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A DESIGN OF EXPERIMENTS TEST TO DEFINE CRITICAL SPRAY CLEANING PARAMETERS FOR BRULIN 815 GD AND JETTACIN CLEANERS

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ABSTRACT

Experimental design testing was conducted to identify critical parameters of a aqueous spray process intended for cleaning solid rocket motor metal components (steel and aluminum). A two-level, six-parameter, fractional factorial matrix was constructed and conducted for two cleaners, Brulin 815 GD and Diversey Jettacin. The matrix parameters included cleaner temperature and concentration, wash density, wash pressure, rinse pressure, and dishwasher type. Other spray parameters: nozzle stand-off, rinse water temperature, wash and rinse time, dry conditions, and type of rinse water (deionized) were held constant. Matrix response testing utilized discriminating bond specimens (fracture energy and tensile adhesion strength) which represent critical production bond lines. Overall, Jettacin spray cleaning was insensitive to the range of conditions tested for all parameters and exhibited bond strengths significantly above the TCA test baseline for all bond lines tested. Brulin 815 was sensitive to cleaning temperature, but produced bond strengths above the TCA test baseline even at the lower temperatures. Ultimately, the experimental design database was utilized to recommend process parameter settings for future aqueous spray cleaning characterization work.

INTRODUCTION

Thiokol Space Operations has been on an aggressive schedule to select non-ODC cleaners to replace the 685 thousand pounds of 1,1,1-trichloroethane currently used annually in two main vapor degreasers. Both immersion and spray processes were evaluated. For this particular application, it was determined that spray cleaning was not only superior, but also more practical. Two cleaners emerged from over 150 tested: Brulin 815 GD and Diversey Jettacin. Once the cleaner selection had been narrowed to these two, a process optimization test was defined. It was determined that a Design-Of-Experiments (DOE) matrix should be used to meet the following objectives:

- Identify critical aqueous spray cleaning process parameters
- Determine operating ranges of the matrix parameters
- Specify the cleaning process for remaining development work

TABLE 1 - TEST MATRIX

6 VARIABLES (2 LEVELS)

For Each Cleaner:

Test	A	B	C	D	E	F
1	+	+	+	+	+	+
2	+	+	+	-	+	-
3	+	+	-	+	-	-
4	+	+	-	-	-	+
5	+	-	+	+	-	-
6	+	-	+	-	-	+
7	+	-	-	+	+	+
8	+	-	-	-	+	-
9	-	+	+	+	-	+
10	-	+	+	-	-	-
11	-	+	-	+	+	-
12	-	+	-	-	+	+
13	-	-	+	+	+	-
14	-	-	+	-	+	+
15	-	-	-	+	-	+
16	-	-	-	-	-	-

Variables	High Level (+)	Low Level (-)
A=Cleaner Temperature	135° ± 5° F	70° ± 5° F
B=Cleaner Concentration	30%	10%
C=Wash Density	2.3 gpm	1.5 gpm
D=Wash Pressure	250 ± 25 psi	70 psi
E=Rinse Pressure	250 ± 25 psi	70 psi
F=Lab Location	HSO	S&E
G=Cleaner	Jettacin	Brulin 815 GD

Wash density = (Flow rate x Cleaning time)/Area of surface cleaned

Time and area were held constant; thus, wash density is expressed in terms of flow rate.

Huntsville tests were conducted by Thiokol Huntsville Space Operations. Utah tests were conducted by Thiokol Science and Engineering.

The matrix was a six-parameter, two-level fractional factorial which was executed once for each cleaner. The matrix, process parameters and the test levels are shown in Table 1. There was concern that division of the matrices at two laboratory locations may compromise the experimental control of each matrix; particularly, because the two labs utilized dissimilar dishwashers (Table 2). However, the effort was divided with confidence that experimental consistency could be achieved because:

- Historically, Huntsville and Utah bond property databases have correlated closely

- Utilization of two different dishwasher systems would demonstrate aqueous cleaning process robustness
- Both labs used the same lot of adhesive for bond specimen assembly
- Lab location was selected as a process variable

The final selection of variables included cleaner temperature, cleaner concentration, wash density, wash pressure, rinse pressure and lab location. Other parameters, deemed less critical through technical discussions, were held constant as indicated in Table 2.

