.e. 86.5/12.5 and 85.5/13.5). The scattering factor of 5.16 was used to convert the weight percent ratios to crystallographic ratios. The difference of the resulting crystallographic ratios is 0.12. Half of this value (0.06) was then

taken as the acceptable error in the crystallographic ratios. The revised crystallographic ratio for 507-18-411 is therefore 1.37 ± 0.06 .

The optical property values measured from the plasma sprayed coating produced by SD-244 are given in Table 5. The solar absorptance is 0.17 and the emittance is 0.79. Based on these values and the values given in the thermal control coatings specification (508-17-30), the revised values for 507-18-411 are 0.17 for solar absorptance and 0.78 for emittance.

A summary of the revised specification powder property values are given in Table 6. The original powder property values are included for comparison.
Table 1 Original Powder Property Requirements (per 507-18-411)					
Powder Property	Original Requirements per 507-18-411	Powder Batch			
Specific Surface Area	$3.12 \pm 0.05 \text{ m}^2/\text{gm}$	Not determined			
Median Particle Size (D ₅₀)	2.7 ± 0.9 μm	SD-189			
Trace Element Impurities	< 0.1 wt %	None			
Crystallographic Phase I _{alumina} /I _{zirconia}	0.80 ± 0.05	SD-189			
Constituent Weight Percent					
Alumina Zirconia Yttria	86 13 1	None			
Plasma Spray Properties					
Solar Absorptance Emittance	None				

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Table 2 Colloid Yield Amounts						
Colloid SD-189 SD-195 SD-210						-210
Material	Yield Used for Batch (%)	Actual Yield from Assay (%)	Yield Used for Batch (%)	Actual Yield from Assay (%)	Yield Used for Batch (%)	Actual Yield from Assay (%)
Zirconia	20.00	29.23	29.22	?	30.29	32.79
Yttria	14.00	16.16	16.16	?	22.98	38.38

	Actual Amounts (We	Table 3 of Constituer ight Percent)	t Materials	
Constituent Material	Correct Weight Percent	SD-189	SD-195	SD-210
Alumina	86	80.99	?	84.52
Zirconia	13	17.91	?	13.83
Yttria	1	1.10	?	1.64

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Table 4 Gross Chemical Composition of SD-244				
Element	Weight Percent			
Oxygen	55.28			
Aluminum	37.07			
Zirconium	6.87			
Yttrium	0.78			
Total	100			

Table 5 Optical Properties of SD-244			
Optical Property	Measured Value		
Solar Absorptance	0.17		
Emittance	0.79		

Table 6 Revised Powder Property Requirements						
(per 507-18-411)						
Powder Property Original Requirements Revised Requirements						
Specific Surface Area	$3.12 \pm 0.05 \text{ m}^2/\text{gm}$	$3.08 \pm 0.05 \text{ m}^2/\text{gm}$				
Median Particle Size (D ₅₀)	2.7 ± 0.9 μm	14.3 ± 0.9 μm				
Trace Element Impurities	< 0.1 wt %	< 0.1 wt %				
Crystallographic Phase I _{alumina} /I _{zirconia}	0.80 ± 0.05	1.37 ± 0.06				
Constituent Weight Percent						
Alumina Zirconia Yttria	86 13 1	86 13 1				
Plasma Spray Properties	Plasma Spray Properties					
Solar Absorptance0.15 maximum0.17 maximumEmittance0.80 minimum0.78 minimum						

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FIGURE 1. SEM PHOTOGRAPH OF SD-244. THE PARTICLES ARE SPHERICAL IN SHAPE, WITH MINIMAL IRREGULARITIES.



FIGURE 2. SEM PHOTOGRAPH OF SD-244. THE PARTICLES ARE SPHERICAL IN SHAPE, WITH MINIMAL IRREGULARITIES.



FIGURE 3. EDXA SPECTRA OF SD-244. THE SPECTRA ARE SCALED TO THE HIGHEST ALUMINUM PEAK.

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FIGURE 4. EDXA SPECTRA OF SD-244. THE SPECTRA ARE SCALED TO SHOW THE MISCELLANEOUS SMALLER PEAKS.

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FIGURE 5. X-RAY DIFFRACTION PATTERN OF SD-244. THE ALUMINA PEAKS ARE LABELED 'A' AND THE ZIRCONIA PEAKS ARE LABLED 'Z'. THE ALUMINA(113) AND ZIRCONIA(111) PEAKS ARE ALSO LABELED.



DESIGN INFORMATION REQUEST - RELEASE LOCKHEED MARTIN VOUGHT SYSTEMS

PROGRAM/MODEL	<u></u>	DIR NO.	REV	DATE	PAGE
CRAD - Space Station		3-47300/98DIR-106		981207	1/21
	DATE	REASON FOR REVISION	<u> </u>	<u> </u>	REV DATE
RE Tate COLAVOS CONC	981207				
PROJECT	DATE	GROUP GTZ GIL	2	4-2	DATE
CK Reed CKReed	191201	JB Hicks J FD & 1	ZAD€	<u> </u>	12/7/98
APPROVED	DATE	APPROVED			DATE
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COMPLETE THIS BLOCK FOR INFO REQUE	EST '	COMPLETE THIS	BLOCK F	OR INFO RELEA	SE
то	UNIT	RELEASED TO			UNIT
		CK Reed			3-56400
REQUESTED BY	UNIT	IN REPLY TO			
		3-56310/98TR-012			
REASON		PURPOSE			
		Summarize acoustic test	data on a	a carbon-carb	on
		honeycomb radiator pane	el segme	nt treated with	a thermal
	\rightarrow	coating using a plasma s	bray tech	nique	<u></u>
					····
DISTRIBUTION					
DESIGN INFORMATION				····	
(See attached.)					
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LMVS DIR No. 3-47300/98DIR-106 Page: 2 Of: 21

1.0 INTRODUCTION/SUMMARY.

A carbon-carbon honeycomb radiator panel, whose construction is similar to that of HRS and PVR radiator panels, successfully completed acoustic testing on 12 November 1998. This acoustic panel test evaluated a plasma spray technique for applying thermal coatings to space radiator structures. The selected thermal coating was $Al_2O_3+ZrO_2+Y_2O_3$. This coating was selected based on prior coupon tests performed IAW LMVS TR 3-56310/97TR-06 and LMVS TR 3-56320/97TR-10. The acoustic excitation levels are representative of those experienced by current Space Station hardware. This acoustic test neither damaged (like wear or surface crazing) nor propagated pre-existing damage that would degrade the thermal coating's function as a thermal barrier.

This acoustic test was performed IAW LMVS TR 3-56310/98TR-12 (see Appendix) under 3A98AE 1001.

2.0 TEST OVERVIEW.

The following general information provides an overview of this acoustic test:

Test Article:	One 24" x 28.4" Radiator Panel Section One 30" x 34" x 0.5" picture frame fixture Four zee-stringer restraints
Type of Test:	Acoustic
Test Level & Duration:	135 dB _{OASPL} , 180 seconds (Acceptance –3 dB) 138 dB _{OASPL} , 180 seconds (Acceptance Level) 141 dB _{OASPL} , 180 seconds (Max Flight Level) 144 dB _{OASPL} , 180 seconds (Qual Level) 144 dB _{OASPL} , 23 minutes, 50 seconds (Qual-Endurance)
Location of Test:	LMVS Environmental Test Laboratory (ETL), Acoustic Laboratory, Jefferson Street Facility, Bldg 128, Grand Prairie, TX

3.0 TEST ARTICLE AND TEST SET-UP.

MT&T Engineering provided the coated test panel. Figure 1 thru Figure 5 presents the instrumented test panel mounted to its fixture and suspended within the ETL's small reverberant chamber. Some of the pictures also depict the microphones used to control the acoustic noise exposure. The test article is comprised of a honeycomb panel with carbon-carbon facesheets and is restrained to a picture frame fixture plate using zee-stringers along all four edges. The basic test panel is $24^{\circ} \times 28.4^{\circ}$ comprised of a 24° square carbon-carbon honeycomb panel with a manifold cover assembly attached along one edge. The picture frame is a $30^{\circ} \times 34^{\circ} \times 0.5^{\circ}$ aluminum plate whose test aperture is roughly $22^{\circ} \times 26.2^{\circ}$. This test panel also incorporates several design features typical of space radiator construction such as flow tubes, edge close-out extrusion and manifold attachments.

4.0 TEST RESULTS.

Figure 6 thru Figure 10 present the acoustic environment levels in ascending order (as tested) of presentation in Section 2.0 as measured by the control microphones. For each test level, the measured 1/3 – octave band levels were within tolerance throughout the spectrum frequency range above 31 Hz with minor exceptions. Below 31 Hz, the electro-pnuematic transducer's excitation cannot be reliably controlled; however, the low frequency roll-off is considered acceptable. In all cases, the overall sound pressure level was within specified tolerances.

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Figure 11 and Figure 12 present strain gauge responses at excitation maximum level and during maximum duration. Likewise, Figure 13 thru Figure 18 presents measured accelerations on the panel and the fixture. Taking the difference between the fixture accelerations and panel center, the panel center acceleration is about 10 g's_{ms}. These responses are representative of typical space station radiator structure.

After panel exposure at each test level, MT&T examined the test panel. No crazing, wear or flaking of the coating was observed. Neither was pre-existing damage observed to propagate. Therefore, the thermal coating successfully passed these acoustic tests.

