1995120373

N95- 31794

Evaluation of Control Parameters for Spray-In-Air (SIA) Aqueous Cleaning for Shuttle RSRM Hardware

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ABSTRACT

HD-2 grease is deliberately applied to Shuttle Redesigned Solid Rocket Motor (RSRM) D6AC steel hardware parts as a temporary protective coating for storage and shipping. This HD-2 grease is the most common form of surface contamination on RSRM hardware and must be removed prior to subsequent surface treatment. Failure to achieve an acceptable level of cleanliness (HD-2 calcium grease removal) is a common cause of defect incidence. Common failures from ineffective cleaning include poor adhesion of surface coatings, reduced bond performance of structural adhesives and failure to pass cleanliness inspection standards. The RSRM hardware is currently cleaned and refurbished using methyl chloroform (1,1,1-trichloroethane). This chlorinated solvent is mandated for elimination due to its ozone depleting characteristics. This report describes an experimental study of an aqueous cleaning system (which uses Brulin 815 GD) as a replacement for methyl chloroform. Evaluation of process control parameters for this cleaner are discussed as well as cleaning mechanisms for a spray-in-air process.

Testing documented in MSFC Technical Report EH33/92-5, published previously, determined that an acceptable level of cleanliness can be achieved with application of a spray-in-air (SIA) process which utilizes aqueous based cleaner formulations. HD-2 contaminated D6AC plates were cleaned using this aqueous method and subsequently bonded for tensile adhesion testing. The test specimens produced cohesive failures within an EA913NA adhesive bond line. The RSRM nozzle fixed housing bond line utilizes EA913NA adhesive and is considered "sensitive" to HD-2 Calcium grease surface film levels above 5 mg/ft^2.

This report documents a study to further evaluate the spray-in-air process utilizing Brulin 815 GD cleaner and the mechanisms of this cleaning method. The ultimate goal is to qualify an environmentally compliant cleaner such as Brulin 815 GD to replace the current chlorinated cleaning system, methyl chloroform.

The test objectives of this study were as follows:

1) Evaluate the main effects and interactions of the following control factors used in spray-in-air aqueous cleaning of D6AC steel: pressure, cleaner temperature, cleaner mix ratio, and cleaner soak time.

2) Determine if cleaning with ambient temperature cleaner solution will yield acceptable bond strengths with the other factor settings optimized.

3) Determine effects of any residual cleaner and HD-2 grease on an Optically Stimulated Electron Emission (OSEE) measurement and on tensile adhesion strength.

The aqueous cleaner used in this study was Brulin 815 GD (Brulin & Company Inc. of Indianapolis, IN). The cleaner is a biodegradable liquid which is mixed with tap water to desired mix ratios for various cleaning applications. The cleaner contains no "butyl" or petroleum solvents but does contain a ferrous metal flash rust inhibitor for temporary corrosion protection. The chemical composition includes detergents, nonylphenoxypolyethyleneoxyethanol (5-10%) and alkaline cleaners.

The adhesive, EA-913NA, is supplied by Dexter Hysol and is currently used to bond nozzle insulation components in the RSRM . The manufacture date of the test adhesive (Lot 0064) was August 7, 1993 with an expiration date of August 7, 1994. This adhesive was chosen because it is extremely sensitive to minute amounts of HD-2 grease contamination on metal substrates..

The silane primer used in this study is a Thiokol formulation designated UF-3296. This primer was used only on the button side of the specimens to drive the failure mode down to the test panel. The detailed formulation is described in Thiokol specification STW5-3215.

The equipment used to wash the test panels in this study was an enclosed high pressure parts washer. The washer (Model 550) is a spray-in-air type unit manufactured by ADF Industries and was modified to prevent the cleaner solution from being recycled. Therefore, when panels were cleaned they were always sprayed with "virgin" cleaner solution. The machine also has a pressure adjustment valve which allows the user to adjust the pressure from 75 to 675 psi. The corresponding flow rates at these pressures were approximately 0.5 and 4 gpm, respectively. The spray gun used a fan type nozzle (Binks nozzle no. 1508) which produced a 4 inch wide fan at a standoff distance of approximately 8 inches.