TABLE 2 - CONSTANT PARAMETERS

PARAMETER		HSV	Utah
Nozzle Stand-off Distance		6 ± 1 inches	8 ± 1 inches
Wash Time		9 minutes	2-3 minutes
Rinse Water Temperature		65 ± 5°F	65 ± 5°F
Rinse Time		10 minutes	2-3 minutes
Dry Conditions		Missile grade, ambient air	Clean room grade, ambient air
Contaminants	(Steel)	Magnaflux + Diala oil + grease	Magnaflux + Diala oil + grease
	(Alum)	Grease	Grease
Water		Deionized	Deionized

NOTE: Cleaning and rinsing times and stand-off distances were corrected for Huntsville and Utah "dishwasher" differences. The Huntsville system configured the specimens horizontally on a rotating table under fixed nozzles. The Utah system configured the specimens vertically on a rack with oscillating nozzles.

RESULTS

Response tests for these matrices are listed as follows:

- Tensile Adhesion Strength (Bond line = EA 913 Adhesive/Steel and EA 913 Adhesive/Aluminum)
- Fracture Energy (Bond line = EA 913 Adhesive/Steel and EA 913 Adhesive/Aluminum)
- Surface Energy Analysis (Contact Angle)
- Surface Chemistry Analysis (ESCA/SIMS/Auger)
- OSEE Analysis

The bond lines represent actual production bond systems and are typically used for screening tests because of their sensitivity to contamination and other substrate characteristics. In addition, the fracture energy and tensile adhesion tests function well in differentiating substrate treatment effects. Essentially, there is much information to be gained from the analysis because of the response tests.

The discussion of the paper is limited to the bond line data analysis because the results of the other tests corresponded closely with the bond line data. Thus, the conclusions of the bond line data are analogous to the other tests. The TCA (1,1,1-trichloroethane) baseline data cited in this paper was processed differently than current RSRM production hardware is processed. In order to maintain similarity with the aqueous cleaned specimens, the TCA baseline panels did not receive a post clean or pre-bond grit blast. A grit blast after aqueous cleaning would prevent discrimination between critical parameters by masking their effect; therefore, for testing purposes it was omitted.

The data from the sixteen runs of each matrix, shown in Figures 1-4, were processed through a least-squares regression analysis which assessed the main effects of high/low parameter variation on each response. In addition, the two-parameter interactive effects were calculated. Parameter variation was regarded significant to the response when the high-to-low change cause the value of response measurement to exceed experimental variability (outside the error bands). A parameter was considered critical if the main effects were statistically significant at a 95% confidence level. Table 3 shows the interactive parameters. The analysis also combined the results of the Brulin 815 and Jettacin matrices, which created a seventh parameter, cleaner. The analyses are plotted in Figures 5 to 9. The charts plot the measured response against each parameter (high and low level). The dotted lines (error bands) show experimental variability. The following paragraphs discuss the results for each bond line response tested. It is important to note that all of the main response average bond values are significantly above the baseline TCA standard database that has been constructed during the ODC testing.

Tensile Adhesion Strength (EA 913/Steel). Figure 5, the main effects chart, shows that cleaner temperature, wash density, and choice of cleaner are critical to the tensile adhesion strength of this bond line. On the average, raising the cleaner temperature to 135 °F increases the tensile adhesion strength by 640 psi. Increasing the wash density to 2.3 gpm also raises the tensile adhesion strength by 455 psi. Finally, cleaning with Jettacin results in a tensile adhesion strength of 476 psi higher than Brulin 815.

There are three significant two-parameter interactions of interest shown in Figure 6. This chart is a composite that plots tensile adhesion strength against cleaner temperature at the two wash densities for both cleaners. The data show that high temperature cleaning is independent of wash density variation. Also, high wash density cleaning is independent of cleaner temperature variation. While the Brulin 815 GD requires the high temperature setting to match the bond strengths of the Jettacin, even the low temperature setting produces bond strengths above the TCA test baseline of 6300 psi. Both cleaners are essentially equivalent at either a high wash density or high temperature setting.