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FIGURE 2 – Test Article Suspended in Reverberant Acoustic Chamber Depicting Microphone Installation



FIGURE 3 – Suspended Test Article (Treated Side)

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FIGURE 4 – Test Article Depicting Control Microphone, Strain Gauge and Accelerometer Installation (Untreated Side)

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FIGURE 5 – Accelerometer and Strain Gauge Installation at Center of Test Article (Untreated Side)



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× 4









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98-11-11 16:19:13 2 9000k 4.890 В Overall RMS = FIGURE 15 – Accelerometer PSD as Measured on Fixture Frame (Qual Level) Meas. ID: Job: Thermal Coating Acoustic Test IIIT TITT ₽ Power Spectral Density ШΠ ШЦ 20.000 Test Date: 11 Nov 98 Program: CRAD 100.00u Log Amp g2/Hz Resolution: 2 Hz 10.000 . Run No: Qual Record No: 196

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FIGURE 17 – Accelerometer PSD as Measured at Panel Center (Qual Level)

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APPENDIX

LMVS TR No 3-56310/98TR-012

TR NO. 3-56310/98-TR- 012

PAGE 1 OF 11

TEST REQUEST

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TITLE			MODEL		G. O. NUMBER			
Acoustic Test of Thermally Coated Radiator Panel Sample			IRAD/CRAD			F33A98AE 1001		
			DATE			REQUESTED	ž T	GROUP
			9809	26'		RE Tate K		3-56310
			DATE DATA	REQ			EST	IMATE
			NLT 98	1029			MAN HOURS	MATL/ODC
DOO JECT TEST ENGINEER		w	ITNESS	YES	NO	SALARY.		
PE Tate								
	ORIG	CU	STOMER			HOURLY	151	\$ 750
UNITASSIGNMENT	GP		0.000			3-56310	747	\$ 500
3-56310	JB Miniatas				X	GTL	• • • •	200
LAB/DEPT RESP FOR TEST	PROJ		LMVS	X		3-56420		Ł
ETL	CK Reed			1		TMTL	22	*254
REFERENCE	LAB Withutetunen		MATER	IAL		1		
	SUPV JA Hutchinson		SEGREGA					
3-56310/97TB-06	PROJ	1 п	YES	- A) NO		173	\$ 754
2.56310/97TP-10	MGR BI Cox			<u> </u>		TOTAL	199	
		L					L	<u> </u>
(See attached)	· · · · · ·							
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	•							
TEST REQUEST DISTRIBUTION			TEST REP	PORT DI	STRIB	JTION		
Signatories plus DR Wright, JB Hicks, SF Smith, Joe Wright		Same as Test Request						

3-56762 R1 (7/94) MW testreq.doc

1.0 INTRODUCTION

The proposed acoustic testing is an extension of testing performed under TR No 3-56310/97TR-06 and TR No 3-56310/97TR-10. Those coupon tests resulted in selecting $Al_2O_3+ZrO_2+Y_2O_3$ thermal coating (hereafter "thermal coating") for possible application on carbon/carbon radiator panels. This acoustic test scales thermal coating technology up to representative radiator panel applications.

2.0 PURPOSE

This TR outlines acoustic testing designed to evaluate a plasma spray technique for a thermal coating application proposed for space radiator structures. The selected thermal coating is $Al_2O_3+ZrO_2+Y_2O_3$. The space radiator structure is a carbon/carbon honeycomb panel constructed like typical space station radiators. This test is conducted to study the structural integrity of the thermal coating during dynamic panel response under acoustic loading. The goal is to precipitate latent durability problems, like wear or crazing, which may degrade the coating's function as a thermal barrier.

3.0 PASS/FAIL CRITERIA

The thermal coating treatment shall be considered viable (pass) if testing activities are completed with acceptable surface crazing or other surface abnormalities indicative of wear or low adhesion strength. Surface flaws resulting from substrate or panel structural problems shall not constitute failure. Loss of measurement transducers (e.g.: strain gauges or accelerometers) shall not constitute failure. MT&T Engineering personnel shall alone disposition local surface damage as to whether further testing may proceed. Otherwise, the test shall be deemed failed.

4.0 TEST OVERVIEW

The following general information provides an overview of these acoustic tests.

Test Article:	One 24" x 28.4" Radiator Panel Section One 30" x 34" x 0.5" picture frame fixture Four zee-stringer restraints
Type of Test:	Acoustic
Test Level & Duration:	135 dB _{OASPL} , 180 seconds 138 dB _{OASPL} , 180 seconds 141 dB _{OASPL} , 180 seconds 144 dB _{OASPL} , 180 seconds 144 dB _{OASPL} , 23 minutes, 50 seconds
Location of Test:	LMVS Environmental Test Laboratory (ETL) Acoustics Laboratory, Jefferson Street Facility, Grand Prairie, TX
Test Schedule:	Fabrication and testing shall be completed NLT 981029

The ETL shall fabricate all fixturing to support the test article within the small reverberant chamber of the Acoustics Laboratory.

5.0 TEST ARTICLE

Figure 1 presents the test article. The test article is comprised of a honeycomb panel with carbon/carbon facesheets and is restrained to a picture frame fixture plate using zee-stringers along all four edges. The basic test panel is $24^{\circ} \times 28.4^{\circ}$ comprised of a 24° square carbon/carbon honeycomb panel with a manifold cover assembly attached along one edge. The picture frame a $30^{\circ} \times 34^{\circ} \times 12^{\circ}$ aluminum plate whose test aperture is $22^{\circ} \times 26.2^{\circ}$ (Figure 2).

Figure 3 thru Figure 5 depict edge restraints. Each edge restraint is designed to capture the edge with minimal binding to prevent scuffing and marking. Silicone elastomer strips (1/8" thicik) also insure uniform contact distribution along each edge. Tedlar tapes prevent chemical interaction between the silicone and thermal coating. Each zee-stringer shall be sized per MEG Design Std 11-13-89 such that the elastomer strips are compressed approximately 3/32" when bolted to the picture frame fixture.

MT&T Engineering shall provide the coated test panel. ETL shall construct attachment fixturing with assistance from M&P Lab and MT&T Engineering personnel.

6.0 ACOUSTIC EXCITATION

Excitation will be introduced into the reverberant chamber through the use of Electro-Pneumatic Transducers (EPTs). Table 1 presents the levels to be used during these tests including tolerances.

7.0 INSTRUMENTATION

Figure 6 presents location of required accelerometer and strain gauge instrumentation. Microphones will be utilized to insure uniform distribution sound pressure field about the test specimen. A minimum of two (2) microphones will be employed. Two microphones will be located near the center of each panel with approximately 18" clearance to the panel. Additional panels may be required. ETL personnel will specify these transducers at time of installation or test. Strain gauges shall be affixed to the test article backside prior to installation in the reverberant chamber. [Scuffing of backside thermal coat down to the carbon/carbon substrate is permitted for installation of strain gauges.] ETL shall provide and install all instrumentation.

8.0 TEST PROCEDURE

8.1 Chamber Equalization

This first phase of the testing will be performed with an empty reverberant chamber and will be used to perform acoustic spectrum equalization and evaluation at each test level. Introduction of the test specimen should result in minimal distortion. Final equalization will occur during the first test.

The procedure to evaluate sound pressure level uniformity is:

- 1. Install measurement microphones described in Section 7.0 and the EPTs.
- 2. Inject acoustic excitation at greater of 123 dB_{OASPL} or minimum controllable level. Adjust the excitation (spectrum shaper, amplifier current, air volume) to achieve the desired spectrum as presented in Table 1. Plot and evaluate the measurement from each of the microphones.
- 3. If the microphone measurements are not favorable after adjusting all excitation variables, reposition the microphones to evaluate a new potential test article location. Repeat step 2 and this step until the desired spectrum is achieved.
- 4. If the results are acceptable, repeat step 2 and step 3 for each each level specified by Table 1 in ascending order of sound pressure level.

Acoustic Test Procedure 8.2

Initial Equalization 8.2.1

Suspend test article within the reverberant chamber at the position determined in Section 8.1. Establish acoustic excitation level at greater of 132 dBOASPL or minimum controllable level to within specified tolerance. Fine tune spectrum levels and shape to minimize acoustic field distortion due to test article presence. Once this spectrum shape is established, this spectrum shape shall remain fixed throughout subsequent test activities.

8.2.2 Acoustic Panel Test

Once the equalized spectrum shape is finalized, the test article shall be exposed to the acoustic environment of Table 1 in ascending order of level and duration.

queater

- Establish acoustic excitation level to tesser of 3 dB or minimum controllable level 1. below the required level of Table 1 within required tolerances.
- Start_all data recorders then proceed to the full level. Test duration begins once 2. Record data for entire duration. test level is stabilized within tolerance. Continuously monitor the spectrum and equalize as required for minor fluctuations.
- Once acoustic exposure is completed, terminate testing and recording. Review 3. test data and inspect article. Record all excitation settings, duration, plotted data and all observed anomalies in the laboratory log book.
- Complete post-test inspection of test article. Document any anomalies in the 4. laboratory logbook. This shall include sketches, photographs and written observations.
- If there exists no degradation of the test article or MT&T Engineering deems 5. degradation to be not critical, then proceed to the next test level and duration.

Post-Test Inspection 8.2.3

Before the test article is removed from the test chamber, MT&T Engineering personnel shall perform a detailed post-test inspection of the test article. Document any anomalies or degradation from initial testing in the laboratory logbook. This shall include sketches, photos of effected regions and written descriptions including disposition rationale.

DATA DELIVERABLES 9.0

The following items shall be delivered by ETL to project engineering within five (5) business days of test completion:

- Photography of test article installed in reverberant chamber including 1. instrumentation and excitation transducers.
- Copy of laboratory logbook. 2.
- Plots of acoustic spectrum from control microphones at the beginning and end of 3. each test. Plots of measurement transducers at the beginning and end of each test.
- Photography of all observed damage or degradation. 4.

SCHEDULE 10.0

All test activities shall be completed NLT 29 October 1998.