The specimens used in these tests were 4 X 8 inch D6AC steel plates. Silane primed tensile adhesion buttons having 1.0 square inch bonding area were bonded to each plate. A total of seven tensile adhesion buttons were bonded to each plate with one plate representing one run of the test matrix shown in Table 1.

D6AC steel test panels were placed in a methyl chloroform vapor degreaser conforming to MIL-T-81533. The degreasing time was 15 minutes. Surface preparation grit blast was performed using 60 mesh silicon carbide grit at 80 psi and a 90 degree grit blast angle. The grit blast operation was intended to achieve a 125 Ra nominal finish. Following the blasting operation D6AC steel test specimens were placed in a vapor degreaser with methyl chloroform conforming to MIL-T-81533. The degreasing time was 15

RUN NO.	PANEL NO.	TEMPERATURE (DEG. F)	SOAK TIME (MINUTES)	CONCENTRATION (PERCENT)	PRESSURE (PSI)
1	2	72	2	10	75
2	3	72	2	30	675
3	7	140	2	30	75
4	9	140	2	10	675
5	13	72	10	10	75
6	10	140	10	30	75
7	12	140	10	10	675
8	5	72	10	30	675
9	14	140	2	10	75
10	15	72	2	30	75
11	16	72	2	10	675
12	8	140	2	30	675
13	11	140	10	10	75
14	6	72	10	30	75
15	4	72	10	10	675
16	17	140	10	30	675

Table 1 Spray-In-Air Cleaning Test Matrix Including Factors

minutes. The specimens were removed from the degreaser and checked for stains. Additional degreasings were permitted if stains were found. Bonding surfaces were prepared by grit blasting surfaces of each test specimen using "virgin" 100 to 200 mesh Zirclean grit. A grit blast pressure and angle of 80 psi and 45 degrees was used. A 4 to 5 inch standoff distance for the blast gun was also used. The resulting speed of travel was approximately 3-4 inches per second. The grit blast dust was removed using missile grade air.

It should be noted that normally the nozzle grit blast pressure, angle, and standoff distance for RSRM nozzle hardware is 40-70 psi, 45-90 degrees, and 12 to 15 inches. However, due to differences in this test grit blasting chamber the pressure and standoff distance were adjusted (as stated above) to produce the uniform dull white bonding surfaces called out in the RSRM manufacturing instructions.

Baseline OSEE inspection was performed within 5 minutes of grit blasting and the readings were documented. A heavy layer of HD-2 grease was applied to the test panels using a poly-foam brush. The test panels were placed in an oven for 24 hours at 100±5 °F to allow grease to flow into the micro crevices of the test panels. Excess grease was scraped from each test panel using wooden tongue depressors. More than one pass with the tongue depressor was necessary. Each panel was cleaned according to the factors shown in Table 1. The panels were placed vertically for cleaning. Figure 1 illustrates the parameters of the cleaning cycle. The panels were rinsed using deionized water pressurized to 75 psi in a small pressure rinser. The test panels were dried using missile grade air. The test panels were placed in a nitrogen purged plastic bag for transport to the OSEE laboratory. They were situated such that the bag surface did not contact the bonding substrate of the panels.

OSEE inspection was performed and the measurements were documented. Visual and black light inspection was performed and the approximate amount of grease remaining on the surface of the panel was documented. Coverage was estimated and documented in terms of percent. The test panels were placed in a nitrogen purged bag and transported to the lay-up facility.

The adhesive was prepared in accordance with Thiokol specification STW5-3292 followed by vacuum mixing for 15 minutes (mix time was recorded). Seven tensile buttons were bonded to each prepared tensile plate for each run from the Table 1 matrix. All tensile buttons had a 30 ± 5 mil bond line thickness. Lay-up start times and lay-up finish times were recorded. The test specimens were cured at 105±5°F for 72 hours minimum.

Figure 2 is a flow diagram of the fabrication process used for these test specimens.