Tensile Adhesion Strength (EA 913/Aluminum). The main effects chart, Figure 7, for this response test analysis reveals that cleaner temperature and type are critical. Higher temperature cleaning improved tensile adhesion strength by 1,134 psi. Also, Jettacin cleaning resulted in a 1,054 psi higher average bond strength than Brulin 815. Again, these effects was primarily due to the sensitivity of Brulin 815 at the lower temperature. The low parameter values for the 70° F and Brulin 815 cleaned specimens, around 6600 psi, was still 1500 psi above the TCA baseline of 5100 psi for this bond line.

Fracture Energy (EA 913/Steel). The main effects chart for this response property is plotted in Figure 8. This chart shows a critical effect from cleaner temperature, test lab, and cleaner type. Variation in cleaner temperature from 70 °F to 135 °F increases fracture energy from approximately 12.5 to 16.0 in•lbs/in^2. Similar to Figure 6, the two-parameter interaction analysis showed that this effect was due to Brulin 815 cleaning sensitivity to low temperature. Again, Jettacin performed essentially the same at the two ends of the cleaner temperature spectrum. All of the average bond strengths were significantly above the TCA baseline of 2.0 in•lbs/in^2.

Fracture energy also demonstrated significant sensitivity to lab location/dishwasher type. Cleaning in the Huntsville dishwasher reduced fracture energy by 2.8 in•lbs/in^2. The sixteen runs of cleaning in the Huntsville dishwasher provided an average fracture energy of 12.8 in•lbs/in^2, which is substantially higher than nominal baseline cleaning process, TCA vapor degreasing, values.

The main effects analysis also showed that fracture energy effected by the type cleaner. Jettacin cleaning provides a fracture energy of 2.0 in•lbs/in^2 greater than Brulin 815.

Fracture Energy (EA 913/Aluminum). This response demonstrated significant dependance on cleaner temperature due to Brulin 815 temperature sensitivity (Figure 9). The higher cleaner temperature increased the fracture energy from approximately 8.8 to 14.0 in•lbs/in². As with the other bond lines, even the low values are significantly above the TCA baseline values of 0.4 in•lbs/in² established during the ODC testing program.

Similar to the steel fracture energy data, lab location strongly affected this response. There was a delta 2.6 in•lbs/in² decrease in fracture energy when the specimens were cleaned in the Huntsville dishwasher.

Wash density demonstrated main effects on this property. Cleaning at the higher wash density improved fracture energy by 2.1 in•lbs/in².

Table 3 itemizes the significant and interactive effects of each of the parameters of each design of experiments matrix.

CONCLUSION

The individual parameter recommendations can be found in Table 4. To summarize, the data analysis leads to the following conclusions:

- Aqueous spray-in-air cleaning produced statistically significant higher bond properties than the baseline process, TCA vapor degreasing.
- Jettacin cleaning was relatively insensitive to process parameter variation.
- Brulin 815 cleaning was sensitive to variation of cleaner temperature, higher temperature provided higher bond strengths. However, the lower temperatures still provided bond strengths above the TCA baseline.
- Fracture energy sensitivity to the lab location parameter is probably due to differences in the dishwashers.
- Bond properties were insensitive to variation in wash pressure, rinse pressure, and cleaner concentration.
- Aqueous spray cleaning is a robust process in terms of the parameters tested.
- Because all bond line responses were significantly above the testing TCA baseline, factors other than bond strength (recyclability, OSHA, environmental) will play a more significant role in cleaner selection.

Other conclusions that were drawn from this testing included:

- The rinse and dry processes require more definition.
- Aqueous cleaning will effect OSEE readings.
- Black light inspections should be quantified with % clean and intensity of fluorescence rather than pass/fail.
- NVR should be performed concurrent with all testing.