ASSEMBLED TEST ARTICLE (FRONT VIEW)

RET/3-56320/980922 FIGURE 1 205
1/3 Octave Band		1/3 Octave Band Sour	nd Pressure Levels Test 3	Test 4	Test 5
Center Frequency	1 851 1 180 car avnosira	180 Sec exposure	180 sec exposure	180 sec exposure	23 min, 50 sec exposure
	Acceptance Level -3 dB)	Acceptance Level	(Max Flight Level)	(Qual Level)	(Qual Level)
ç	103	106	109	112	112
5	106	109	112	115	115
, C.21	107	110	113	116	116
91		112	115	118	118
20	113	116	119	122	122
22	110	122	125	128	128
01.0 04	101	124	127	130	130
5 5	100 5	125.5	128.5	131.5	131.5
00	124	127	130	133	133
88	105	128	131	134	134
B S	105 E	128.5	131.5	134.5	134.5
00 101	105	129	132	135	135
6 <u>7</u> 1	105	129	132	135	135
091 002	105 F	128.5	131.5	134.5	134.5
200	104	197	130	133	133
250	421 403	106	129	132	132
315	123	105	128	131	131
400	221	123	126	129	129
009	118 5	121.5	124.5	127.5	127.5
630	117	120	123	126	126
800	444 5	1175	120.5	123.5	123.5
1000	0.411	110.0	110	122	122
1250	113	011		061	120
1600	111	114		071	118
2000	109	112	115	011	
2500	107	110	113	116	011
OASPL=	135.3	138.3	141.3	144.3	144.3

Band Tolerance=-2dB to +4 dB

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OASPL Tolerance=+/- 1.5 dB

Duration Tolerance= -2%, smaller of 10% or +10 seconds

SPL dB re 2 x 10^{*} (N/m²)

TABLE L



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FILLING 4



France S

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ACCELEREDMETER

- STRAD GALLE







Appendix I EO-1 Spacecraft Carbon-Carbon Radiator

DESIGN INFORMATION REQUEST - RELEASE

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LOCKHEED MARTIN VOUGHT SYSTEMS

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1.0 Purpose

This DIR documents information pertinent to the design/analysis tasks performed for the EO-Carbon-Carbon Radiator⁽⁴⁾ project.

2.0 Structual Description

The general configuration of the radiator is typical of aluminum honeycomb sandwich panels and as such the design/analysis approach employed was characteristic of the same. However, Carbon-Carbon facesheets are atypical of aluminum honeycomb panel construction As a result, special considerations were necessary during the integration design process.

The Radiator consists of two (2) .025 inches thick Carbon-Carbon facesheets bonded to 2.0 P.C.F. aluminum honeycomb (see figure 3). The panel is aproximately 28 inches on a side and 1.0 inch in thickness (see figure 2). The radiator serves as the attach platform for two (2) electronics packages, PSE and LEISIA. These packages are affixed to the panel by means or fasteners common to through holes in the packages and threaded inserts potted into the radiator.

The radiator is attached to the EO-1 spacecraft by means of eighteen (18) fasteners located at the perimeter of the panel. The holes common to the panels were required to be through holes with the attach fasteners treading into attach hardware common to the EO-1 spacecraft proper. Because of the nature of honeycomb core, potted inserts are required at these locations; said inserts to function as the mechanism of load transfer from the spacecraft attach fasteners to radiator.

3.0 Task Description

LMVS was tasked with the following:

- 1. Selecting potted inserts local to the spacecraft attach points.
- 2. Formulating a method of potting all inserts common to the radiator.
- 3. Selecting core type/density.
- 4. Selecting adhesive/primer system for facesheet bonding.

Structural analysis is required to verify the valid resolution of the above items.

4.0 LOADS AND ANALYSIS

4.1 Loads

Loads environments can be divided into two categories:

- 1. Inertia.
 - 2. Thermal.

Inertial loads can be divided further into two categories:

- 1. Flight
- 2. Ground Handling.

Flight acceleration and Ground Handling load levels were supplied by the prime Spacecraft contractor SWALES Aerospace, to the LMVS Loads and Dynamics Group, in the form of an Interface Control Document, SAI-ICD-028 dated September 2 1997⁽⁵⁾.

4.1.1 Inertial Loads

A Finite Element Model (NASTRAN) was subsequently created by LMVS Loads and Dynamics and a numerical representation of the inertial loads environment was applied. Radiator internal loads and interface reactions were thus obtained from the F.E.M. The pertinent output data from the F.E.M. is presented in appendix A.

4.1.2 Thermal Loads

As per section 3.2.3.2 of reference(5) an overall spacecraft thermal load of delta 72° F. is identified as a critical design condition. It was identified by SWALES Aerospace Engineering that as a result of differences in material thermal coefficients of expansion between the EO-1 Spacecraft and the Carbon-Carbon Radiator that relatively high loads could be generated at the interface locations. Analysis/design effort was placed on possible reducing the, and compensating for the effects of 'thermal mismatch' loading. The method employed to reduce the magnitude of this loading condition follows.

Swales Aerospace created a carve-out F.E.M. representing Spacecraft attach structure common to the Carbon-Carbon Radiator. The radiator was also modeled. Both models were joined together rigidly at the proposed interface locations. The modeled thermal environment was applied to the F.E.M. A maximum interface load of 1800 pounds was subsequently derived.

The LMVS Stress analysis group considered this derivation of interface loading overly conservative. Specifically, it is known that the manner in which the Radiator interfaces with the Spacecraft, ie.potted inserts, is not rigid in nature. An insert/potting compound system, as with any finite system, has a characteristic flexibility associated with it. This model simulated the behavior of a insert/potting compund system with dimensions 1.25" X 1.25" X 1.0". In order to reduce the apparent load level generated as a result modeling a rigidly attached Radiator to Spacecraft system, LMVS generated a Finite Element Model (see figure 1) that simulated the effects of potted inserts at the Spacecraft interface locations.

For the sake of expediency, the Spacecraft interface structure would not be modeled. Also because of lack of information, the thermal environment would also not be simulated. LMVS' approach to the problem was to calculate the stiffness of the Radiator and calculate the resultant deflections due to the applied 1800 pound load. The stiffness of the Potted Insert system was then calculated and integrated into the Radiator stiffness previously calculated. Thus a complete system stiffness was obtained. Finally utilizing the deflection initial obtained, the Spacecraft interface load was calculated for the integrated Radiator/Potted Insert stiffness. The value of this load is:

Spacecraft Interface Load: 748[#] (max operating) applied in the plane of the Radiator.

This load is typical of the four (4) corner inserts. The other edge inserts are less highly loaded, but were however design identically to the corner inserts.

4.2 Analysis

It was LMVS' responsibility to confirm the structural integrity of the Carbon-Carbon Radiator assembly. The analysis tasks were broken down as follows:

1. Analysis of Potting Compound at all insert locations.

- 2. Honeycomb core analysis.
- 3. Facesheet/Core interface analysis.

The loads used in these analyses is resolved into panel maximum resultant in-plane and panel normal components.

There are three (3) distinct type/locations for the potted inserts common to the Radiator:

- 1. Inserts local to the Spacecraft interface points.
- 2. Threaded inserts local to the affixed electronics packages.
- 3. Threaded inserts local to the GSE interface points.

Because of the nature of the Insert / Potting Compound system, in-plane load carried by the Radiator is distributed directly to the Carbon_Carbon facesheets. Therefor, the honeycomb core is analyzed with no load applied in the plane parallel to that of the facesheets.

The component of load normal to the facesheets must in part be carried by the core. A conservative assumption was made in that all normal load is carried as vertical shear in the core.

4.2.1 Inserts local to the Spacecraft interface points

The maximum in plane load is a result of the thermally induced mismatch load. The maximum out of plane load is inertial induced.

 $P_{\text{in-plane}} = 748^{\#}$ (max operating). $P_{\text{normal}} = 171^{\#}$ (max operating).

The in-plane thermal loads are present during the on orbit condition, while the normal component is generated during launch. The loads are therefor mutually exclusive and are applied as separate conditions.

4.2.1.1 In-plane

Hand and F.E.M. analysis were used to derive stresses present in the potting compound employed to affix the inserts to the Radiators. A margin of safety was calculated for both types of analysis and the lesser of the two used as the valid result.

The hand analysis utilized simple uniaxial loading and potting compound tensile allowables⁽¹⁾. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = +.43 (ULTIMATE).

Internal loads were obtained from the same model used in the derivation of the Potted Insert stiffness derivation. The stress analysis was performed using techniques contained with in reference (3), Section 6.4.1.2 and Interaction Curves Figure 6-9. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this condition is:

M.S. = +.14 (ULTIMATE).

Because the latter analysis obtained the minimum of the two, this value will be used as the final margin.

A margin was also calculated for the Potting Compound / Facesheet interface shear analysis. The was assumed to be distributed over an area equal to the contact area of the potting compound to facesheet. A conservative assumption was made in that the load was assumed to be distributed over only a single facesheet. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this condition is:

M.S. = +2.76 (ULTIMATE).

4.2.1.2 Normal Component

The analysis for the normal component of load for those inserts local to the electronics packages are of more sever condition.

This potting compound analysis was performed as a simple shear out hand analysis utilizing potting compound shear allowables⁽¹⁾. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = + LARGE (ULTIMATE).

4.2.2 Threaded Inserts Local to Affixed Electronics Packages

Data provided by the LMVS Loads and DynamicsGroup (appendix A) indicates that two (2) locations local to the PSE packages have the peek attachment loads (see figure 2). The resultant load due to inertia has a value of:

 $P_{in-plane} = 86^{*}$ (max operating). $P_{normal} = 280^{*}$ (max operating).

4.2.2.1 <u>In-plane</u>

The magnitude of in-plane load is substantially less than that of the Thermally induce Spacecraft interface load. Because the geometry of the insert system used at this location is similar to that of the Spacecraft interface locations, the Spacecraft Interface location in-plane analysis is more severe of the two. Therefor an analysis for Potting compound local material failure due to this loading condition was not required.

4.2.2.2 Normal Component

The normal component is the peek value for all locations. This analysis was performed as a simple shear out hand analysis and potting compound shear allowables⁽¹⁾. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = + LARGE (ULTIMATE).

The honeycomb core adjacent to the location in question was analyzed for shearout. The applied normal load was carried over an area subtended by a cylinder of radius indicated on the interface radiator interface drawing and of height equal to the core thickness. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = +.00 (ULTIMATE).

