

NATIONAL BUREAU OF STANDARDS REPORT

10 403

EVALUATION OF THE "TYPE A" POLYESTER ADHESIVE USED
IN THE MATERIAL SYSTEMS CORP. HOUSING



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Abstract

The Type A polyester adhesive used in the Materials Systems Corporation Housing System was evaluated using lap type specimens. The specimens were grouped according to adhesive thickness in the area of the lap joint because of the wide range of observed thicknesses. Tensile shear tests were conducted on the specimens at various temperatures up to 180°F and at various rates of loading. The effect of temperature, adhesive thickness, and accelerated laboratory aging on the bond strength was determined. Both increasing temperature and adhesive thickness resulted in decreasing bond strength while the laboratory aging test exhibited very little effect on bond strength.

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EVALUATION OF MSC TYPE A ADHESIVE

INTRODUCTION

A study was initiated to evaluate the adhesive (Type A, polyester) used in the Materials Systems Corporation Housing System. The system consists of two glass-fiber reinforced polymer composite sheets sandwiched together by a corrugated core material of similar composition. Type A adhesive is used to bond the core to the sandwich panel facings. The objectives of the study were to (1) determine the effect of temperature on bond strength, (2) determine the effect of adhesive thickness on bond strength, (3) determine the effect of accelerated laboratory aging on the bond strength, and (4) estimate the expected durability of the adhesive.

TESTS USING NOTCH TYPE 1

Test specimens 1/2 inch wide and seven inches long were cut from the bonded areas. Notches were cut in the panels with a band saw in such a way as to provide a lap panel with an effective area of 1/2 inch². The first eighteen specimens were notched so that the thickness of plastic provided the only connection between the bonded area of interest and the grip areas at the end of the panels. This type of notch is illustrated by Figure 1 and referred to in the text as "notch type" 1.

The samples were tested in tensile shear using a Tinius-Olsen testing machine with a load application rate of approximately 300 lb/min and at temperatures of 73°F, 120°F, and 180°F. These results are summarized in Table 1. All those samples tested at room temperature

failed in the outer composite layer and parallel to the adhesive bond. The higher temperature tests yielded failure of the adhesive bond to the core composite.

TESTS USING NOTCH TYPE 2

Approximately one hundred additional specimens were cut in a manner similar to the first eighteen but with the exception that the band saw notch at position A was made through the composite material but not through the adhesive. This type of notch will be referred to as notch type 2 and is illustrated in Figure 2. The purpose of this type cut was to provide additional stability to the composite material that had previously failed in room temperature tests. Tensile shear tests of these samples at room temperature did not consistently yield failure at the adhesive bond, although three of twelve samples did fail at the bond. As for the other nine samples - three failed by an internal separation of the outer composite layer, as before, and the other six failed as a result of shear of the composite material at notch B. These results are presented in Table 2.

Tests conducted at temperature increments of five degrees from 75° to 110°F indicated that adhesive bond failure could be obtained consistently at 105°F. Therefore, temperatures of 105°, 120°, 150° and 180°F were chosen for the tests to determine curves of load at failure versus temperature.

Large variations in adhesive thickness at various sampling locations and within some individual samples necessitated the grouping

of samples by adhesive thickness. The thickness increments chosen were: < 1.0mm (Group A), 1.0 - 2.0mm (Group B), 2.0 - 3.0mm (Group C), and 3.0 - 5.0mm (Group D).

Approximately seventy samples with notch Type 2 were then tested in tensile shear at the various chosen temperatures. The results of these tests are presented in Table 3. The failure of all these samples was at the adhesive bond to the core composite material. The effect of increasing temperature on the adhesive bond strength is illustrated in Figure 3. Since the composite material failed in most room temperature tests, the actual adhesive bond strength at 73°F is somewhat greater than the points indicate. As would be expected, the adhesive bond strength decreases significantly with increasing temperature. Figure 4 is a graph of adhesive bond strength versus adhesive thickness. Substantial differences in bond strength may be noted at the various thicknesses with the thinner adhesive application yielding higher bond strengths. The adhesive thickness effect is however, less than the temperature effect.

STRESS-RUPTURE ENDURANCE LIMIT

Additional samples with notch type 2 were tested in tensile shear at various loading rates. The test method of Prot^{1,2} for estimating the stress-rupture endurance limit was then applied to the data. Table 4 summarizes the loading rates used and the data obtained are presented in Table 5. The data from samples requiring approximately 0.4 minutes to fail are extracted from Table 3. Figure 5 graphically

illustrates the data in Table 5 for Group B. The data for other groups provide similar curves.