During a mid-scale test a high degree of foaming was noted from both of these cleaners. The two cleaners tested are foaming cleaners intended for immersion type cleaning operations. Nearly identical non-foaming cleaners are available. It has not yet been decided whether work will continue with these foaming cleaners or their non-foaming "sister" cleaners.

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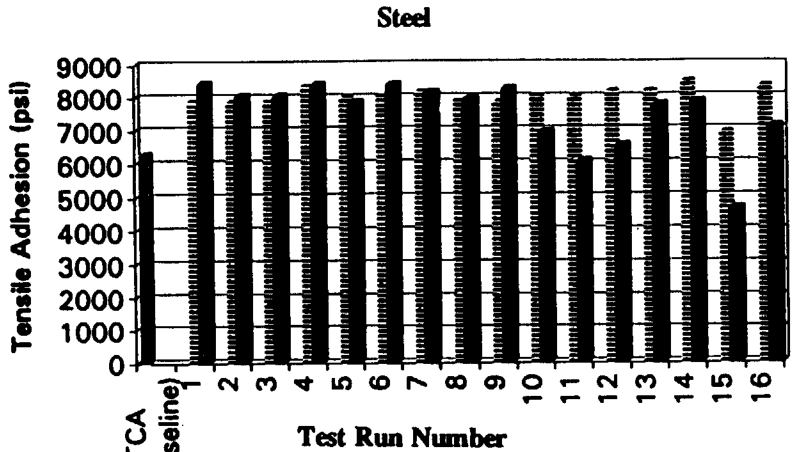
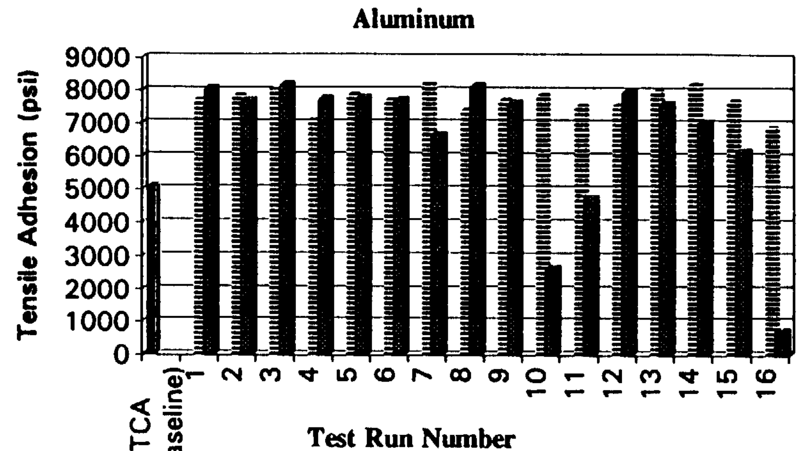


Figure 1



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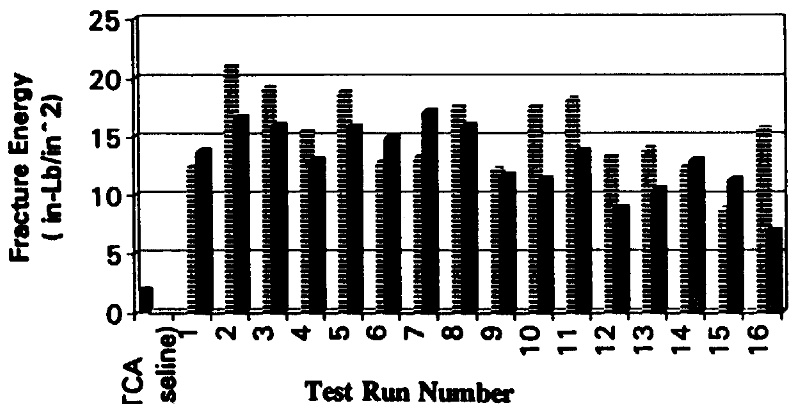