All sixteen of the test panels used in this study were processed in a controlled manner so that the elapsed time from operation to operation was the same for each panel. For example, each panel was un-bagged, grit blasted, OSEE inspected, and re-bagged before the next panel was un-bagged. The cleaning, rinsing and bonding processes used for the panels were also performed in this manner.

OSEE inspection was also performed for each panel. The data were reduced to a difference between the baseline OSEE reading taken when the panel was clean and freshly grit blasted and after the panel had undergone its designated cleaning cycle. The data were reported in terms of centivolts which indicated the amount of ultraviolet light reflected off of the surface of the test panel.

Visual and black light inspection was performed on each of the panels to quantify the amount of grease remaining on the surface of the panels. Grease coverage was estimated and documented in terms of percent coverage.

Tensile adhesion testing was performed to determine the effect of the specified cleaning process on bond strength for each of the panels. As mentioned Hysol EA-913NA adhesive was used in this study because of its high sensitivity to HD-2 grease contamination. Seven tensile buttons were bonded to each test panel using Teflon spacers which yielded a 0.030 inch bond line thickness. The specimens were pulled at 72 ±5°F with a crosshead speed of 0.05 inch per minute.

Visual inspection of the test panels indicated that eight of the panels were successfully cleaned using the described factors. A successful cleaning was defined as a panel in which no grease remained on the bonding surface (as evident from the black light inspection). In addition, panels which were