The normal load induces a 'resisting shear' of the form VO/I between the Carbon-Carbon Facesheets and the Honeycomb Core. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = +.02 (ULTIMATE).

Because of the very large relative difference in magnitudes of applied and allowable⁽²⁾ shear stresses the Ultimate Margin of Safety is considered to be a positive large value for the core/facesheet adhesive system.

4.2.3 Threaded Inserts Local to the GSE Interface Points

4.2.3.1 In-plane

In-plane loads were minimal compared to that of other locations.

4.2.3.2 Normal Component

Loads normal to the radiator at these locations have a maximum value of:

 $P_{normal} = 154^{\#}$ (max operating).

As previous analysis has shown that a similar geometry and greater load has a positive Margin of Safety for Potting compound allowables, no analysis is required. However, this load will be subsequently used for core analysis.

The honeycomb core adjacent to the location in question was analyzed for shear-out. The applied normal load was carried over an area subtended by a cylinder of radius indicated on the interface radiator interface drawing and of height equal to the core thickness. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = +.48 (ULTIMATE).

The normal load induces a 'resisting shear' of the form VO/I between the Carbon-Carbon Facesheets and the Honeycomb Core. Utilizing the appropriate Factor of Safety, the Ultimate Margin of Safety for this method and condition is:

M.S. = +.52 (ULTIMATE).

REFERENCES

- 1. EA9318, 9319 and 934NA Tensile Shear and Bulk Poperty Data. Rockwell International, Columbus A/C Division
- Evaluation of EA9689/EA9205R Adhesive System, DIR#3047300/1DIR-004 LTV Missiles and Electronics, Missiles Division
- 3. LTV Structures Manual
- Technical Proposal, Thermal Control Coating for High Thermal Conductivity (k) Substrates, Report No. 3-47300/7R-020, dated 13 October 1997
- 5. Interface Control Document, SWALES Aerospace, SAI-ICD-028, dated November 2, 1997

POTTING CONTRUND / INSERT MODEL DESCRIPTION

MOORL NAME Trewis/C-C/insert/insert3. bdf

TILORE 1





FIGURE 3

E0-I CARBON-CARBON RADIATOR CONSTRUCTION (TYPICAL)



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	E ₁	E2 Lan	G12 400	3013 C13	G23 445	21.405
Γ	1.61E+07	1.61E+07	6.11E+06	3.52E+05	3.52E+05	0.32

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1 025.05	4 10E+04	0.499	6177	3526



APPENDIX A

<u>Flight and Ground Handling</u> <u>Load Analysis</u> FLIGHT AND GROUND HANDLING LOAD ANALYSES

FINITE ELEMENT MODLE FOR THESE ANALYSES SHOWN IN FIGURE 1 ٥,

O LOAD FACTORS (REFERENCE 1)

15 G (LIMIT), IN ANY DIRECTION FOR FLIGHT

5 G (ULTIMATE), IN ANY DIRECTION FOR GROUND HANDLING

O LOAD RESULTS SUMMARIZED IN TABLES I, II AND III

Reference 1: "EO-1 Space to Carbon-Carbon Radiator Interface Control Document", SAI-ICD-028, Swales Aerospace, september2, 1997

R FINITE ELEMENT MODEL	·	MODEL STATISTICS: 5010 DEGREE OF FREEDOM 783 QUADRILATERAL PLATE ELEMENTS 54 ELASTIC SPRING ELEMENTS	MODEL WEIGHT SUMMARY: Weight : 60.24 lb Xcg : 14.85 in Ycg : 11.15 in Zcg : -5.17 in	S USED IN THIS ANALYSIS	= 6.114E+6 psi, = 0.3223 LYSIS	• • • •	ET 1266, 1267, 1694, D ANALYSIS
FIGURE CARBON-CARBON RADIATO		10 1435 1435		0 MID MID MID MID MID MID MID	El = 1.617E+7 psi, E2 = 1.617E+7 psi, G12 G13 = 3.516E+6 psi, G23 = 3.516E+6 psi, v12 o EDGE INSERT STIFFNESSES FOR FLIGHT LOAD ANA	IN-PLANE STIFFNESS : 126000 1b/in OUT-PLANE STIFFNESS : 65900 1b/in o NON-STRUCTURAL WEIGHTS	PSE BOX : 44.09 lb WITH 5.31 inch OFFS o SINGLE POINT CONSTRIAN AT GRIDS 1244, 1245, 1695, 1716 AND 1717 FOR GROUND HANDLING LOA

TABLE I

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EDGE INSERT ATTACHMENT FLIGHT LIMIT LOADS (15 G FLIGHT, UNIT: LBF)

INSERT NO.		X-FLIGHT		Å	-FLIGHT		Z-	FLIGHT	
	FX	FΥ	FZ	FX	FУ	FZ	FX	FУ	FZ
, T	4.79E+01	1.40E+01	-6.97E+00	1.47E+01	3.91E+01	-1.72E+01	-4.40E-13	4.86E-13	-2.84E+01
2	6.00E+01	3.58E+00	-7.17E+01	9.54E+00	6.08E+01	-4.06E+01	-6.95E-13	7.77E-13	7.50E+01
£	6.65E+01	-1.33E-01	-3.31E+01	1.73E+00	6.68E+01	-6.53E+01	-9.76E-13	7.10E-13	8.42E+01
4	6.65E+01	8.81E-01	3.35E+01	1.23E+00	6.75E+01	-6.68E+01	-1.02E-12	2.54E-13	9.13E+01
ŝ	5.99E+01	-2.92E+00	7.14E+01	-7.11E+00	6.26E+01	-4.38E+01	-8.21E-13	-7.19E-14	9.02E+01
و	4.77E+01	-1.37E+01	8.08E+00	-1.32E+01	4.09E+01	-1.75E+01	-6.05E-13	-2.87E-14	-2.68E+01
7	6.41E+01	3.38E+00	-4.82E+01	1.47E+00	4.86E+01	1.40E+01	-5.72E-13	8.07E-13	7.98E+01
æ	6.43E+01	-3.12E+00	4.59E+01	-4.03E-01	5.20E+01	1.22E+01	-5.58E-13	-2.15E-13	8.80E+01
6	5.98E+01	-8.72E+00	-3.57E+01	-3.75E+00	4.71E+01	5.35E+01	-4.28E-13	7.58E-13	1.13E+0 2
10	6.16E+01	8.39E+00	3.36E+01	2.59E+00	5.21E+01	5.04E+01	-3.34E-13	-1.89E-13	1.27E+02
11	4.37E+01	-1.53E+01	-8.96E+00	-6.52E+00	4.08E+01	2.13E+01	-2.49E-13	5.84E-13	4.22E+01
12	4.83E+01	1.51E+01	1.52E+01	3.57E+00	4.79E+01	2.03E+01	-1.87E-13	-1.32E-13	5.39E+01
13	2.66E+01	-1.65E+01	6.19E+00	-1.21E+01	3.12E+01	-1.52E+01	-1.31E-13	4.25E-13	-3.42E+0
14	3.29E+01	-8.50E+00	-1.04E+01	-9.91E+00	4.22E+01	1.06E+01	-9.13E-14	4.00E-13	2.22E+0
15	3.97E+01	-5.03E+00	-3.93E+01	-5.47E+00	5.22E+01	3.69E+01	-7.85E-14	2.94E-13	6.50E+0
16	4.11E+01	1.40E+00	-1.67E+01	3.16E+00	5.65E+01	4.07E+01	-1.19E-13	3.55E-14	6.94E+0
17	4.08E+01	1.06E+01	5.06E+01	7.77E+00	5.93E+01	2.00E+01	-1.70E-13	-5.46E-14	2.81E+0
18	3.31E+01	1.66E+01	6.55E+00	1.27E+01	3.68E+01	-1.32E+01	-1.07E-13	-7.64E-14	-3.61E+0

TABLE II

PSE AND LEISA/AC BOXES INSERT ATTACHMENT FLIGHT LIMIT LOADS (15 G FLIGHT, UNIT: LBF)

INSERT NO.		X-FLIGHT		•	Y-FLIGHT		-2	-FLIGHT	
	FX	FΥ	FZ	FX	FY	FZ	FX	FУ	Fz
1102	-7.56E+01	-2.79E+00	1.28E+01	8.72E+00	-5.94E+01	1.10E+02	1.75E-12	-1.70E-13	-4.14E+01
1109	-7.55E+01	2.13E+00	-1.52E+01	-1.19E+01	-6.01E+01	1.11E+02	1.61E-12	-1.08E-12	-4.47E+01
1127	-9.22E+01	1.39E+00	1.40E+02	2.26E+01	-8.55E+01	7.08E+01	1.88E-12	-2.82E-12	-1.15E+02
1144	-9.21E+01	-2.69E+00	-1.39E+02	-2.67E+01	-8.71E+01	7.70E+01	1.88E-12	-3.83E-13	-1.45E+02
1247	-4.91E+01	-6.15E+00	1.96E+01	1.83E+00	-6.37E+01	-4.71E+00	-2.34E-13	-2.77E-12	4.47E+01
1264	-4.70E+01	5.96E+00	-1.91E+01	-1.69E+00	-6.29E+01	-3.08E+00	4.43E-13	-5.84E-14	3.44E+01
1397	-6.77E+01	-2.32E+01	9.73E+01	-5.35E+00	-8.27E+01	-1.33E+02	-6.71E-13	-2.87E-12	-2.82E+02
1414	-5.78E+01	3.00E+01	-7.46E+01	5.71E+00	-7.34E+01	-1.39E+02	-5.80E-13	-1.25E-13	-2.53E+02
1432	-5.90E+01	-9.15E+00	-4.12E+01	5.41E+00	-4.55E+01	-4.16E+01	-1.33E-12	-2.55E-12	5.41E+01
1439	-4.57E+01	4.49E+00	2.03E+01	1.38E+00	-4.16E+01	-4.84E+01	-1,31E-12	-9.91E-13	8.51E+01
1495	-8.50E+00	3.42E+01	1.67E+01	1.13E+00	-5.81E+00	6.03E+01	1.16E-12	-2.16E-13	-1.54E+00
1504	-3.45E+01	-3.86E+01	-1.67E+01	9.92E+00	-1.56E+01	1.81E+01	1.64E-12	-7.74E-14	-8.12E+01
1765	-5.94E+01	2.02E+01	9.07E+01	-2.52E+01	-6.17E+01	-6.03E+01	-2.09E-13	-4.06E-13	-8.11E+01
1774	-6.31E+01	-1.59E+01	-9.07E+01	1.42E+01	-8.24E+01	-1.81E+01	1.44E-13	-5.24E-13	-1.65E+00

