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EVALUATION OF PROPELLANT-LINER-INSULATION SAMPLES REPRESENTING --ETC(U)
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HEADQUARTERS
OGDEN AIR LOGISTICS CENTER
UNITED STATES AIR FORCE
HILL AIR FORCE BASE, UTAH 84406

EVALUATION OF PROPELLANT-LINER-INSULATION SAMPLES REPRESENTING STAGE II MOTORS AA21010 AA21015 AA21032 AND AA21053

See AD033689

PROPELLANT LAB SECTION

MANCP REPORT
NR 357 (76)

DECEMBER 1976

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report covers the testing of selected lined cartons of ANB-3066 propellant which were also tested by Aerojet Solid Propulsion Company (ASP). Comparisons are made with data from ASPC and reference to prior testing by Ogden ALC is included. No definitive conclusions have been reached at this time. The necessity for continued testing ;has been established which includes chemical as well as physical tests.

The statistical discussion is presented in the Appendix to this report.

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EVALUATION OF PROPELLANT-LINER-INSULATION SAMPLES REPRESENTING STAGE II MOTORS AA21010, AA21015, AA21032 AND AA21053

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ABSTRACT

This report covers the testing of selected lined cartons of ANB-3066 propellant which were also tested by Aerojet Solid Propulsion Company. A statistical analysis of the test data indicated no differences were found between the recent Ogden ALC data and the Aerojet data.

TABLE OF CONTENTS

I. INTRODUCTION:

Initial testing of propellant-liner-insulations bonds began at Ogden ALC in 1974 by testing high rate shear specimens. The rapidity with which these specimens failed when tested at 100 in/min under 600 psig N_2 indicated that there were problems with the bond system. Since there were many epoxy bond failures, bonding was assumed to be the basic problem.

The constant load shear and tensile specimens were cut in April 1974, but were stored at 0°F until February 1975, awaiting test equipment. For the tensile test, 2 inch diameter x 0.2 inch thick "poker chip" specimens were used instead of sleeved specimens. As the specimens were tested, it became obvious that some specimens were much stickier than others and the time-to-failure was very short for all specimens (see MANCP Report Nr 324 (75)).

In June 1975, four half-cartons, (AA21010 and AA21015 representing buff, relatively non-sticky blocks and AA21032 and AA21053 representing pink tacky liners), were sent to Aerojet Solid Propulsion Company (ASPC) for testing. The remaining half-cartons were stored at 0°F until receipt in May 1976 of Aerojet's report (Report 0162-06 SAA5-15, Addendum 1, 12 April 1976).

II. SCOPE:

This report presents results of tests conducted by Ogden ALC in June 1976 and compares these results with the data generated by ASPC and the earlier testing by Ogden ALC.

III. . TEST PLAN:

Three mechanical property and two chemical property tests were conducted according to the matrix shown in Figure 1.

IV. SAMPLES AND SPECIMEN CONFIGURATIONS:

The bond system, ANB-3066 propellant/SD-851-2 liner/V-45 rubber insulation, was obtained from one-half of 9 x 9 x 9 inch foil-lined carton for each of the four blocks tested. The blocks were removed from 0°F storage where they had been placed at the time the other half block was sent to Aerojet.

The position of each type of sample is shown in Figure 2. The test specimens were prepared in configurations similar to those shown in Figure 3. Individual specimen cross sectional areas are given in the tables of test results.

V. TECHNICAL DISCUSSION:

A. Bond Strength:

1. Constant Load Tensile and Shear:

a. Figures 4 and 5 graphically compare test data obtained to date. The solid line is the regression line for 45 - 49 months Ogden ALC data. The dashed line is the regression line for ASPC data. The 32 - 37 months Ogden ALC data has been added for visual comparison. The two regression lines appear to have different slopes, especially for AA21010 and AA21015, but statistically they were found to be the same.

b. Comparison of test results obtained by Ogden ALC with those of ASPC is shown in Figure 6. Individual test information is given in Figures 7 and 8. Examination of the types of failure shows that Ogden ALC is finding less cohesive failure than ASPC (when compared with the results in ASPC report) with far more failure in the propellant. One specimen of Block

AA21032 did not show tensile failure at 100,000 minutes, but finally failed at 117,939 minutes. This specimen was not used in analysis of variance. Both ASPC and Ogden ALC found failure between the liner and insulation on this carton. Statistics for constant load tests are shown in Figure 9.

2. Constant Rate Tensile Strength (Mini DPT):

The mini double plate tensile (mini DPT) specimen developed by ASPC is tested at a crosshead speed of 0.5 in/min. A correlation made by ASPC showed that the predicted strength for aged samples was much lower than for qualification standard DPT specimens. The actual test strength found by ASPC was lower than the predicted strength. A statistical comparison of the test results show no statistically significant differences between data obtained by Ogden ALC and ASPC (Figures 6 and 10).