Figure 3

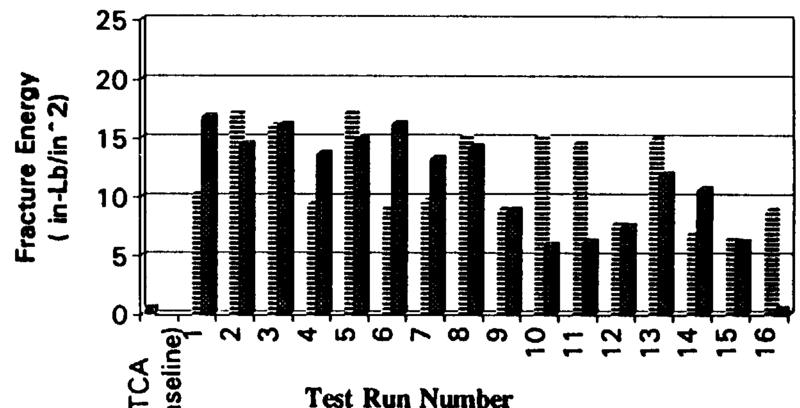


Figure 4

TCA (Baseline) Jettacin Brulin

Table 3 - ETP G Table I Significant Main Effects and Interactions

Response Material/ Test	A Cleaner Temperature	B Cleaner Concentration	C Wash Density	D Wash Pressure	E Rinse Pressure	F Lab Location	G Cleaner
D6AC/ Tensile Adhesion	significant AG interaction AC interaction	not sig	significant CA interaction CG interaction	not sig	not sig	not sig	significant GA interaction GC interaction
D6AC/Fracture Energy	significant AG interaction	not sig BF interaction	not sig	not sig DG interaction	not sig	significant FG interaction FB interaction	significant GA interaction GD interaction GF interaction
Alum/Tensile Adhesion	significant AG interaction AF interaction	not sig	not sig	not sig	not sig	not sig FA interaction	significant GA interaction
Alum/Fracture Energy	significant AG interaction	not sig	significant	significant DF interaction	not sig	significant FD interaction FG interaction	not sig GA interaction GF interaction
D6AC panel/ConScan (post clean)	significant	not sig	not sig	not sig	not sig	significant	not sig
D6AC TDCB/ConScan (post clean)	significant AG interaction AF interaction	significant	not sig	not sig	not sig	significant FA interaction	significant GA interaction
D6AC panel/ConScan (delta pre-post clean)	significant	not sig	not sig	not sig	not sig	significant	not sig
D6AC TDCB/ConScan (delta pre-post clean)	significant AB interaction	significant BA interaction	not sig CG interaction	not sig	not sig	significant	significant GC interaction