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TABLE III

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GROUND SUPPORTED EQUIPMENT ATTACHMENT (GSEA) ULITMATE LOADS (5 G FLIGHT, UNIT: LBF)

GSEA NO.)-X	GROUND-HAND	DNIT	УС	SROUND-HAND	DNID	2-	GROUND-HANI	DVIJO
-	FX	FY	БZ	FX	FΥ	FZ	FX	FΥ	FZ
1244	-2.95E+01	6.29E-01	2.81E+00	6.98E-01	-2.72E+01	-3.43E-01	-8.15E-15	1.38E-13	1.20E-01
1245	-4.68E+01	-6.70E-01	5.61E+01	-2.54E-01	-4.41E+01	-2.99E+01	1.16E-14	2.20E-13	-1.12E+02
1266	-4.72E+01	2.65E-01	-5.04E+01	6.61E-01	-4.58E+01	-2.55E+01	2.13E-14	2.28E-13	-1.18E+02
1267	-2.98E+01	-5.58E-01	-2.93E+00	-2.60E-01	-2.90E+01	-3.06E-01	1.16E-14	1.67E-13	5.80E-02
1694	-1.01E+01	5.91E+00	-1.95E+00	5.00E+00	-1.15E+01	3.82E+00	-2.84E-14	5.50E-14	4.79E+00
1695	-1.34E+01	3,91E+00	7.91E+00	3.44E+00	-1.70E+01	-1.56E+01	-2.26E-14	8.78E-14	-2.18E+01
1716	-2.07E+01	-3.82E+00	-1.51E+01	-1.88E+00	-2.84E+01	-1.98E+01	-4.31E-14	3.41E-13	-3.28E+01
1717	-1.45E+01	-5.66E+00	8.70E-02	-5.34E+00	-1.50E+01	4.23E+00	2.09E-14	1.65E-13	5.42E+0(

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Appendix J Flight and Ground Handling Load Analysis

FLIGHT AND GROUND HANDLING LOAD ANALYSES

FINITE ELEMENT MODLE FOR THESE ANALYSES SHOWN IN FIGURE 1 ο

o LOAD FACTORS (REFERENCE 1)

15 G (LIMIT), IN ANY DIRECTION FOR FLIGHT

5 G (ULTIMATE), IN ANY DIRECTION FOR GROUND HANDLING

O LOAD RESULTS SUMMARIZED IN TABLES I, II AND III

Reference 1: "EO-1 Space to Carbon-Carbon Radiator Interface Control Document", SAI-ICD-028, Swales Aerospace, september2, 1997

R FINITE ELEMENT MODEL	MODEL STATISTICS:	5010 DEGREE OF FREEDOM 783 QUADRILATERAL PLATE ELEMENTS 54 ELASTIC SPRING ELEMENTS	MODEL WEIGHT SUMMARY:	Weight : 60.24 lb Xcg : 14.85 in Ycg : 11.15 in Zcg : -5.17 in	S USED IN THIS ANALYSIS	= 6.114E+6 psi, = 0.3223	SISAT	- - -		3ET	, 1266, 1267, 1694, AD ANALYSIS
CARBON-CARBON RADIATO		R 10		12441 241 1241 1266 1266 1266 1266 1266	O MRD MECHANICAL PROPERTIES FOR C-C FACESHEETS	El = 1.617E+7 psi, E2 = 1.617E+7 psi, G12 G13 = 3.516E+6 psi, G23 = 3.516E+6 psi, v12	O EDGE INSERT STIFFNESSES FOR FLIGHT LOAD ANA	IN-PLANE STIFFNESS : 126000 lb/in OUT-PLANE STIFFNESS : 65900 lb/in	O NON-STRUCTURAL WEIGHTS	PSE BOX : 44.09 lb WITH 5.31 inch OFFS	o SINGLE POINT CONSTRIAN AT GRIDS 1244, 1245, 1695, 1716 AND 1717 FOR GROUND HANDLING LOA

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TABLE I

EDGE INSERT ATTACHMENT FLIGHT LIMIT LOADS (15 G FLIGHT, UNIT: LBF)

INSERT NO.		X-FLIGHT		×	-FLIGHT		[-2	FLIGHT	
	FX	Fy	FZ	FX	FΥ	FZ	FX	FУ	E E
	4.79E+01	1.40E+01	-6.97E+00	1.47E+01	3.91E+01	-1.72E+01	-4.40E-13	4.86E-13	-2.84E+01
7	6.00E+01	3.58E+00	-7.17E+01	9.54E+00	6.08E+01	-4.06E+01	-6.95E-13	7.77E-13	7.50E+01
e	6.65E+01	-1.33E-01	-3.31E+01	1.73E+00	6.68E+01	-6.53E+01	-9.76E-13	7.10E-13	8.42E+01
4	6.65E+01	8.81E-01	3.35E+01	1.23E+00	6.75E+01	-6.68E+01	-1.02E-12	2.54E-13	9.13E+01
ŝ	5.99E+01	-2.92E+00	7.14E+01	-7.11E+00	6.26E+01	-4.38E+01	-8.21E-13	-7.19E-14	9.02E+01
9	4.77E+01	-1.37E+01	8.08E+00	-1.32E+01	4.09E+01	-1.75E+01	-6.05E-13	-2.87E-14	-2.68E+01
6	6.41E+01	3.38E+00	-4.82E+01	1.47E+00	4.86E+01	1.40E+01	-5.72E-13	8.07E-13	7.98E+01
8	6.43E+01	-3.12E+00	4.59E+01	-4.03E-01	5.20E+01	1.22E+01	-5.58E-13	-2.15E-13	8.80E+01
6	5.98E+01	-8.72E+00	-3.57E+01	-3.75E+00	4.71E+01	5.35E+01	-4.28E-13	7.58E-13	1.13E+02
10	6.16E+01	8.39E+00	3.36E+01	2.59E+00	5.21E+01	5.04E+01	-3.34E-13	-1.89E-13	1.27E+02
11	4.37E+01	-1.53E+01	-8.96E+00	-6.52E+00	4.08E+01	2.13E+01	-2.49E-13	5.84E-13	4.22E+01
12	4.83E+01	1.51E+01	1.52E+01	3.57E+00	4.79E+01	2.03E+01	-1.87E-13	-1.32E-13	5.39E+01
13	2.66E+01	-1.65E+01	6.19E+00	-1.21E+01	3.12E+01	-1.52E+01	-1.31E-13	4.25E-13	-3.42E+01
14	3.29E+01	-8.50E+00	-1.04E+01	-9.91E+00	4.22E+01	1.06E+01	-9.13E-14	4.00E-13	2.22E+01
15	3.97E+01	-5.03E+00	-3.93E+01	-5.47E+00	5.22E+01	3.69E+01	-7.85E-14	2.94E-13	6.50E+01
16	4.11E+01	1.40E+00	-1.67E+01	3.16E+00	5.65E+01	4.07E+01	-1.19E-13	3.55E-14	6.94E+01
17	4.08E+01	1.06E+01	5.06E+01	7.77E+00	5.93E+01	2.00E+01	-1.70E-13	-5.46E-14	2.81E+01
18	3.31E+01	1.66E+01	6.55E+00	1.27E+01	3.68E+01	-1.32E+01	-1.07E-13	-7.64E-14	-3.61E+01

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TABLE II

PSE AND LEISA/AC BOXES INSERT ATTACHMENT FLIGHT LIMIT LOADS (15 G FLIGHT, UNIT: LBF)

INSERT NO.		X-FLIGHT			Y-FLIGHT		Ŕ	-FLIGHT	
	Fx	FУ	ы И И	FX	FY	F	FX	FΥ	FZ
1102	-7.56E+01	-2.79E+00	1.28E+01	8.72E+00	-5.94E+01	1.10E+02	1.75E-12	-1.70E-13	-4.14E+01
1109	-7.55E+01	2.13E+00	-1.52E+01	-1.19E+01	-6.01E+01	1.11E+02	1.61E-12	-1.08E-12	-4.47E+01
1127	-9.22E+01	1.39E+00	1.40E+02	2.26E+01	-8.55E+01	7.08E+01	1.88E-12	-2.82E-12	-1.15E+02
1144	-9.21E+01	-2.69E+00	-1.39E+02	-2.67E+01	-8.71E+01	7.70E+01	1.88E-12	-3.83E-13	-1.45E+02
1247	-4.91E+01	-6.15E+00	1.96E+01	1.83E+00	-6.37E+01	-4.71E+00	-2.34E-13	-2.77E-12	4.47E+01
1264	-4.70E+01	5.96E+00	-1.91E+01	-1.69E+00	-6.29E+01	-3.08E+00	4.43E-13	-5.84E-14	3.44E+01
1397	-6.77E+01	-2.32E+01	9.73E+01	-5.35E+00	-8.27E+01	-1.33E+02	-6.71E-13	-2.87E-12	-2.82E+02
1414	-5.78E+01	3.00E+01	-7.46E+01	5.71E+00	-7.34E+01	-1.39E+02	-5.80E-13	-1.25E-13	-2.53E+02
- 1432	-5.90E+01	-9.15E+00	-4.12E+01	5.41E+00	-4.55E+01	-4.16E+01	-1.338-12	-2.55E-12	5.41E+01
1439	-4.57E+01	4.49E+00	2.03E+01	1.38E+00	-4.16E+01	-4.84E+01	-1,31E-12	-9.91E-13	8.51E+01
1495	-8.50E+00	3.42E+01	1.67E+01	1.13E+00	-5.81E+00	6.03E+01	1.16E-12	-2.16E-13	-1.54E+00
1504	-3.45E+01	-3.86E+01	-1.67E+01	9.92E+00	-1.56E+01	1.81E+01	1.64E-12	-7.74E-14	-8.12E+01
1765	-5.94E+01	2.02E+01	9.07E+01	-2.52E+01	-6.17E+01	-6.03E+01	-2.09E-13	-4.06E-13	-8.11E+01
1774	-6.31E+01	-1.59E+01	-9.07E+01	1.42E+01	-8.24E+01	-1.81E+01	1.44E-13	-5.24E-13	-1.65E+00