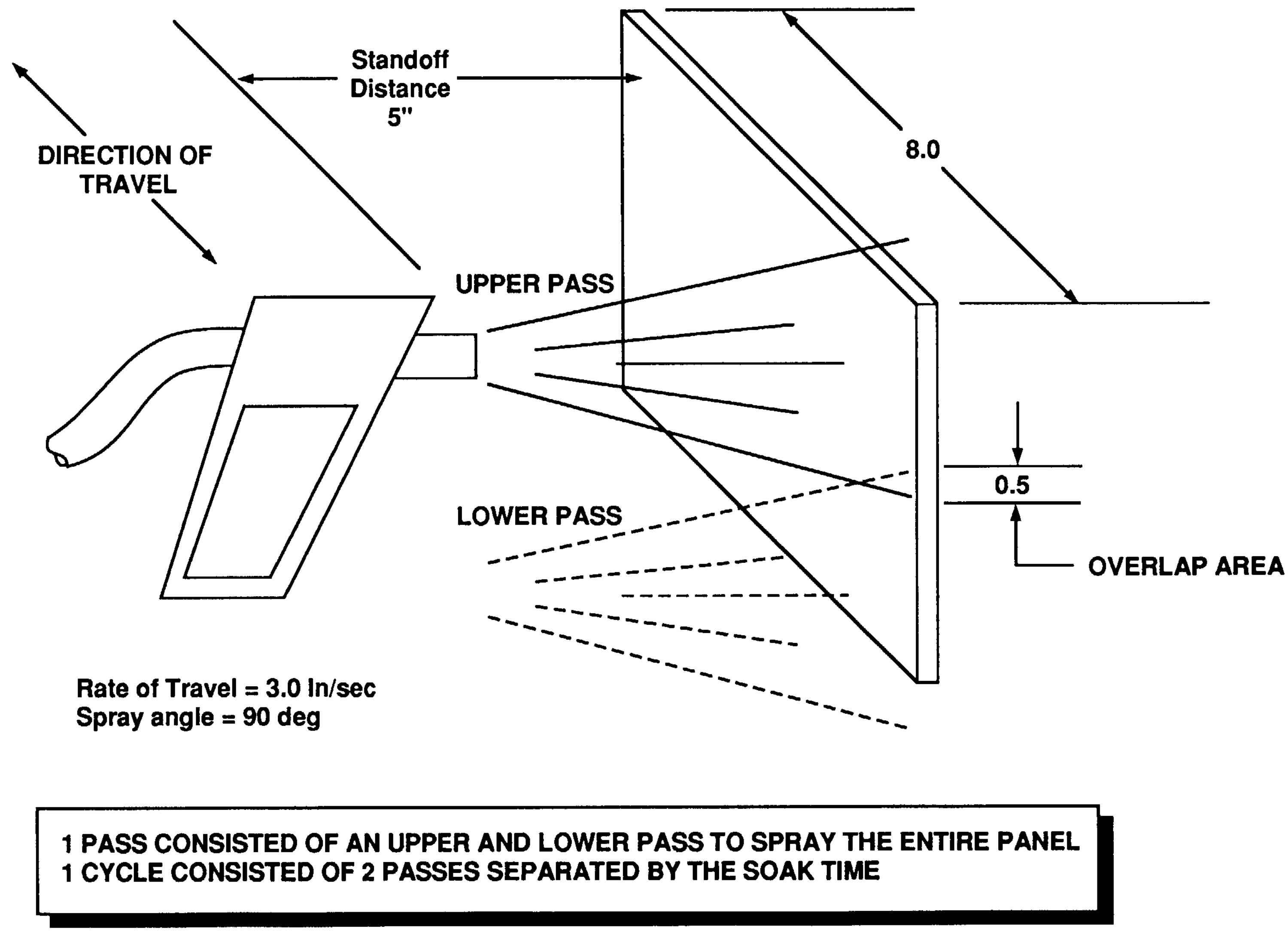

Figure 1 Test Panel Spray Cleaning Parameters

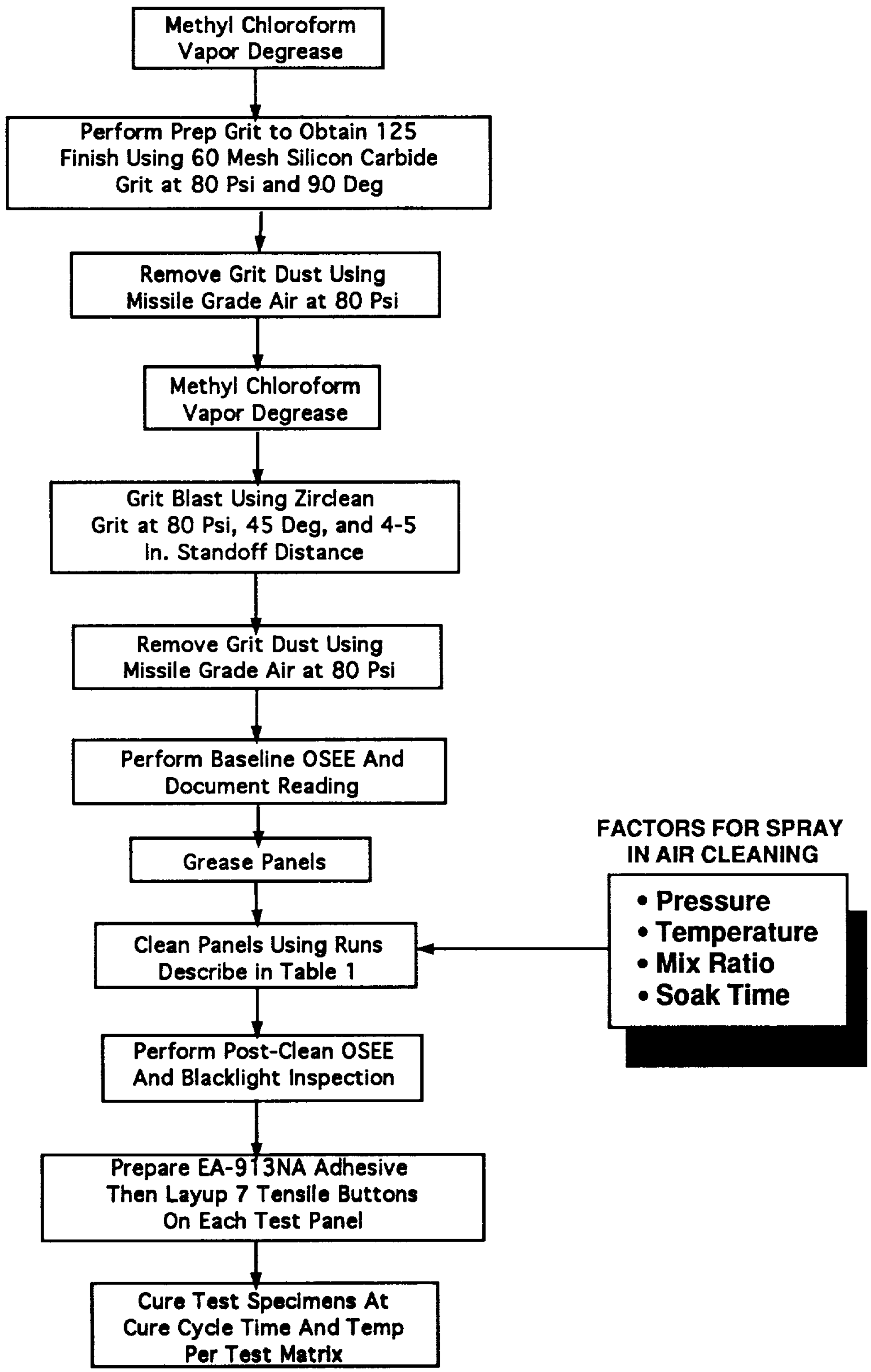

Figure 2 Test Specimen Preparation Flow Diagram

successfully cleaned also exhibited fast dry times following the rinsing operation. Six of the test panels appeared visually clean following the cleaning sequence and also yielded high tensile adhesion strengths.

Optically Stimulated Electron Emission readings were taken to provide a delta value between the baseline OSEE reading taken when the panel was clean and freshly grit blasted and after the panel had undergone its designated cleaning cycle. These values are shown in Table 2.

Also listed in Table 2 are the results from black light inspection of the panels. This method provided a more striking indication of the residual grease remaining on the panels. Each panel was inspected and given a percent residual grease value (0 - 100%).

The results of the tensile adhesion testing are tabulated in Table 2 and graphed in Figure 3. The panels which appeared "clean" through visual and black light inspection had the highest tensile adhesion strengths as expected. Panel to panel comparison of these "clean" panels shows individual tensile button strengths with less variation from button to button on a panel. The failure modes for these panels were almost exclusively cohesive failures. The panels with a percentage of residue remaining typically resulted in adhesive failure modes. The tensile strengths from these panels depended primarily on location of button bonding on the panel.