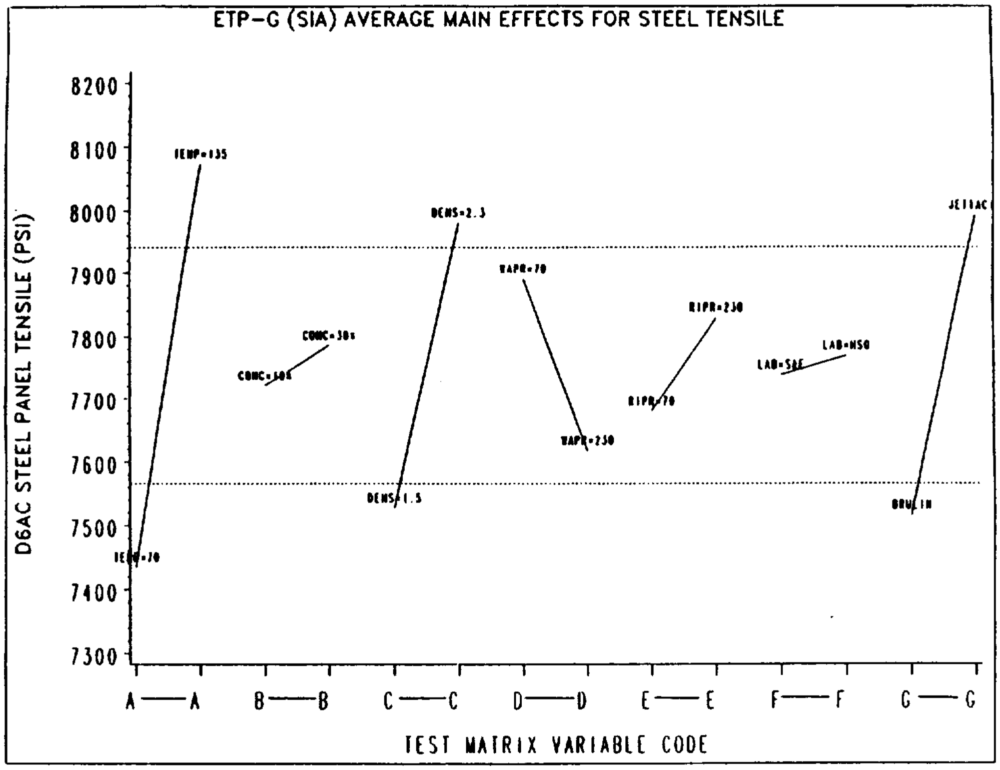


Figure 5

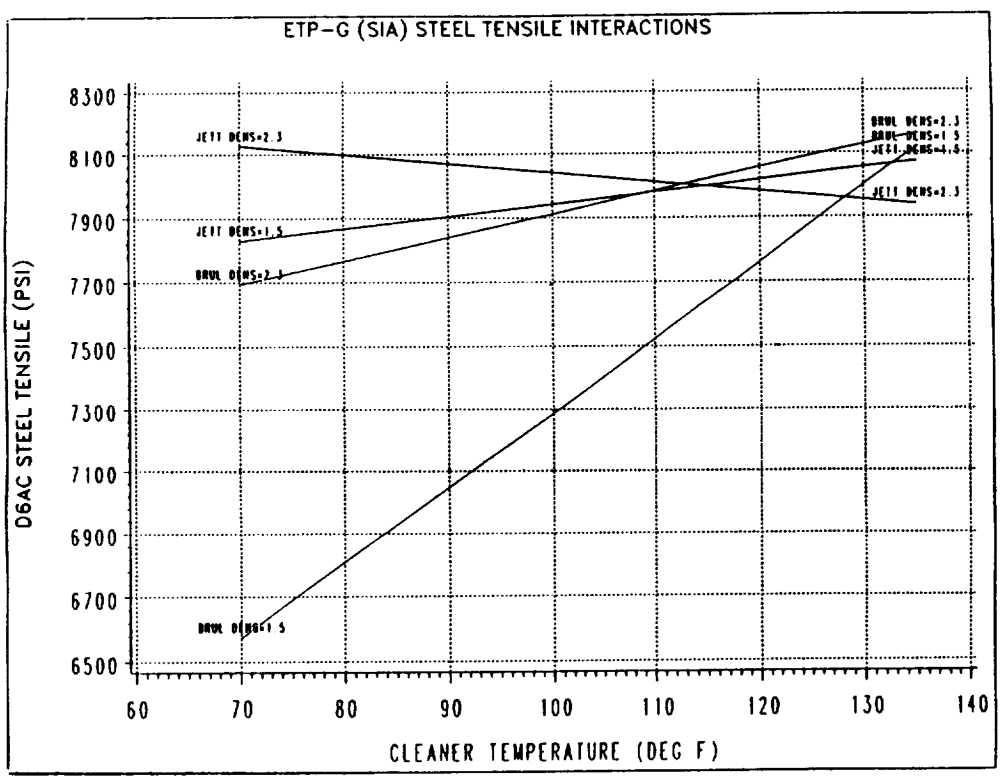