The data obtained from the Group B samples at 150°F were plotted as described in the modified Prot method described by Boller. The basic equation is $\sigma = E + Ka^n$.

Where σ = the failing load

E = the estimated endurance limit

a = the rate of progressively increasing stress

K = a constant

$$n = \log \frac{\sigma_3 - \sigma_2}{\sigma_2 - \sigma_1}$$

$$\log \frac{a_2}{a_1}$$

The observed values of σ were plotted at their respective values of a^n on Cartesian coordinates. A line drawn through the points to intersect the ordinate at zero stress rate should give an estimated Prot endurance limit. A line through the points from this data did not intersect the ordinate in the positive region indicating the change of failing stress with rate of loading to be too large for this particular system for the Prot test to be applicable.

An alternate method to estimate the endurance limit consists of subjecting a successive number of specimens to different stress levels to determine the time that a stress level can be maintained. These tests will be conducted and the results reported in the near future.

LABORATORY AGING TEST

Sixteen samples with notch type 2 were subjected to the ASTM Laboratory Aging Test, C-481, Cycle A, before obtaining tensile shear values. One cycle of the aging test consists of the following steps:

- 1) Total immersion in water at $120 \pm 3^{\circ}\text{F}$ for one hour.
- 2) Spray with steam and water vapor at $200 \pm 5^{\circ}\text{F}$ for 3 hours.
- 3) Store at $10 \pm 5^{\circ}\text{F}$ for 20 hours.
- 4) Heat at $210 \pm 3^{\circ}\text{F}$ for 3 hours.
- 5) Spray with steam and water vapor at $200 \pm 5^{\circ}\text{F}$ for 3 hours.
- 6) Heat in dry air at $210 \pm 3^{\circ}\text{F}$ for 18 hours.

The test consists of repeating the cycle six times and equilibrating the samples at $73 \pm 2^{\circ}\text{F}$ and $50 \pm 2\%$ relative humidity to constant weight. The tensile shear tests were carried out immediately following the equilibration period. The tensile shear values obtained are summarized in Table 6. Table 7 contains a comparison of the average values obtained with and without laboratory aging. This comparison indicates that the aging test did not significantly reduce the bond strength of the adhesive. The values at room temperature are again indicative of composite failure rather than adhesive failure.

REFERENCES

1. PROT, E. Marcel, "Fatigue Testing Under Progressive Loading, A New Technique for Testing Materials", Wright Air Development Center Technical Report 52-148.
2. BOLLER, K. H., "Application of Prot Test Method to Stress-Rupture Curves of Glass-Reinforced Plastic Laminates", Forest Products Laboratory Report #2118, September 1958.

TABLE 1

TENSILE SHEAR RESULTS OF INITIAL SAMPLES

<u>SAMPLE NUMBER</u>	<u>ADHESIVE THICKNESS</u> (mm)	<u>TEST TEMP</u> (°F)	<u>LOAD AT FAILURE</u> (lbs)	<u>TYPE OF FAILURE</u>
1-1	2.0-2.5	73	182	Separation within outer composite parallel to bond
2-1	2.0	73	132	"
4-1	2.5-3.0	73	161	"
11-1	2.0	73	176	"
16-1	1.0	73	165	"
18-1	1.25-1.50	73	195	"
8-1	2.5	120	130	Adhesive bond to core composite
9-1	2.0-2.5	120	135	"
10-1	2.0	180	101	Adhesive bond to core composite
13-1	1.5	180	126	"
17-1	1.5-1.75	180	73	"

TABLE 2

TENSILE SHEAR RESULTS AT 73°F USING NOTCH TYPE 2

<u>SAMPLE NUMBER</u>	<u>ADHESIVE THICKNESS</u> (mm)	<u>LOAD AT FAILURE</u> (lbs)	<u>TYPE OF FAILURE</u>
1-2	2.5-4.0	171	Adhesive bond to core composite
2-2	2.0-3.0	115	"
3-2	2.5	213	"
19	1.0	220	Shear composite at Notch B
10	1.5	305	Separation within outer composite parallel to bond
12	1.5-1.75	264	Shear composite at Notch B
13	1.0-2.5	237	"
15	2.0	168	"
9	2.0-2.5	283	"
4	3.5-4.0	154	"
157	0.5-1.25	*284	Separation within outer composite parallel to bond
187	0.75	351	"