B. Chemical Tests:

Swelling ratio of the liner was calculated by measuring 2 cm long x 1 to 2 mm wide specimens before and after swelling in toluene for 12 - 16 hours. Duplicate specimens were run (Figure 11). The swelling ratios which ranged from 1.72 to 1.80 (Figure 6) are comparable to the predicted value of 1.8, but are less than the values reported by ASPC.

Moisture content of the insulation adjacent to the liner was determined using a DuPont Instrument Model 26-321. Samples weighing 10 to 15 mg were used. The moisture content ranged from 1.383 to 1.537 (Figure 11) and these values are lower than those reported by ASPC.

There is no apparent pattern for either the swelling ratio or moisture. Where ASPC found a high swell ratio, high moisture and low mini DPT strength for block AA21053, Ogden ALC found a low swell ratio, low mini DPT strength and high moisture.

VI. CONCLUSIONS:

A. The regression analysis for predicted stress to cause failure at 100 minutes for constant load tests did not show a statistical difference between Ogden ALC (45 - 49 months) data and ASPC's (35 - 40 months) data.

B. The constant rate tensile (mini DPT specimen) test results were compared statistically with ASPC data. No statistical differences were shown between Ogden ALC data and ASPC data.

C. Results were consistent between the two facilities for the blocks tested.

D. A block to block analysis conducted on the four blocks showed no significant differences between blocks; the data obtained from relatively non-sticky blocks AA21010 and AA21015 could be said to be the same as data obtained from sticky blocks AA21032 and AA21053.

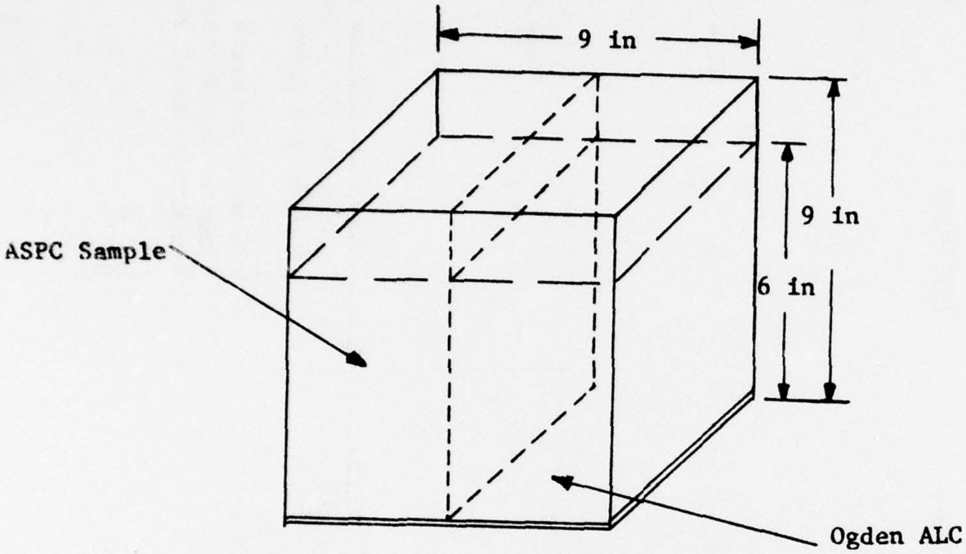
E. There is no reason to dispute the possibility of material degradation as reported by ASPC in their May 1976 report based on the analyses conducted by Ogden ALC.

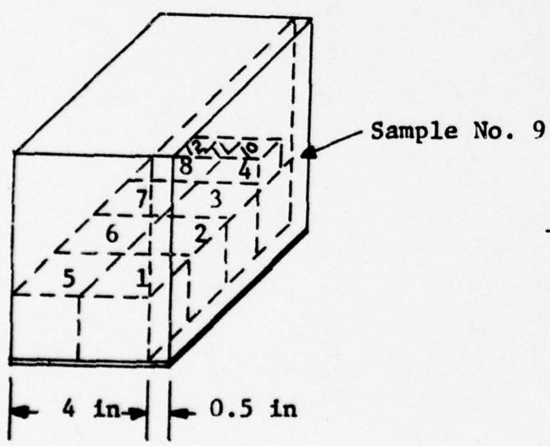
Propellant-Liner-Insulation

Test Mechanical Properties	Material	Description	No of Specimens	Remarks
Constant Load Tensile	Propellant-Liner-Insulation	Sleeved Poker Chip	5	20 - 65 psi stress
Constant Load Shear	Propellant-Liner-Insulation	Poker Chip	3	12 - 30 psi stress
Constant Rate Tensile	Propellant-Liner-Insulation	Mini DPT	3	0.5 in/min Cross-head Rate
Chemical				
Swelling	Liner	2.0 cm X 1-2 mm liner thickness		Swell in toluene for 12-16 hours at 77°F. Measure length before and after swelling.
Moisture Content	Insulation	0.1 - 0.2 in. dia		Measure moisture content using DuPont instruments Moisture Analyzer Model 26-321