Initial intentions on measuring the foaming affects of Brulin 815 GD consisted of spraying the cleaner onto a vertically oriented 8 X 12 inch clean steel plate and catching the solution in a transport pan as it drained off. Once this was done the solution was to be transferred into a 3500 ml graduated beaker where the amount of liquid and foam could be measured. However, during the test it was surprising to observe that foaming of this cleaner at 10% or 30% and 675 psi yielded a post spray solution which consisted entirely of foam. The foam varied in density as part of the slurry could be poured while other parts were more like soap suds. Due to a lack of a controlled method for characterizing the ability of the Brulin 815 GD to foam the factor was not investigated further in this test.

As shown in Table 2 the factor having the greatest effect on black light, OSEE, and tensile adhesion strength was spray pressure. This is due primarily to the mechanical energy of the spray stream which breaks the grease down into a very thin layer allowing the Brulin 815 GD to effectively interact and remove the remaining grease. This displacement ability of pressure can be seen in the high and low pressure condition of the post cleaned panels. Inspection of these low pressure panels revealed streaks which in some cases appear clean and in others appear streaked with grease. The scraping was not performed with a "true" straight edge across the width of the scrape, thus an uneven grease layer was produced for the cleaner to interact with. The thin layers of grease were effectively cleaned (at 75 psi spray pressure) while adjacent thick areas of grease were not. A button-to-button comparison on individual panels sprayed at low pressure revealed a high coefficient of variation for tensile strength values. This variation was significantly lower with the high spray pressure test runs.