*Some weakening in composite noted before test

TABLE 4

LOADING RATES FOR PROT METHOD

<u>RATE OF PULL</u> (cm/min)	<u>APPROXIMATE</u> Time for Sample Failure (min)	<u>ESTIMATED</u> <u>LOADING RATE</u> (lb/min)
.5	0.4	300
.05	3.5	25
.005	28-30	2

TABLE 4

LOADING RATES FOR PROT METHOD

<u>RATE OF PULL</u> (cm/min)	<u>APPROXIMATE</u> Time for Sample Failure (min)	<u>ESTIMATED</u> <u>LOADING RATE</u> (lb/min)
.5	0.4	300
.05	3.5	25
.005	28-30	2

TABLE 5

TENSILE SHEAR STRENGTH AT VARIOUS LOADING RATES

SAMPLE NUMBER	GROUP A (< 1 mm)				GROUP B (1.0-2.0mm)				GROUP C (2.0-3.0mm)					
	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	TIME TO FAILURE (min)	LOAD (lbs)	SAMPLE NUMBER	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	TIME TO FAILURE (min)	LOAD (lbs)	SAMPLE NUMBER	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	TIME TO FAILURE (min)	LOAD (lbs)
159	0.75	120	0.4	220	See Table 3	1.5-2.5	120	0.4	$\bar{X}=150\pm 23$	See Table 3	2.0	105	0.4	$\bar{X}=134\pm 17$
160	0.25-0.75	120	0.4	242	114	1.5-2.5	120	3.1	84	120	2.0	105	1.9	95
188	0.50	120	0.4	230	116	1.5-2.0	120	3.0	94	122	2.0	105	2.0	85
				$\bar{X}=231\pm 11$					$\bar{X}=89\pm 7$					$\bar{X}=90\pm 7$
150	0.5-0.75	120	3.6	192	121	1.5	120	25.8	78	123	2.0	105	19.3	105
151	0.5-1.0	120	3.6	176	128	1.5	120	24.8	67	124	2.5	105	14.8	52
				$\bar{X}=184\pm 11$					78					
153	0.3-1.5	120	32.4	120	132	1.5-2.0	120	26.4	78	See Table 3	2.5-3.0	120	0.4	$\bar{X}=118\pm 9$
154	0.5-0.75	120	27.6	137	See Table 3	1.5-2.0	150	0.4	$\bar{X}=74\pm 6$	130	2.5-3.0	120	3.4	62
				$\bar{X}=129\pm 12$					$\bar{X}=115\pm 16$	131	2.0	120	3.2	56
178	0.75-1.0	180	0.4	92	152	1.0-1.5	150	3.2	86	134	2.5-3.0	120	3.4	70
190	0.5	180	0.4	87	155	1.0-1.5	150	3.0	66	133	2.5-3.5	120	4.0	62
				$\bar{X}=90\pm 4$					$\bar{X}=76\pm 7$					$\bar{X}=63\pm 6$

Continued on Next Page

TABLE 5 (Cont'd)

TENSILE SHEAR STRENGTH AT VARIOUS LOADING RATES

GROUP A (< 1 mm)				GROUP B (1.0-2.0mm)				GROUP C (2.0-3.0mm)						
SAMPLE NUMBER	ADHESIVE THICKNESS (mm)	TEST TEMP ($^{\circ}$ F)	TIME TO FAILURE (min)	LOAD (lbs)	SAMPLE NUMBER	ADHESIVE THICKNESS (mm)	TEST TEMP ($^{\circ}$ F)	TIME TO FAILURE (min)	LOAD (lbs)	SAMPLE NUMBER	ADHESIVE THICKNESS (mm)	TEST TEMP ($^{\circ}$ F)	TIME TO FAILURE (min)	LOAD (lbs)
162	0.75	180	2.6	67	156	1.0-2.0	150	29.6	56	125	2.0	120	30.8	81
165	0.5-0.75	180	2.8	70 69 \pm 2	158 163	0.75-1.25 0.5-1.5	150 150	29.4 24.4	70 50 $\bar{X}=59\pm 10$	126 127	2.0 2.5-3.0	120 120	27.2 27.6	70 36
173	0.75-1.0	180	25.6	53	See Table 3 164 167 168	1.25 1.0-1.75 1.0-1.25	180 180 180	0.4 2.2 2.2 2.4	$\bar{X}=76\pm 4$ 55 60 49					
					169 171	1.25 0.5-2.0	180 180	21.4 19.6	45 46 $\bar{X}=55\pm 6$ $\bar{X}=46\pm 1$					