ASPC Proposed Matrix

Figure 1

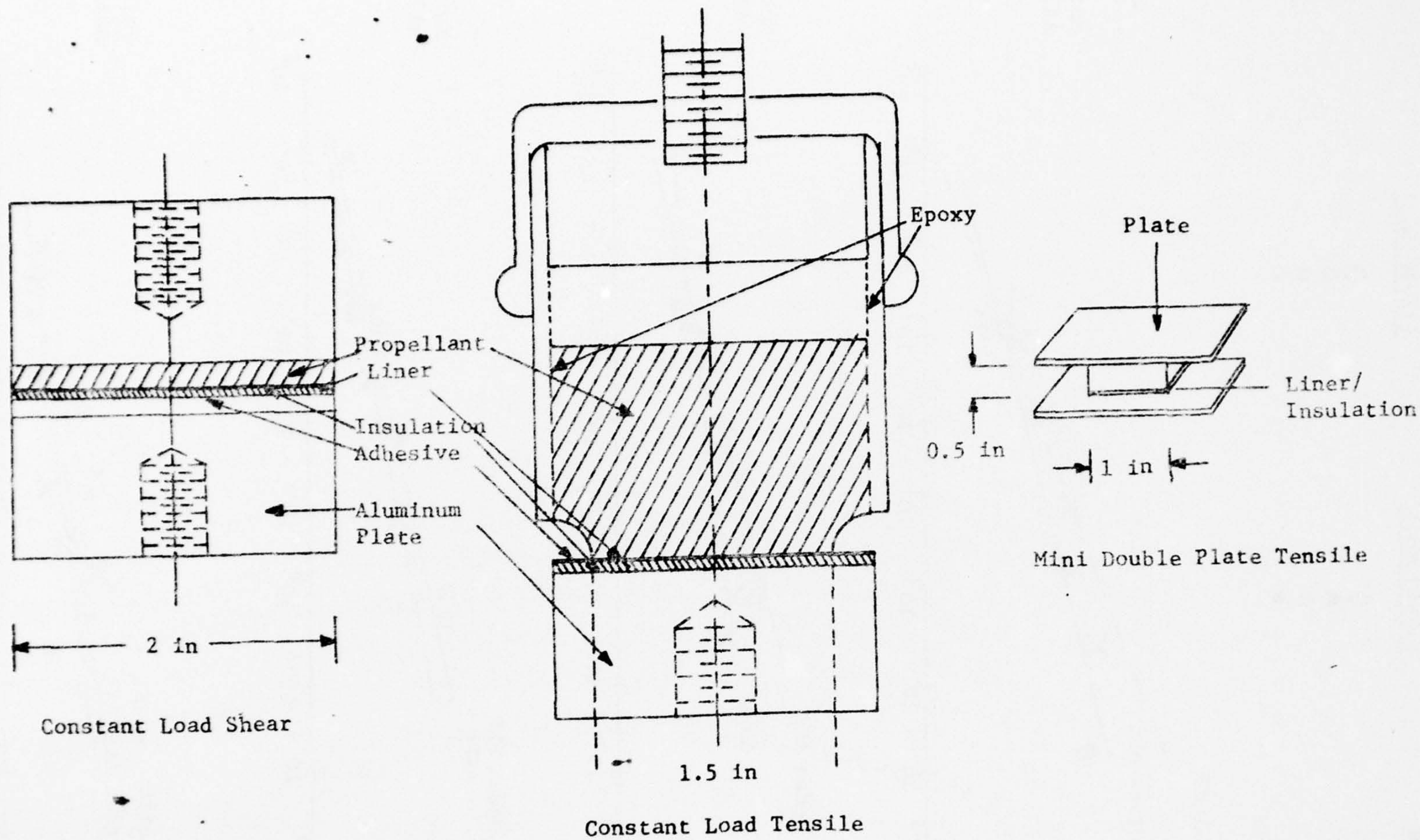




Sample No.	Type Specimen
1	Sleeve Poker Chip
2	Sleeve Poker Chip
3	Sleeve Poker Chip
4	Sleeve Poker Chip
5	Sleeve Poker Chip
6	Poker Chip
7	Poker Chip
8	Poker Chip
9	Swelling and Moisture Content
10	Mini DPT
11	Mini DPT
12	Mini DPT