Figure 6

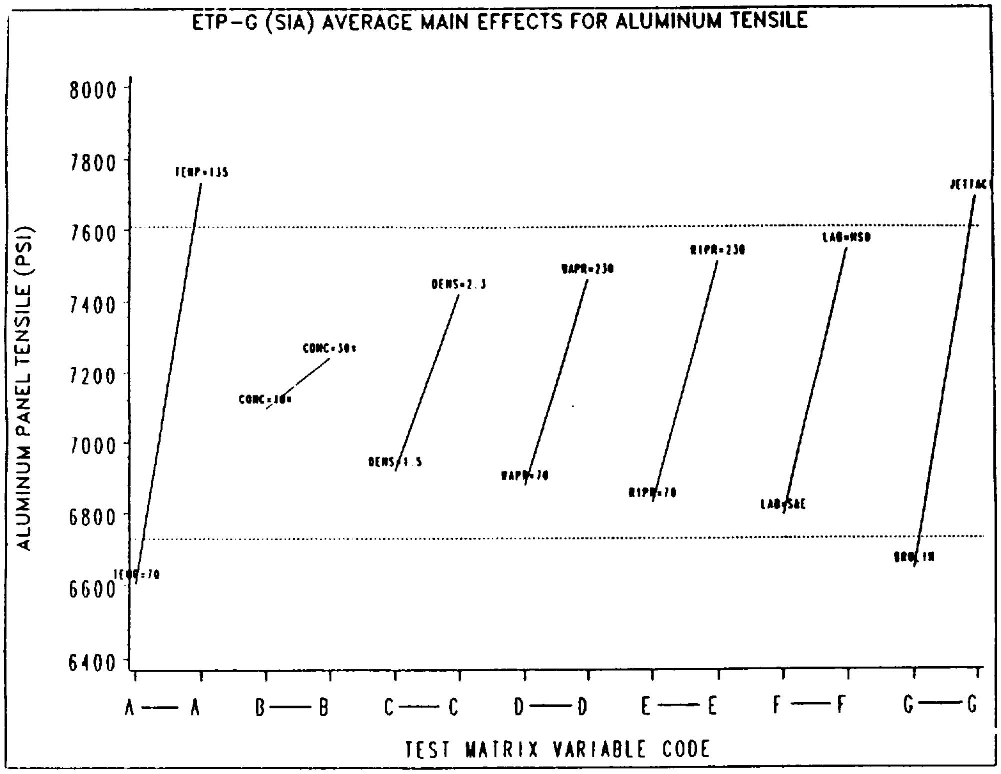
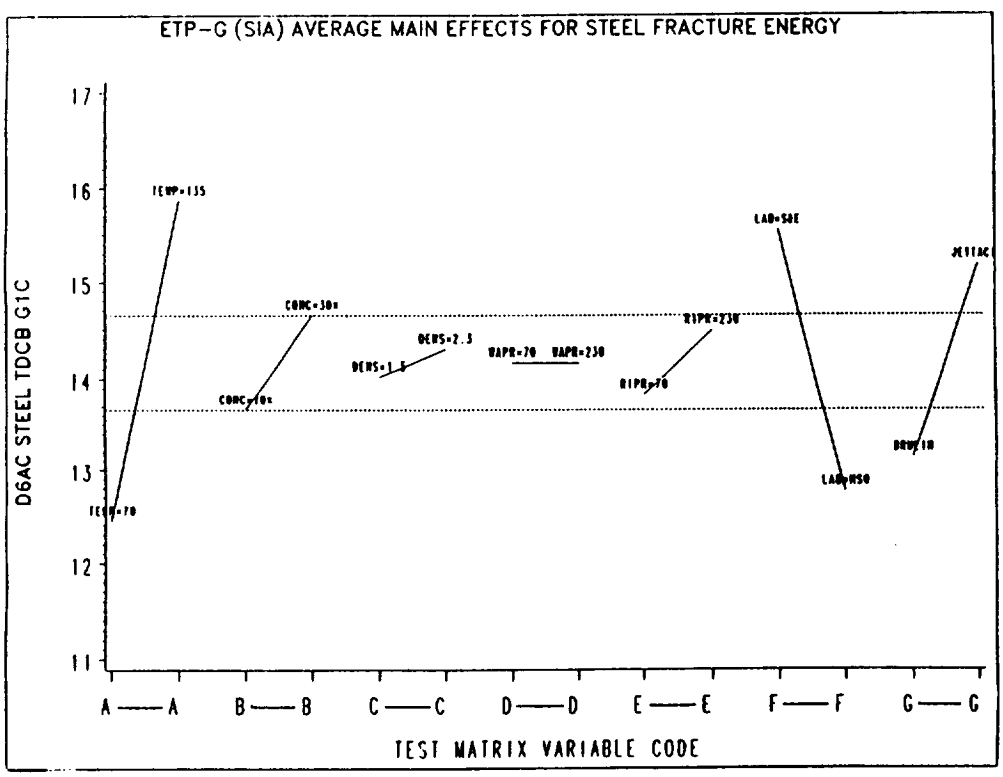