TABLE 6

TENSILE SHEAR OF MSC PANELS FOLLOWING

ASTM C-481, CYCLE A TEST

GROUP B (1.0-2.0 mm)			GROUP C (2.0-3.0 mm)			GROUP D (3.0-5.0 mm)					
#	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	LOAD (lbs)	#	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	LOAD (lbs)	#	ADHESIVE THICKNESS (mm)	TEST TEMP (°F)	LOAD (lbs)
102	1.5-1.75	73	247	101	2.0	73	181	204	3.5	73	158
103	1.5	105	186	104	2.0	105	148	129	3.0	105	104
110	1.5-2.0	120	132	105	2.0-2.5	105	155				
111	1.5-2.0	150	96	106	2.0-3.0	120	126				
				107	2.0	120	134				
				108	2.0-2.5	150	84				
				118	1.75-2.5	150	78				
112	1.75-2.0	180	62	113	2.0-2.5	180	78				
				117	2.0-2.5	180	56				

TABLE 7

COMPARISON OF TENSILE SHEAR VALUES
WITH AND WITHOUT LABORATORY AGING

GROUP B (1.0-2.0 mm)			GROUP C (2.0-3.0 mm)			GROUP D (3.0-5.0 mm)		
TEST TEMP	MAX LOAD W/O AGING	MAX LOAD W. AGING	TEST TEMP	MAX LOAD W/O AGING	MAX LOAD W. AGING	TEST TEMP	MAX LOAD W/O AGING	MAX LOAD W. AGING
(°F)	(lbs)	(lbs)	(°F)	(lbs)	(lbs)	(°F)	(lbs)	(lbs)
73	263 ± 20	247	73	203 ± 21	181	73	163 ± 14	158
105	162 ± 16	186	105	134 ± 15	152 ± 5	105	104 ± 12	104
120	156 ± 17	132	120	118 ± 9	130 ± 6			
150	115 ± 16	96	150	67 ± 11	81 ± 4			
180	76 ± 4	62	180	62 ± 13	67 ± 15			