Sample Location and Type

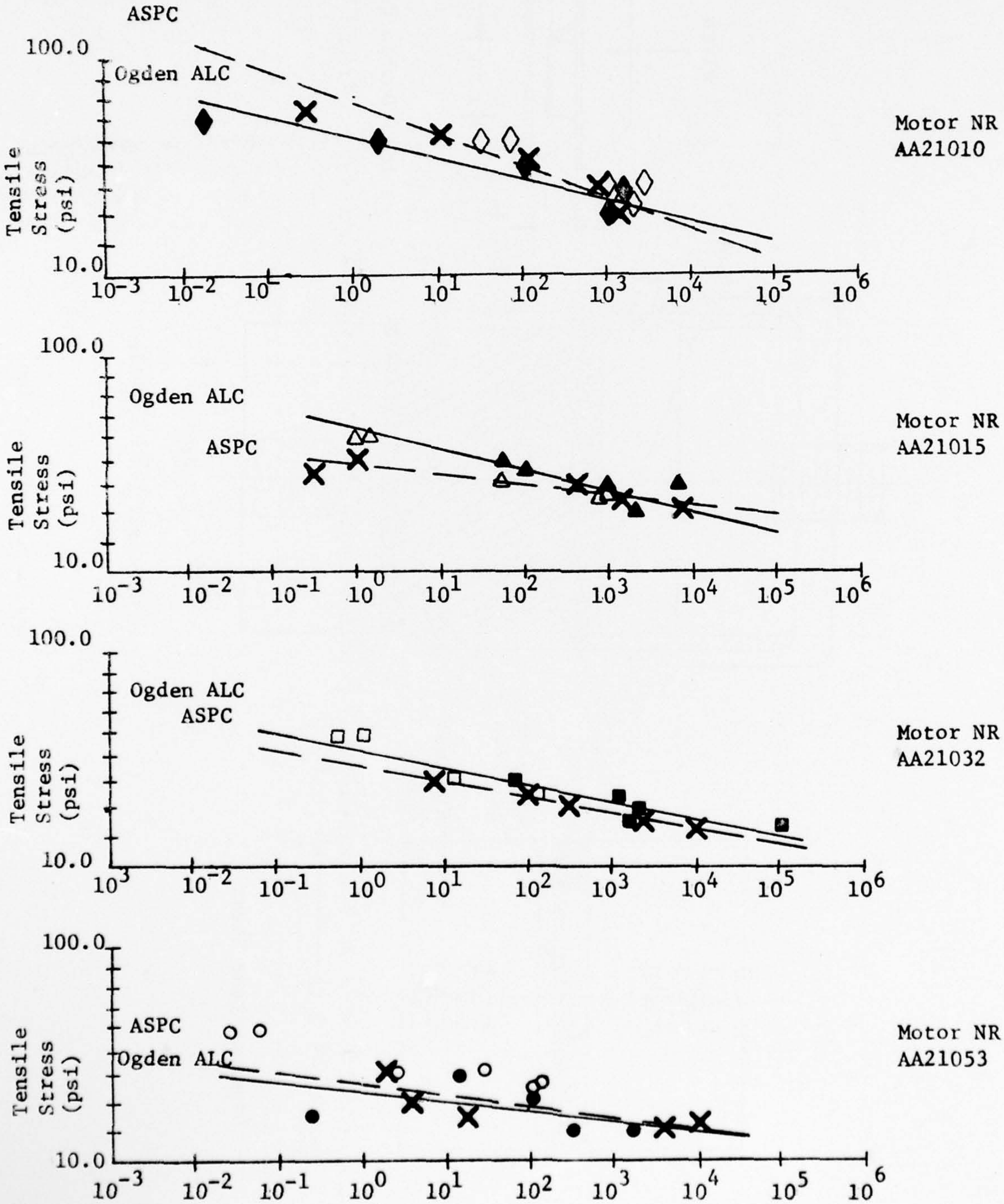
Figure 2



Specimen Configuration

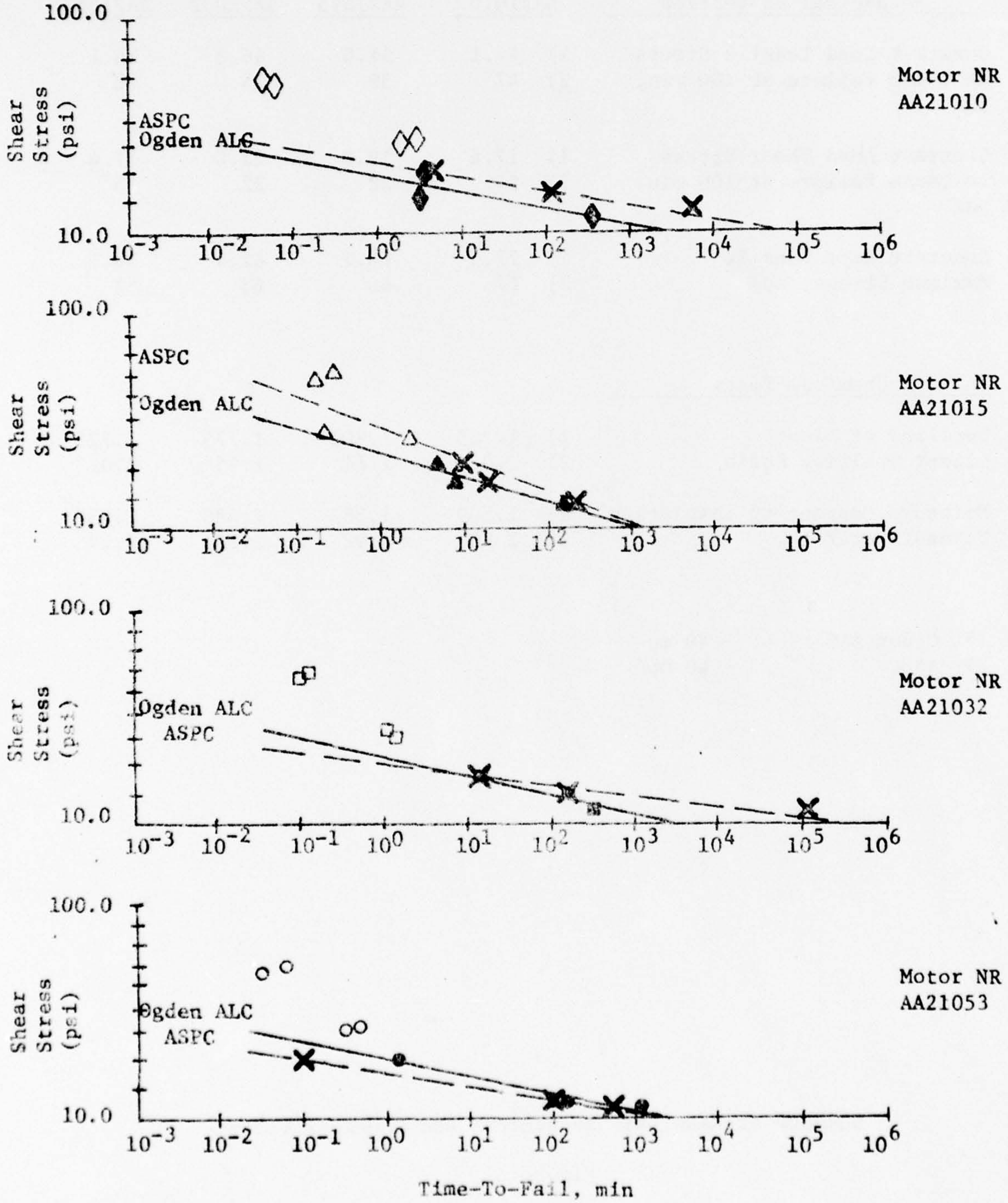
Figure 3

MOTOR NR	OALC (45-49 mo.)	OALC (32-37 mo.)	ASPC
AA21010	◆	◇	✕
AA21015	▲	△	✕
AA21032	■	□	✕
AA21053	●	○	✕



Time-To-Fail, min
Constant Load Tensile
Figure 4

Motor NR	OALC (45-49 mo.)	OALC (32-37 mo.)	ASPC
AA21010	◆	◇	×
AA21015	▲	△	×
AA21032	■	□	×
AA21053	●	○	×



Constant Load Shear

Figure 5

Mechanical Properties		Sample Identification AA21010	AA21015	AA21032	AA21053
Constant Load Tensile Stress to Cause Failure at 100 min, psi	1)	44.1	51.0	46.0	24.1
	2)	47	39	34	28
Constant Load Shear Stress to Cause Failure at 100 min, psi	1)	17.6	19.8	21.0	17.4
	2)	24	22	22	15
Constant Rate Tensile Maximum Stress, psi	1)	77.3	69.7	62.4	50.5
	2)	67	66	63	52
Chemical Tests					
Swelling of Liner Linear Swelling Ratio	1)	1.785	1.800	1.775	1.725
	2)	2.02	1.84	1.95	2.02
Moisture Content of Insulation Percent Water	1)	1.509	1.383	1.480	1.537
	2)	2.23	1.98	2.19	2.19