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TABLE III

GROUND SUPPORTED EQUIPMENT ATTACHMENT (GSEA) ULITMATE LOADS (5 G FLIGHT, UNIT: LBF)

GSEA NO.	X -X	GROUND-HANDI	DNIT	у-ү	ROUND-HANDI	CING	2	GROUND-HANI	DIING
	FX	FΥ	FZ	FX	FΥ	FZ	FX	FY	FZ F
1244	-2.95E+01	6.29E-01	2.81E+00	6.98E-01	-2.72E+01	-3.43E-01	-8.15E-15	1.38E-13	1.20E-01
1245	-4.68E+01	-6.70E-01	5.61E+01	-2.54E-01	-4.41E+01	-2.99E+01	1.16E-14	2.20E-13	-1.12E+02
1266	-4.72E+01	2.65E-01	-5.04E+01	6.61E-01	-4.58E+01	-2.55E+01	2.13E-14	2.28E-13	-1.18E+02
1267	-2.98E+01	-5.58E-01	-2.93E+00	-2.60E-01	-2.906+01	-3.06E-01	1.16E-14	1.67E-13	5.80E-02
1694	-1.01E+01	5.91E+00	-1.95E+00	5.00E+00	-1.15E+01	3.82E+00	-2.84E-14	5.50E-14	4.79E+00
- 1695	-1.34E+01	3.91E+00	7.91E+00	3.44E+00	-1.70E+01	-1.56E+01	-2.26E-14	8.78E-14	-2.18E+01
1716	-2.07E+01	-3.82E+00	-1.51E+01	-1.88E+00	-2.84E+01	-1.98E+01	-4.31E-14	3.41E-13	-3.28E+01
1717	-1.45E+01	-5.66E+00	8.70E-02	-5.34E+00	-1.50E+01	4.23E+00	2,09E-14	1.65E-13	5.42E+00

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Appendix K Insert Pullout and Flatwise Tensile Test Program for P30X Carbon EO-1 Radiator Sandwich Panel

APPENDIX 1

Insert Pullout and Flatwise Tensile Test Program for P30X Carbon - EO1 Radiator Sandwich Panel

Test Report T 33998-1

for

Lockheed Martin Astronautics P.O. Box 179 Denver, CO 80201

Prepared by

Delsen Testing Laboratories, Inc. 1024 Grand Central Ave. Glendale, CA 91201

June 17, 1998

		Date: 6/17/98	Page: 1 of 5
Lockheed Martin Astron	autics		
P. O. Box 179		W.O. No. T 33988-1	P.O. No. RG7-280889
Denver, CO 80201			
		Identification: As noted	Shipper: None
IDENTIFICATION :	A sandwich p carbon-carbon testing. As inserts with t panel was ide Panel.	panel consisted of an alumin facings were submitted f received, the sandwich was hread size of 10-32 at pre-d entified by the client as P30	hum honeycomb core and for mechanical properties implanted with threaded etermined locations. The X Carbon - E01 Radiator
REFERENCE :	Lockheed Ma	urtin Purchase Order No. RG7	-280889.
TEST REQUESTED :	The client re machined and machined and conducted at	quested that (a) four (4) ins I tested and (b) eight (8) flat I tested from the submitted par room temperature.	ert pull-out specimens be twise tensile specimens be nel. All testing was to be
SPECIMEN PREPARATION :	All specimens geometry and	were prepared by Delsen. A dimensions specified by the c	They were machined to the lient.
	For preparing first identified at the center dimension of (1).	specimens for insert pull-out from the test panel. A square was then machined off from the square specimens was 4.0	t test, insert locations were e piece containing an insert the panel. The nominal) inches (W) by 4.0 inches
	For preparing nominal dime machined from pair of steel lo by 1.5 inches two part paste	specimens for flatwise tens nsion of 1.5 inches (W) by n the panel. Each square spec ading blocks also of planar di (l). The adhesive used was adhesive, Dexter Hysol EA9	sile test, square pieces of 1.5 inches (l) were first timen was then bonded to a imension of 1.5 inches (W) a room temperature curing, 309NA.
SPECIMEN			

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IDENTIFICATION : All specimens were identified with their test type and specimen number as follows:
Page:	2 of 5
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B ----Α

designated the test type: "INPUL" for insert pull-out; A: Where "FL" for flatwise tension.

> designated specimen number (i.e. 1, 2,) **B**:

TEST **PROCEDURES**:

Insert Pull-out

All specimens were tested in accordance with the procedures outlined in a test document supplied by the client and with reference to Lockheed Process Specification 5PTPTT01, Method 4.24.

The loading fixture used was an open-sided box type frame. It consisted of two surface plates and two side support plates. The surface plates functioned as a coupling action-reaction mechanism. They were connected by a pair of side plates which served to transfer load from the bottom plate to the upper plate. The lower plate was mounted to the stationary base of the test machine. The upper plate served as a constraint, thereby applying a reaction force to the specimen when the insert was loaded. The upper plate had a circular opening for allowing the insert to be fully exposed (unconstrained). A plate having a standard opening of 2.275 inches in diameter was used.

In short, the specimen was first installed into the fixture. A 4-inch threaded, 10-32, steel rod was then installed into the center insert. The other end of the rod was connected to the load cell. The specimen was loaded at a constant crosshead rate of 0.05 inch/minute and tested to the point where the load bearing capacity was substantially reduced. For each test, a new strain rod was used.

The tests were performed on a screw-driven, United Calibration Corp. universal testing machine. The load and crosshead displacement were recorded simultaneously by a PC based data acquisition system during each test.

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Flatwise Tension

All specimens were tested in accordance with the procedures outlined in ASTM C 297-94. In short, tensile loading was applied to the specimen in the through-thickness (Z) direction until failure occurred. To comply with the requirement that the specimen failed within 3 to 6 minutes, an optimum crosshead rate of 0.03 inch/minute was used through each test. The loading was exerted by a pair of clevis which were connected to the specimen/block assembly. To ensure proper alignment, a universal joint was connected to each end of the clevis. In turn, the end of one joint was connected to the load cell and the other joint anchored to the test frame.

The tests were performed on a screw-driven, United Calibration Corp. universal testing machine. The load and crosshead displacement were recorded simultaneously by a PC based data acquisition system during each test.

TEST RESULTS: See pages 4 through 5.

REMARKS: In the insert pull-out test, specimens INPUL-2 and -4 exhibited unusually high load and even then the insert was not being pulled to detachment from the surrounding core material. A post mortem examination of the specimens revealed that the adhesive bonding the insert to the core had extended from the insert end to the inner surface of the opposite facing and thus anchoring the insert. This had effectively increased the loading capacity of the insert.

APPENDIX I: Photographic Documentation of Specimen Layout on Test Panel

APPENDIX II: Insert Pull-Out Test - Load vs. Displacement Curves

Respectfully submitted,

Robert W. Ko Program Coordinator DELSEN TESTING LABORATORIES, INC. Jack H.C. Ching, Ph.D. Laboratory Director

Jhc L1D73 T33988-1LMAw

Delsen Testing Laboratories, Inc. is accredited by the American Association for Laboratory Accreditation in the field of mechanical testing, as listed in the current A2LA Directory of Accredited Laboratories.

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Date:	6/17/98
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INSERT PULL-OUT Rate of test: 0.05 inch/minute

TEST METHOD:	Test document supplied by the client and with reference to Lockheed Process Specification 5PTPTT01, Method 4.24
TEST DIRECTION:	Load applied to the through-thickness, Z, direction
MATERIAL ID:	P30X Carbon - E01 Radiator Panel, Core density = 2.0 pcf
NUMBER OF	
INSERTS:	1
PRE-	
CONDITIONING:	None
CONDITIONING:	None
TEST CONDITION:	Tested "as received" at room ambient temperature

		MAXIMUM		
SPECIMEN	THICKNESS	WIDTH	<u>LENGTH</u>	<u>LOAD</u>
<u>BI LCIMLIA</u>	inches	inches	inches	pounds
	0 988	3.998	4.000	550
	0.992	3.996	4.000	1,274
INFUL-2	0.994	3,998	3.999	607
INPUL-4	0.995	3.996	4.004	980
			AVERAGE:	853
		TANDARD I	DEVIATION:	339.5
	COEFFICI	ENT OF VAR	NATION(%):	39.8

- NOTES: 1. All specimens were tested using a reaction plate having an opening of 2.275 inches in diameter.
 - 2. Failure of specimens 1 and 3 involved fracturing of the upper facing and pull-out of the insert along with core cells adjacent to it.
 - 3. For Specimen 2 and 4, the insert was not being pulled to detachment from the surrounding core cells during test. A post mortem examination of the specimens revealed that the adhesive bonding the insert to the core had extended from the insert end to the inner surface of the opposite facing and thus anchoring the insert.