Figure 7



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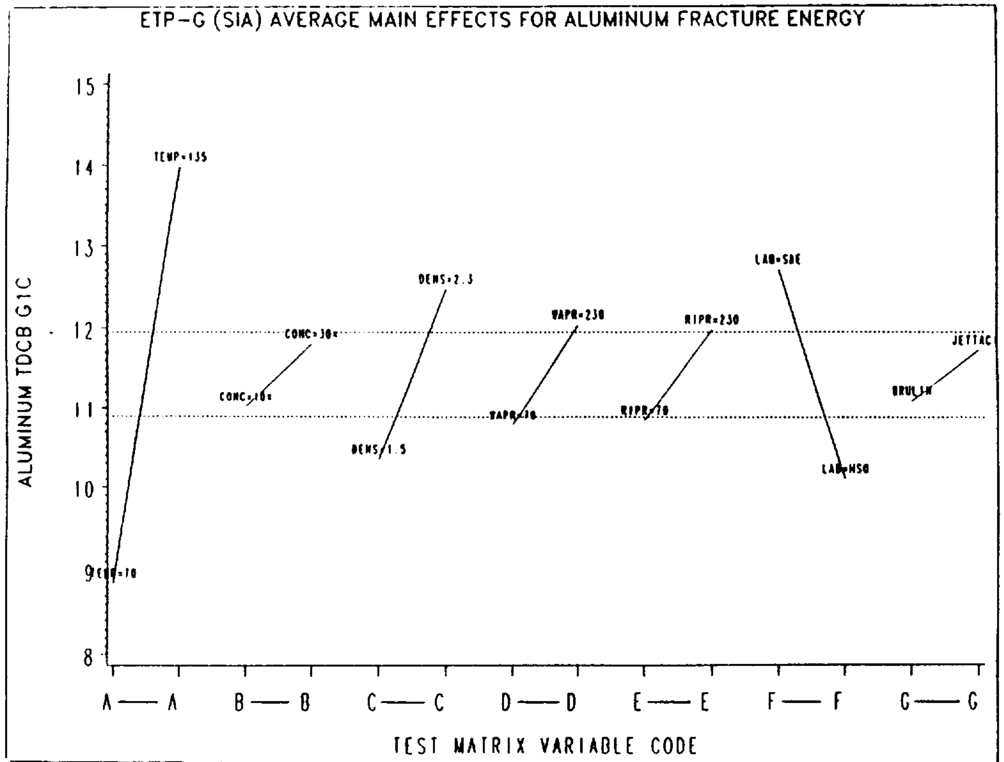

Figure 9

Table 4 Parameter Recommendations	
Parameter	**Recommendation**
(A) Cleaning Temperature	Fix temperature at 70° F for Jettacin, 135° F for Brulin 815 GD. No further testing needed.
(B) Cleaner Concentration	Fix at 10% for subsequent cleaning test and 15% for corrosion testing to allow for worst case.
(C) Wash Density	Fix at 2.5 gpm in subscale testing. Use comparable flow densities (gal/in^2) in full-scale facility.
(D) Wash Pressure	Fix at 100 psi. No further testing needed.
(E) Rinse Pressure	Fix at 100 psi. No further testing needed.
(G) Cleaner	Use Quality Function Deployment (QFD) method.
Potable vs Deionized Wash Water	Use deionized water.
Potable vs Deionized Rinse Water	Use deionized water.
Rinse Process	Further testing recommended.
Drying Process	Further testing recommended.