1) Ogden ALC 45 - 49 mo
2) ASPC 35 - 40 mo

Summary of Results: Mechanical and Chemical Tests

Figure 6

AF S/N Lot	Load (lbs)	A (in^2)	σ_m (psi)	t_f (min)	Type of Failure (1) CPP	CPL	CPR	P_2	P_1
AA21010	157	2.246	70	.03	90	5		5	
Lot 50	135	2.256	60	3.7	85	15			
	113	2.256	50	121.0	20	15		65	
	41	1.021	40	2562.0				100	
	67	2.238	30	1512.0	30	70			
AA21015	114	2.275	50	72.0	80	20			
Lot 50	102	2.264	45	112.0	30	70			
	91	2.267	40	7324.0	40	60			
	50	1.421	35	964.0	10	90			
	68	2.283	30	3930.0			Bond Failure		
AA21032	90	2.256	40	83.0	50	45		5	
Lot 52	79	2.256	35	1842.0	50	50			
	68	2.270	30	3706.0	60	40			
	26	1.048	25	2490.0	50	40		10	
	50	2.270	22	117939.0					
AA21053	90	2.243	40	11.18	45	52		3	
	67	2.243	30	134.0	3	12	85		
	56	2.243	25	.43	5	10	85		
	33	1.647	20	2971.0		85	10		
	45	2.264	20	532.0	40	40		20	5

(1) See Figure 12 for definition of terms

Constant Load Tensile

Figure 7

AF S/N Lot	Load (lbs)	A (in^2)	σ_m (psi)	t_f (min)	(1) Type of Failure CPP	CPL	CPR	P
AA21010	94	3.126	30	5.9	30	68	2	
Lot 50	68	3.113	22	5.2	30	68		2
	13	.865	15	569.0	30	70		
AA21015	100	3.120	32	6.9	20	70	10	
Lot 50	78	3.113	25	8.9	30	60	10	
	29	1.613	18	276.0	30	70		
AA21032	78	3.113	25	25.2	35	65		
Lot 52	62	3.113	20	268.0	40	60		
	17	1.150	15	501.0	20	75		5
AA21053	93	3.113	30	2.03	35	40	25	
Lot 54	47	3.120	15	230.0	25	65	10	
	17	1.444	12	1800.0		100		

Constant Load Shear

Figure 8

		Intercept		Slope					Predicted Stress to Cause Failure					
									1 min. 95% Confidence Limits			100 min. 95% Confidence Limits		
Test	Motor #	Mean a	Std Dev s_a	Mean b	Std Dev s_b	Std (1) Error syx	Correl Coeff r	# of Spec n	1	UL	LL	100	UL	LL
Constant Load Tensile	AA21010	22.801	6.059	-12.652	3.595	1.045	-0.767	5	63.41	103.91	34.69	44.10	75.44	26.60
	AA21015	15.175	6.2119	- 7.715	3.890	0.682	-0.753	5	92.67	145.91	28.06	51.02	75.93	27.40
	AA21032	17.304	4.2233	- 9.411	2.8618	0.603	-0.885	5	68.98	113.82	28.99	42.28	62.07	24.71
	AA21053	10.060	8.386	- 5.833	5.904	1.506	-0.496	5	53.04	86.18	11.16	24.09	62.72	10.41
Constant Load Shear	AA21010	10.485	3.704	- 6.810	3.604	0.590	-0.884	3	34.64	129.42	7.52	17.61	60.70	5.57
	AA21015	10.599	3.647	- 6.630	2.628	0.216	-0.930	3	39.69	105.55	13.04	19.81	45.89	9.05
	AA21032	9.514	3.081	- 5.680	2.379	0.140	-0.922	3	47.31	189.71	9.06	21.03	42.04	10.29
	AA21053	11.017	0.586	- 7.268	0.467	0.137	-0.998	3	32.79	43.14	24.802	17.40	21.60	14.02

Regression Model: log (time-to-failure) = a + b (log stress, psi)

(1) Standard error is stated in terms of log time since time is dependent variable