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Date:	6/17/98
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<u>FLATWISE TENSION</u> Rate of test: 0.03 inch/minute

TEST METHOD:	ASTM C 297-94
TEST DIRECTION:	Load applied to the through-thickness, Z, direction
MATERIAL ID:	P30X Carbon - E01 Radiator Panel, Core density = 2.0 pcf
PRE-	
CONDITIONING:	None
CONDITIONING:	None
TEST CONDITION:	Tested "as received" at room ambient temperature

	SANDWICH			MAXIMUM	ULTIMATE
SPECIMEN	THICKNESS	WIDTH	<u>LENGTH</u>	LOAD	<u>STRENGTH</u>
	inches	inches	inches	pounds	psi
FLTEN-1	0.990	1.502	1.500	992	440
FLTEN-2	0.990	1.499	1.501	945	420
FLTEN-3	0.989	1.503	1.498	1,001	445
FLTEN-4	0.990	1.498	1.503	970	431
FLTEN-5	0.992	1.501	1.503	979	434
FLTEN-6	0.991	1.502	1.503	836	370
FLTEN-7	0.991	1.502	1.503	983	435
FLTEN-8	0.992	1.503	1.503	1,037	459
				AVERAGE:	429
			STANDA	ARD DEVIATION:	26.5
COEFFICIENT OF VARIATION(%):				6.18	

NOTE: All specimens exhibited core failure.

APPENDIX 1A

Photographic Documentation of the Specimen Layout on Test Panel

Photomacrograph



Photographic Documentation of Specimen Layout on P30X Carbon - EO1 Radiator Panel

APPENDIX 1B

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Insert Pull-Out Test

Load vs. Crosshead Displacement Curves





INSERT PULL-OUT Lockheed Martin Astro. Test Report: T.33988 Test Temp (°F): 73 Material: P30X Carbon—EO1 Radiator Sandwich Panel Number of Inserts = 1 e = N/AFacing Thickness: 0.0212 in. Dim. = 3.998 in. x 4.000 in

Maximum Load: +549.89 lbs (+2.4460 KN)

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INSERT PULL-OUT Lockheed Martin Astro. Test Report: T.33988 Test Temp (°F): 73 Material: P30X Carbon—EO1 Radiator Sandwich Panel Number of Inserts = 1 e = N/AFacing Thickness: 0.0212 in. Dim. = 3.996 in. x 4.000 in

Maximum Load: +1,273.92 lbs (+5.6660 KN)





INSERT PULL-OUT Lockheed Martin Astro. Test Report: T.33988 Test Temp (°F): 73 Material: P30X Carbon—EO1 Radiator Sandwich Panel Number of Inserts = 1 e = N/AFacing Thickness: 0.0212 in. Dim. = 3.998 in. x 3.999 in

Maximum Load: +607.04 lbs (+2.7000 KN)





INSERT PULL-OUT Lockheed Martin Astro. Test Report: T.33988 Test Temp (°F): 73 Material: P30X Carbon—EO1 Radiator Sandwich Panel Number of Inserts = 1 e = N/AFacing Thickness: 0.0212 in. Dim. = 3.996 in. x 4.004 in

Maximum Load: +979.84 lbs (+4.3580 KN)

APPENDIX 2

Insert Shear Bearing and Sandwich Core Shear Test Program For P30X Carbon-EO1 Radiator Sandwich Panel

Test Report T 34327

For

Lockheed Martin Astronautics P.O. Box 179 Denver, CO 80201

Prepared by

Delsen Testing Laboratories, Inc. 1024 Grand Central Ave. Glendale, CA 91201

June 18, 1998

		Date: 6/18/98	Page: 1 of 7
LOCKHEED MARTIN	ASTRONAUTICS		
P. O. Box 179		W.O. No. T 34327	P.O. No.RG7-280889
Denver, CO 80201		Identification: As noted	Shipper: None
IDENTIFICATION :	A sandwich panel cor carbon-carbon facings testing. As received, unthreaded inserts at identified by the clier	nsisted of an aluminum hone s were submitted for mechan , the sandwich was implanted pre-determined locations. T at as P30X Carbon-E01 Radi	ycomb core and ical properties I with threaded and The panel was ator Panel.
REFERENCE :	Lockheed Martin Pu	rchase Order No. RG7-2808	89.
TEST REQUESTED :	The client requested machined and tested machined and tested conducted at room te	that (a) five (5) insert shear l and (b) two (2) sandwich co from the submitted panel. emperature.	bearing specimens be re shear specimens be All testing was to be
SPECIMEN PREPARATION :	All specimens were p geometry and dimense For preparing specim were first identified containing an insert a Since four (4) of the having had an insert distance and the to determined by the di nearest edge/end. The and the length was do insert, defined as the nearest free end, was	prepared by Delsen. They sions specified by the client. nens for insert shear bearing 1 from the test panel. at each end was then maching five (5) insert sets designant t implanted at a corner of total width of the specific stance from the center of the he nominal width of the specific 5.50 inches. The nominal he distance from the center a 0.52 inch.	were machined to the test, insert locations A rectangular piece ed off from the panel. ated for the test were the panel, the edge mens were therefore is corner insert to the timen was 1.05 inches edge distance of each of the insert to the
,	For preparing specin of nominal dimensio machined from the p determined in compl	nens for core shear test, two n of 3.0 inches (w) by 12.0 in panel. The planar dimension liance with the requirement the	rectangular specimens nches (l) were of the specimens was hat the length should

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be at least 12 times the thickness of the sandwich and the width be at least 2.0 inches. Of the two specimens, one was machined with the length along the core ribbon direction and the other with the length transverse to the ribbon direction. Each specimen was then bonded to a pair of aluminum loading plates, also of planar dimension of 3.0 inches (w) by 12.0 inches (l). The thickness of the plates was 0.5 inch. One end of the plates was machined with a taper with an angle of 30°. The adhesive used was a room temperature curing, two part paste adhesive, Dexter Hysol EA9309NA.

SPECIMEN All specimens were identified with their test type and specimen **IDENTIFICATION** : number as follows: Α B A: designated the test type: "INSHEAR" for insert shear Where bearing; "CORESHEAR" for core shear. a. "PR" for test parallel to ribbon direction b. "TR" for test transverse to ribbon direction B: designated specimen number (i.e. 1, 2,) TEST Insert Shear Bearing PROCEDURES Insert shear bearing tests were performed in accordance with the instructions given the client and with discretionary input from Delsen.

A tensile loading method was employed for the test. The loading apparatus were two symmetrical loading plates made of hardened steel. The plates were of rectangular configuration with nominal dimension of 1.0 inch (w) x 7.0 inches (l) x 0.20 inch (t). Each plate had a loading hole along its centerline. The upper portion of the plate served as a loading end so as to exert a global force. The hole at the lower end served to engage with an insert bolt, thereby transferring load to the insert.

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The specimen was fastened to a loading plate through each insert using a hardened steel bolt of 0.189 inch diameter and a nut. Each bolt was tightened with a torque of 10 inch-pound. This assembly was then pulled at each end via the loading action of a pair of mechanical wedge grips until failure occurred. A constant crosshead rate of 0.05 inch/minute was used throughout each test.

The relative displacement of the two loading bolts was measured by a dual-averaging extensometer.

The client specified in the test plan that a 0.189 inch diameter loading bolt be used for the test. However, this was below the size of the insert hole, which was at 0.208 inch. With the bolt not fully engaged with the insert, this would increase the tendency that the bolt would tilt at the onset of or thereafter loading. By consultation with and subsequent approval of the client, three of the five specimens were tested as per the original test plan, the other two were tested having a steel bushing filling the gap between the bolt and the insert. Accordingly, the loading plates were first machined with a 0.189 inch diameter loading hole. After using on the first three specimens, the hole size was enlarged to 0.208 inch to accommodate for the bushing.

The tests were performed on a screw-driven, United Calibration Corp. universal testing machine. The load, bolt displacement and crosshead displacement were recorded simultaneously by a PC based data acquisition system during each test.

Sandwich Core Shear

Sandwich core shear tests were conducted in accordance with the procedures outlined in ASTM C 273-94, whereby the compression method was employed. Two specimens were tested; one with the coupling shear force applied parallel to the core ribbon direction and the other with the shear force applied transverse to the ribbon direction.

In short, compressive loading was applied to the specimen at the diagonally opposite, tapered ends of the loading plates. The test frame loading mechanism consisted of two loading blocks. The center of each block was machined with a groove, which enabled it to engage with the

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loading plates of the specimen assembly. The two loading blocks were positioned relative to each other with an offset such that the global line of action would pass as closely as possible through diagonally opposite corners of the specimen. To comply with the requirement that the specimen fail within 3 to 6 minutes, an optimum crosshead rate of 0.02 inch/minute was used throughout each test.

Shear deformation exhibited by the specimen was measured from the relative displacement of the loading plates of the specimen assembly. A dual-averaging extensometer was used for this measurement. The shear strain, or distorted right-angle change, was assessed by taking the arc-tangent function of the ratio of the plate displacement to the core thickness. Note, for small angle change, the shear strain was equal to the displacement-core thickness ratio.

The tests were performed on a screw-driven, United Calibration Corp. universal testing machine. The load and plate displacement were recorded simultaneously by a PC based data acquisition system during each test.

TEST RESULTS : See pages 6 through 7.

REMARKS

: In the insert shear bearing test, all specimens exhibited tensile failure on the carbon-carbon facing rather than bearing failure. This could be attributed to the inherent weakness of the facing, which involved only two angle plies oriented at $\pm 22.5^{\circ}$ to the loading axis. It was also contributed in part to the low utilization of the facing material, which was limited by the narrow distance of the insert locations to the free edges of the sandwich panel. In retrospect, had there been more facing material available for the width of the specimens, it would have increased the load bearing capacity and probably entailed to a bearing failure.