It should also be noted that in the previous study (documented in NASA report EH33/92-5) 75 psi pressure was effectively used to clean several test panels. In this study 75 psi was not effective. This is due to the fact that two different cleaning cycles were used in the previous study. Two continuous one minute washes which were separated by a 5 minute soak time resulted in a very high volume of cleaner per unit area of test substrate for the wash cycle. In the present study the cycle was changed and reduced to effectively evaluate the factors of mix ratio, temperature, pressure, and soak time. This new cycle was also more representative of the RSRM motor case spraying process being proposed by Thiokol.

As previously mentioned the factor of pressure had the most significant positive effect on the responses of black light and tensile adhesion strength results. The primary "cleaning" or grease removal mechanism identified was material displacement. This displacement reduces the thickness of the HD-2 grease layer enough so that the Brulin 815 GD can effectively remove it for the given cleaning cycle. The results of test runs using high pressure illustrate the insignificance of cleaner temperature, concentration, and soak time for these test runs. The results of test runs using low pressure illustrate the importance of the other factors. Temperature, cleaner concentration, and cleaner soak time affect the process with significance in the order listed herein. To further understand the contribution from these other factors additional testing should include a uniform amount of contamination applied across the test substrate. This

RUN NO.	PANEL NO.	TEMPERATURE (DEG. F)	SOAK TIME (MINUTES)	CONCENTRATION (PERCENT)	PRESSURE (PSI)	BLACK LIGHT % RESIDUE COVERAGE	OSEE DELTA (CENTIVOLTS)	TENSILE ADHESION (PSI)
16	17	140	10	30	675	0	441	8743
2	3	72	2	30	675	0	425	8584
8	5	72	10	30	675	0	360	8568
7	12	140	10	10	675	0	476	8554
12	8	140	2	30	675	0	484	8512
11	16	72	2	10	675	0	293	8277
15	4	72	10	10	675	0	444	7939
4	9	140	2	10	675	0	408	7391
6	10	140	10	30	75	15	481	7038
10	15	72	2	30	75	25	290	4478
3	7	140	2	30	75	30	407	4476
13	11	140	10	10	75	20	417	4349
5	13	72	10	10	75	40	283	2420
9	14	140	2	10	75	40	299	1518
14	6	72	10	30	75	40	441	1153
1	2	72	2	10	75	95	445	871