Summary of Regression Analysis, Stress vs. Time-to-Failure

Figure 9

AF S/N Lot		CSA	S_m	t_m	Type of Failure (1) CPP	CPL	CPR	P_1	P_2
AA21010	#10	1.057	75.66	.06	95			5	
Lot 50	#11	.993	84.81	.06	98	2			
	#12	1.075	71.34	.06	100				
			$\bar{X}$ 77.27	$\bar{X}$ .06					
AA21015	#10	1.057	66.25	.06	100*				
Lot 50	#11	.985	66.51	.223	100				
	#12	1.028	76.35	.053	100**				
			$\bar{X}$ 69.70	$\bar{X}$ .112					
AA21032	#10	1.074	63.33	.27	65	35			
Lot 52	#11	1.012	65.0	.28	60	30		10	
	#12	1.086	58.92	.24	70	25		5	
			$\bar{X}$ 62.42	$\bar{X}$ .263					
AA21053	#10	1.021	53.67	.31	40	45	15		
Lot 54	#11	1.096	46.52	.36	50	40	8	2	
	#12	.977	51.20	.26	49	50	1		
			$\bar{X}$ 50.46	$\bar{X}$ .31					

* Liner so thin that insulation was exposed about 5%
** Approximately 10% of insulation was not lined
(1) See Figure 12 for definition of terms

Mini - DPT Tensile Strength

Figure 10

S/N	Lot	Moisture, % Insulation	Liner Linear Swelling Ratio
AA21010	50	1.535 - 1.483	1.84 - 1.73
AA21015	50	1.361 - 1.405	1.81 - 1.79
AA21032	52	1.462 - 1.497	1.82 - 1.73
AA21053	54	1.525 - 1.549	1.72 - 1.73

Moisture and Swelling

Figure 11

CPP	Adhesive Propellant/Liner
CPL	Cohesive in Liner
CPR	Adhesive Liner/Insulation
P_1	Cohesive in Propellant at Interface
P_2	Cohesive in Propellant

Definition of Types of Bond Failure Used at Ogden ALC

Figure 12

APPENDIX

STATISTICAL DISCUSSION

A. Bond Strength:

1. Constant Load Tensile:

Initial comparison of Ogden ALC and ASPC bond tensile data was made using regression analysis. Time-to-fail data were regressed on stress and comparisons made to test for significance between regression coefficients. The stress levels required to effect failure at 100 minutes were also compared for similarity.

The constant load tensile specimens were subjected to varied levels of stress in order to obtain times-to-failure over a broad spectrum. The wide range of failure times was compensated for by transforming the test data logarithmically and performing a log time-to-fail vs log stress regression. The regression equation that resulted from this transformation is: log (time-to-failure) = a + b (log stress, psi).

Predicted values of stress at 100 minutes were obtained by solving the regression equation for stress. In all cases, the predicted values of stress for the ASPC data are within the 95% confidence interval computed for the Ogden ALC data. The regression analyses indicate no statistical differences between the regression slopes and intercepts for the four blocks.

The specimens tested by ASPC ranged in age from 35 to 40 months. Those tested by Ogden ALC ranged from 45 to 49 months. Due to this time lapse between testing, covariance analyses were performed to test for possible degradation due to material aging. For these analyses, stress level and material age were assigned as covariates. Using this technique, it was possible to adjust the test data with respect to one covariate.

(stress or age), then test directly for the effect of the remaining covariate on time-to-failure. In addition, the analyses of covariance also allowed motor-to-motor and facility-to-facility comparisons. These comparisons provided a method to determine if any statistical differences existed among blocks and/or among test facilities. Due to stress level the results of these analyses indicate a significant effect on time-to-failure. Material age has no significant effect on times-to-failure and no significant differences due to motor or test facility effects. These results are consistent for all motors.

In summary, the comparison of Ogden ALC and ASPC bond tensile strength and times-to-failure, indicate no statistically significant differences in the test data from the two facilities.

Analysis of variance and regression analysis results are given in Tables A-1 and A-2. All statistical test were conducted at the 5% significance level. Comparative graphs showing Ogden ALC and ASPC data and the respective regression lines are shown in Figure 4.

2. Constant Load Shear:

Statistical analyses identical to those conducted for constant load tensile were performed on these data. Material age of the bond shear specimens was similar to the bond tensile specimens.

Analysis of bond shear data indicate statistically similar values of shear stress required for failure at 100 minutes as predicted by Ogden ALC and ASPC. These results are consistent for all motors at the 95% confidence level. As with the bond tensile results, there were no significant differences among blocks or test facilities. Analysis of variance and regression results are given in Tables A-3 and A-4. Comparative graphs are shown in Figure 5.

3. Constant Rate Tensile:

Mini double plate tensile data obtained by Ogden ALC were compared with corresponding data obtained by ASPC. At the present time the quantity of test data was insufficient to perform regression analyses. The test parameters analyzed for these data were maximum stress and time-to-maximum stress.

Bartlett's Chi Square test[1] was performed to determine if levels of variability in the data among blocks were similar. This test was applied to data from both test facilities separately. The test results indicate homogeneity of variance among motors tested at each facility with the exception of block AA21010 tested at Ogden ALC. Time-to-maximum stress data reported by Ogden ALC for this motor showed no variation among replicate test specimens.