APPENDIX I		:	Photographic Documentation of Specimen Layout on Test Panel				st Panel	
APPENDIX II	:	:	Insert Shear Bearing Test Curves	-	Load vs.	Relative	Bolt	Displacement

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APPENDIX II

: Sandwich Core Shear Test - Shear Stress vs. Geometric Shear Strain Curves

Respectfully submitted,

Robert W. Ko Program Coordinator DELSEN TESTING LABORATORIES, INC. Jack H.C. Ching, Ph.D. Laboratory Director

Jhc L1D73 T34327LMAw

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 T 34327

INSERT SHEAR BEARING

Rate of test: 0.05 inch/minute

TEST METHOD TEST DIRECTION	:	Per client instructions Load applied at $\pm 22.5^{\circ}$ to facings material axes	
MATERIAL ID	:	P30X Carbon-E01 Radiator Panel, Core density = 2.0 pcf 0.0212 inch	
INSERT	3		OUTER
DIAMETER INSERT INNER	:	0.550 inch	
DIAMETER PRE-	:	0.208 inch	
CONDITIONING	:	None	
CONDITIONING	:	None	
TEST CONDITION	:	Tested "as received" at room ambient temperature	

SANDWICH						
			INSERT EDGE	MAXIMUM	BEARING	
SPECIMEN	THICKNESS	WIDTH	DISTANCE	LOAD	<u>STRESS</u>	
	inches	inches	inches	pounds	Ksi	
INSHEAR-1	1.001	1.047	0.525	402	34.5	
INSHEAR-2	1.000	1.050	0.528	323	27.7	
INSHEAR-3	1.004	1.055	0.522	285	24.4	
INSHEAR-4	0.994	1.052	0.520	294	25.2	
INSHEAR-5	0.995	1.054	0.515	222	19.0	
				AVERAGE:	26.2	
			STANDARD	DEVIATION:	5.64	

COEFFICIENT OF VARIATION(%): 21.5

NOTES: 1. All specimens exhibited tensile failure.

- 2. Bearing stress was calculated based on the maximum load attained by the specimen at which tensile failure occurred. These values should be viewed with caution since the "true" bearing strength might be higher, had there been a bearing failure occurring.
- 3. Insert edge distance is defined as the distance from the center of the insert to the nearest free end.

Page: 7 of 7 Date: 6/18/98 W.O. #: T 34327

SANDWICH CORE SHEAR

Rate of test: 0.02 inch/minute

TEST METHOD	:	ASTM C 273-94			
TEST DIRECTION	:	As noted		-20 mof	
MATERIAL ID	:	P30X Carbon-E01 Radiator Par	hel, Core density	– 2.0 pci	
PRE-					
CONDITIONING	:	None			
CONDITIONING	:	None			
TEST CONDITION	:	Tested "as received" at room an	nbient temperatur	e	
		SANDWICH	MAXIMUM	SHEAR	SHEAR

<u>SPECIMEN</u>	THICKNESS inches	WIDTH inches	LENGTH inches	LOAD pounds	<u>STRENGTH</u> Psi	<u>MODULUS</u> Msi
Tested with sh	iear force applie	d parallel 1	o core ribbon	direction:		
CORESHEAR -PR-1	1.000 (0.958)	3.002	11.970	3,241	90.2	0.0284
Tested with shear force applied transverse to core ribbon direction:						

CORESHEAR				2.250	62 1	0.0135
-TR-1	0.998 (0.956)	2.990	11.983	2,239	05.1	0.0155

NOTES: 1. All specimens exhibited core failure.

- Thickness measurement in parenthesis denotes core values.
 - 3. Strength and modulus were calculated, in part, based on the core thickness. The thickness was determined by subtracting the facings from the specimen overall thickness. An average thickness of the facings was used in the calculation. This was obtained by first picking three representative sections from the panel and then burning off the core from the facings. The debonded face sheets were then measured to obtain an average, which was 0.0212 inch.
 - 4. Modulus was determined between 500 and 1,500 microstrain using a secant method.

APPENDIX 2A

Photographic Documentation of the Specimen Layout on Test Panel

Photomacrograph



Photographic Documentation of Specimen Layout on P30X Carbon - E01 Radiator Panel

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APPENDIX 2B

Insert Bearing Test

Load vs. Relative Bolt Displacement Curves

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1. 1999/200



Delsen Testing Laboratories, Inc.

Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 73 Conditioning: None Material: P30X Carbon—EO1 Radiator Sandwich Panel Sandwich Width: 1.047 in. Sandwich Thickness: 1.001 in. Facings Average Thick.: 0.0212 in. Insert Outer Dia.: 0.550 in. Insert Inner Dia.: 0.208 in. Insert Edge Distance: 0.525 in. Max. Load, lbf: 401.9





Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 73 Conditioning: None Material: P30X Carbon-EO1 Radiator Sandwich Panel Sandwich Width: 1.050 in. Sandwich Thickness: 1.000 in. Facings Average Thick.: 0.0212 in. Insert Outer Dia.: 0.550 in. Insert Inner Dia.: 0.208 in. Insert Edge Distance: 0.528 in. Max. Load, lbf: 322.6





Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 73 Conditioning: None Material: P30X Carbon—EO1 Radiator Sandwich Panel Sandwich Width: 1.050 in. Sandwich Thickness: 1.004 in. Facings Average Thick.: 0.0212 in. Insert Outer Dia.: 0.550 in. Insert Inner Dia.: 0.208 in. Insert Edge Distance: 0.522 in. Max. Load, lbf: 284.5

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Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 73 Conditioning: None Material: P30X Carbon—EO1 Radiator Sandwich Panel Sandwich Width: 1.052 in. Sandwich Thickness: 0.994 in. Facings Average Thick.: 0.0212 in. Insert Outer Dia.: 0.550 in. Insert Inner Dia.: 0.208 in. Insert Edge Distance: 0.520 in. Max. Load, lbf: 293.9



Delsen Testing Laboratories, Inc.

Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 73 Conditioning: None Material: P30X Carbon—EO1 Radiator Sandwich Panel Sandwich Width: 1.054 in. Sandwich Thickness: 0.995 in. Facings Average Thick.: 0.0212 in. Insert Outer Dia.: 0.550 in. Insert Inner Dia.: 0.208 in. Insert Edge Distance: 0.515 in. Max. Load, lbf: 221.7

APPENDIX 2C

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Sandwich Core Shear Test

Shear Stress vs. Geometric Shear Strain Curves

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Delsen Testing Laboratories, Inc.

SANDWICH CORE SHEAR

Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 72 Material: P30X Carbon—EO1 Radiator Sandwich Panel Test Direction: Parallel to Ribbon Width: 3.002 in. Length: 11.970 in. Core Thick.: 0.958 in. Maximum Load: 3,241.0 lbs (14.416 KN) Maximum Shear Stress: 90.2 psi (0.622 MPa) Secant Shear Modulus: 0.02837 Msi (0.1956 GPa) File: PR-1 {500 / 1500} {12/41}



Delsen Testing Laboratories, Inc.

SANDWICH CORE SHEAR

Lockheed Martin Astro. Test Report: T.34327 Test Temp (°F): 72 Material: P30X Carbon—EO1 Radiator Sandwich Panel Test Direction: Transverse to Ribbon Width: 2.990 in. Length: 11.983 in. Core Thick.: 0.956 in. Maximum Load: 2,259.2 lbs (10.049 KN) Maximum Shear Stress: 63.1 psi (0.435 MPa) Secant Shear Modulus: 0.01349 Msi (0.0930 GPa) File: PR-1 {500 / 1500} {5/18}

APPENDIX 3

TPRL 1967

Thermophysical Properties of P30X/C EO-1 Specimens

A Report to Lockheed Martin Astronautics

by

H. Groot and D. L. Taylor

January 1998

Thermophysical Properties of P30X/C EO-1 Specimens

Two P30X/C EO-1 samples were submitted for thermophysical property testing from room temperature to 250°C.

The Kohlrausch method involves the determination of the product of the thermal conductivity " λ " and the electrical resistivity " ρ ." Since the electrical resistivity is also measured at the same time, λ can be calculated. The method involves passing constant direct current through the specimen to heat the sample while the ends are kept at constant temperature. Radial heat losses are minimized by an external heater whose center temperatures are maintained at the sample's midpoint temperatures and whose ends are also cooled by water or liquid nitrogen. With these provisions, at steady-state a parabola-like axial temperature profile is obtained. Thermocouples also act as voltage probes. Numbering the center thermocouple as the "2" position and the other positions as "1" and "3," it is possible to get the products of λ and ρ :

$$\lambda \rho = (V_3 - V_1)^2 / 4 [2T_2 - (T_1 + T_3)]$$

where $V_3 - V_1$ is the voltage drop between the third and first thermocouple, $T_1 + T_3$ is the sum of the temperatures at the outside thermocouples, and T_2 is the center temperature. Since ρ is also measured simultaneously ($\rho = (V_1 - V_3)$ A/IL where A is the cross-sectional area, I is the current, and L is the distance between positions 1 and 3), λ can be calculated. The data collection (T_1 , T_2 , T_2 , V_1 , V_3 , I) are computerized and the results calculated for a set of measurements performed while the sample is under vacuum and the heater temperature matched to that of T_2 . Additional current is used, a new set of equilibrium conditions is obtained, and the process repeated.

Thermal conductivity values accurate well within $\pm 5\%$ are obtained by the Kohlrausch method and all measured quantities are directly traceable to NIST standards. This method is a

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Figure 1. [90°] Thermal Conductivity vs. Temperature Plot of P30X/C (EO-1) Specimen.

Temperature (°C)	Conductivity (W cm ⁻¹ K ⁻¹)	Resistivity (microhms cm)
51.4	2.1450	545.519
72.4	2.1269	531.905
95.9	2.0886	518.609
119.3	2.0246	507.028
144.6	1.9708	496.166
168.5	1.9519	487.016
193.0	1.9027	478.773
216.8	1.8481	471.731
239.6	1.8018	465.805
263.6	1.7542	460.427

 Table 2.
 Sample TC-11: 0° Direction Thermal Conductivity P30X/C (EO-1) Specimen.

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