Table 2 Spray-In-Air Cleaning Test Matrix Including Factors and Responses

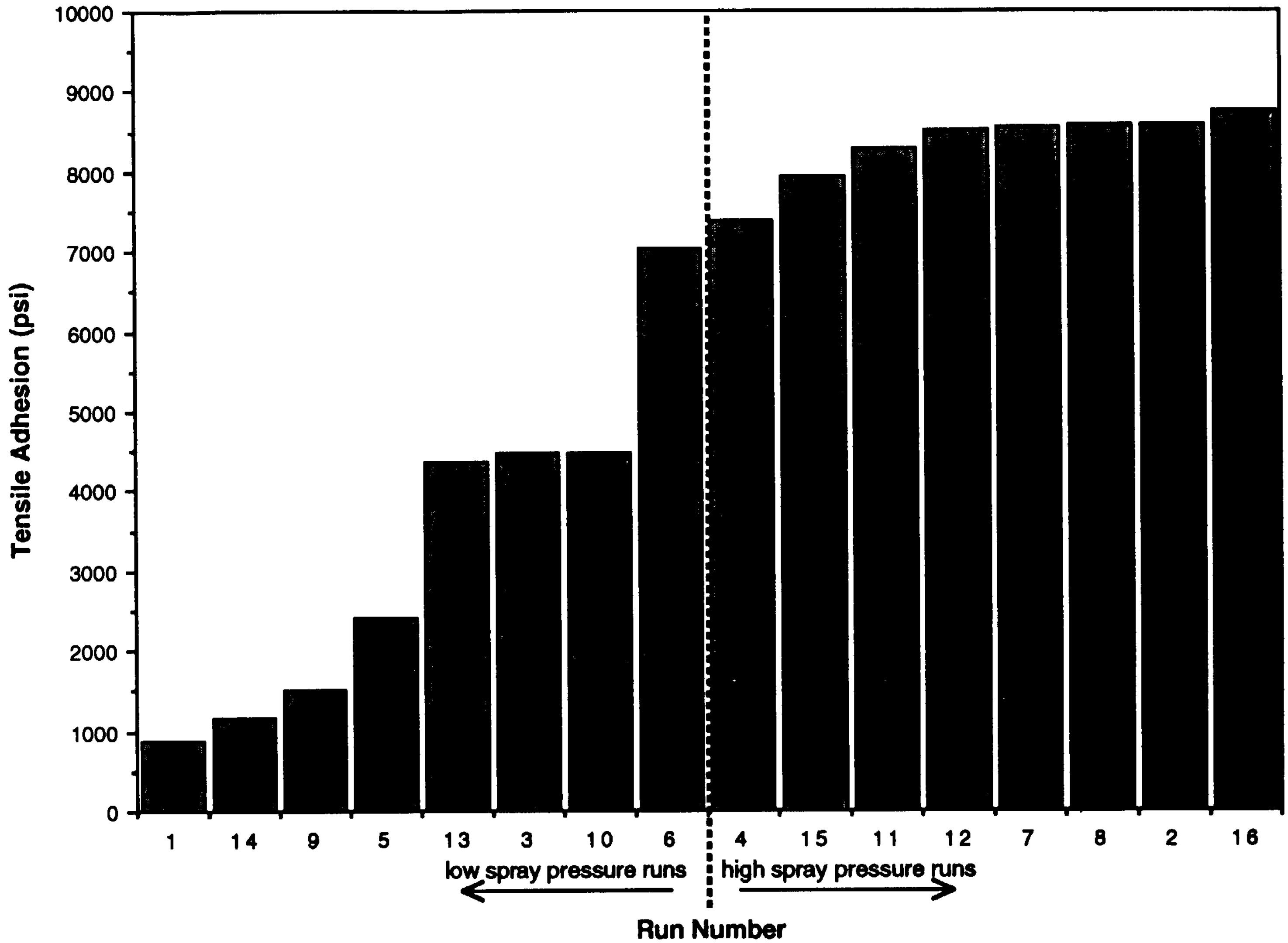

Figure 3 Graph of Tensile Adhesion Data

uniformity should enable evaluation of the cleaner's performance at lower pressure limits so that the contribution and interactions from the other factors can be fully understood.

This study reveals that room temperature cleaning is possible as long as high pressure is used to achieve the displacement discussed earlier. Runs 2,8 and 11 all yielded tensile adhesion strengths of at least 8000 psi., which represents the optimum bond strength for the EA913NA adhesive. These panels were all cleaned at 72 °F and 675 psi.

The effects of residual Brulin 815 GD not rinsed from the panels was difficult to assess because the resulting OSEE data was scattered. NVR (Non Volatile Residue) along with other surface analysis tests should be performed for a better understanding of the residual cleaner remaining. The measured responses of black light, and tensile adhesion strength dramatically displayed the effect of residual HD-2 calcium grease on the test panels (Refer to Table 2). The black light detectable residual grease contamination significantly lowered tensile adhesion strengths for the test runs

Additional testing should involve evaluating a cleaning process which uses a high pressure (< 1000 psi) "pre-rinse" with water only prior to the spray-in-air aqueous cleaning steps. This would significantly reduce recycle or reclamation problems with the contaminated cleaner solutions. The high pressure water-only pre-rinse would also ensure a uniform thickness of contamination across the test substrate, thus allowing a full evaluation of the factors and their interactions.

REFERENCES

1. C. D. DeWeese, Technical Report #EH33/92-5, September, 1992, "Solvent Replacement Studies for RSRM Internal Insulation Vulcanization / Bonding"

2. C. D. DeWeese, Technical Report #EH33/94-3, April, 1994, "Evaluation of Control Parameters for Spray-In-Air (SIA) Aqueous Cleaning for Shuttle RSRM Hardware"