Standard "F" and 't' tests were used to compare the variance and means for each block between test facilities. The results of the "F" and 't' tests are consistent for all motors and indicate no statistically significant differences between data obtained by Ogden ALC and ASPC. In all cases Ogden ALC constant rate tensile means are within 2 standard deviations of the ASPC means.

Comparison of constant rate tensile data are summarized in Table A-5.

1. Ostle, B., Statistics in Research. Second Edition. The Iowa State University Press, Ames, Iowa, 1966.

TABLE A-1

Analysis of Variance
Constant Load Tensile

Source of Variation	df	Sum of Squares	Mean Square
Mean	1	93.642	
Stress	1	0.32166	
Age	1	0.041223	
Motor AA21010 vs. Motor AA21015, AA21032, AA21053	1	0.0166485	
Motor AA21015 vs. Motor AA21032, AA21053	1	0.122635	
Motor AA21032 vs. Motor AA21053	1	0.214779	
Ogden ALC vs. ASPC	1	0.0181803	
Motors	3	0.24969	0.0832312
Facilities	1	0.0065586	0.0065586
Motor x Facility	2	0.0117434	0.00587169
Error	26	0.14188	0.0054568
Total	39	94.483	

TABLE A-2

Analysis of Variance Summary
Constant Load Tensile

Source of Variation	F-Ratio	df for F-Ratio
Stress	45.851 *	1, 26
Age	2.1562	1, 26
Motor AA21010 vs. Motors AA21015, AA21032, AA21053	0.12513	1, 26
Motor AA21015 vs. Motors AA21032, AA21053	0.0250573	1, 26
Motor AA21032 vs. Motor AA21053	1.22860	1, 26
Ogden ALC vs. ASPC	0.77390	1, 26

* Significant at $\alpha = .05$

TABLE A-3

Analysis of Variance
Constant Load Shear

Source of Variation	df	Sum of Squares	Mean Square
Mean	1	87.055	
Stress	1	25.653	
Age	1	0.17413	
Motor AA21010 vs. Motors AA21015 AA21032, AA21053	1	0.26672	
Motor AA21015 vs. Motors AA21032, AA21053	1	0.91150	
Motor AA21032 vs. Motor AA21053	1	5.0517	
Ogden ALC vs. ASPC	1	0.35404	
Motors	3	1.4827	0.49423
Facilities	1	0.0	0.0
Motor x Facility	3	2.9905	0.99684
Error	11	1.9209	0.17463
Total	24	125.86	

TABLE A-4

Analysis of Variance Summary
Constant Load Shear

Source of Variation	F-Ratio	df for F-Ratio
Stress	133.39 *	1, 11
Age	3.1562	1, 11
Motor AA21010 vs. Motors AA21015, AA21032, AA21053	0.002315	1, 11
Motor AA21015 vs. Motors AA21032, AA21053	4.1712	1, 11
Motor AA21032 vs. Motor AA21053	2.8377	1, 11
Ogden ALC vs. ASPC	4.7397	1, 11

* Significant at $\alpha = .05$

TABLE A-5

Comparison of Test Results
Mini Double Plate Tensile

Maximum Stress

Motor Nr	Ogden ALC Data n	$\bar{X}$	s^2	ASPC Data n	$\bar{X}$	s^2	F-Ratio	t
AA21010 Lot 50	3	77.27	47.304	3	66.90	26.893	2.096	0.7162
AA21015 Lot 50	3	69.70	33.151	3	66.40	66.280	1.999	0.1910
AA21032 Lot 52	3	62.42	9.867	3	62.80	24.790	2.512	0.0372
AA21053 Lot 54	3	50.46	13.188	3	52.20	5.290	2.493	0.2337
χ^2 for equality of variance among motors	1.5746 = N.S. α = .05; 3 df			2.7645 = N.S. α = .05; 3 df				

Time-to-Max. Stress

Motor Nr	Ogden ALC Data n	$\bar{X}$	s^2	ASPC Data n	$\bar{X}$	s^2	F-Ratio	t
AA21010 Lot 50	3	0.060	0.0	3	0.110	0.0161		0.3182
AA21015 Lot 50	3	0.112	0.0093	3	0.100	0.0108	1.1675	0.0489
AA21032 Lot 52	3	0.263	0.0004	3	0.240	0.0010	2.3953	0.3475
AA21053 Lot 54	3	0.310	0.0025	3	0.310	0.0021	1.1737	0.0
χ^2 for equality of variance among motors	72.097 = Sig α = .05; 3 df			4.251 = N.S. α = .05; 3 df				