FIGURE 1 NOTCH TYPE 1

ILLUSTRATION OF NOTCH IN SAMPLES

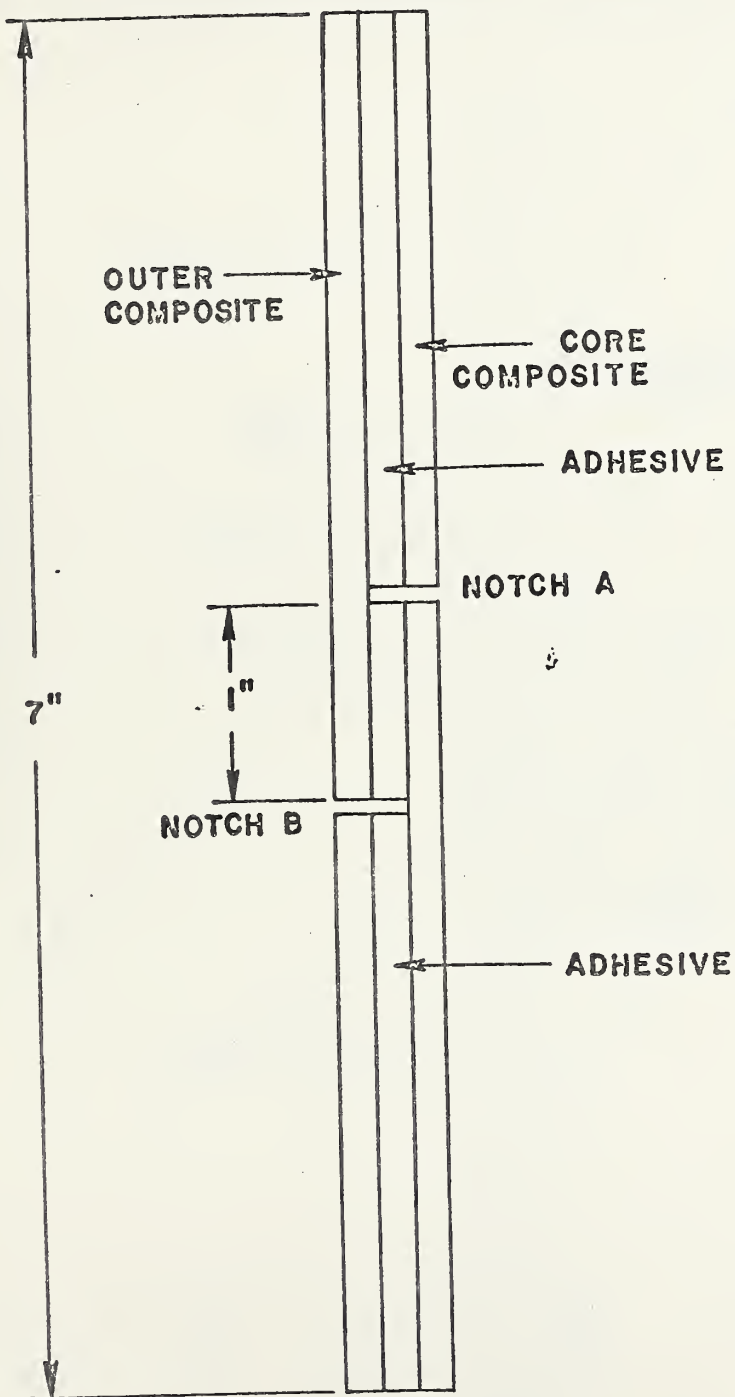
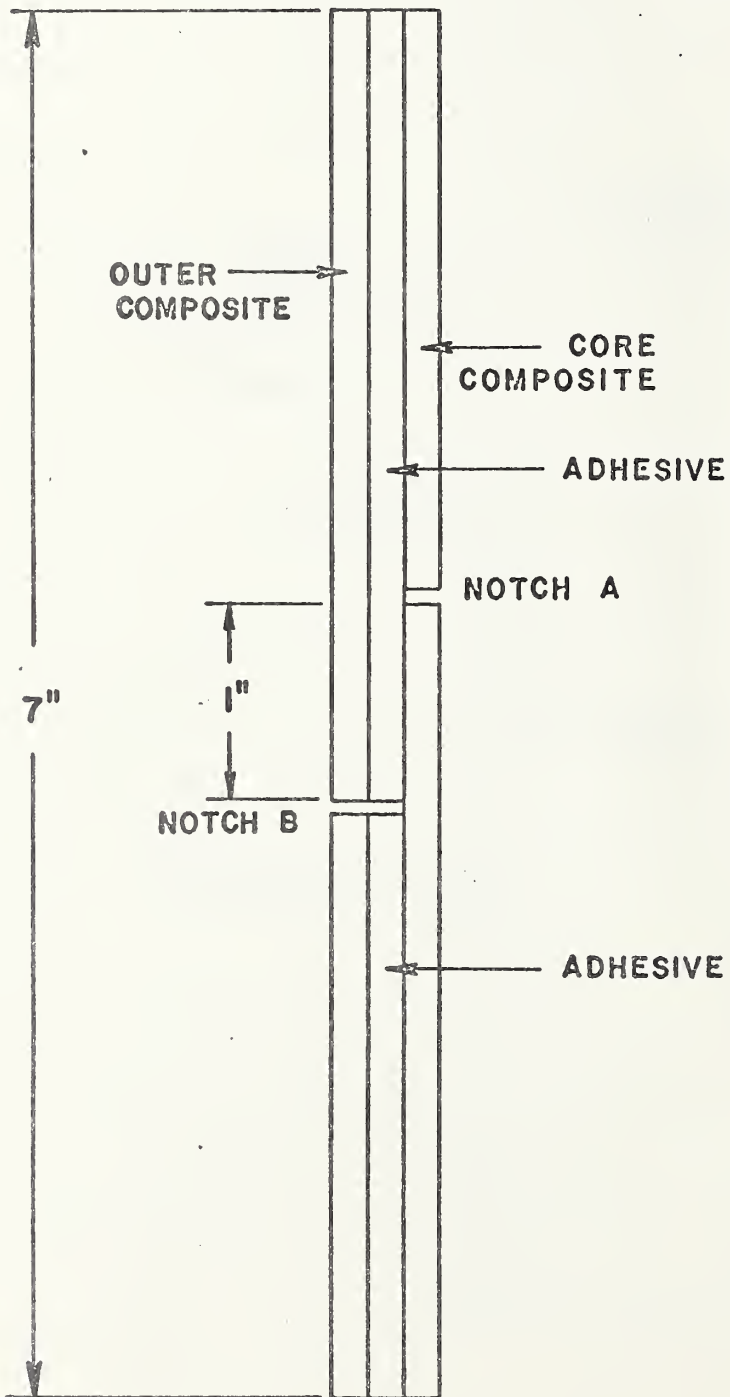


FIGURE 2 NOTCH TYPE 2

ILLUSTRATION OF NOTCH IN SAMPLES



ADHESIVE STRENGTH AT VARIOUS TEMPERATURES

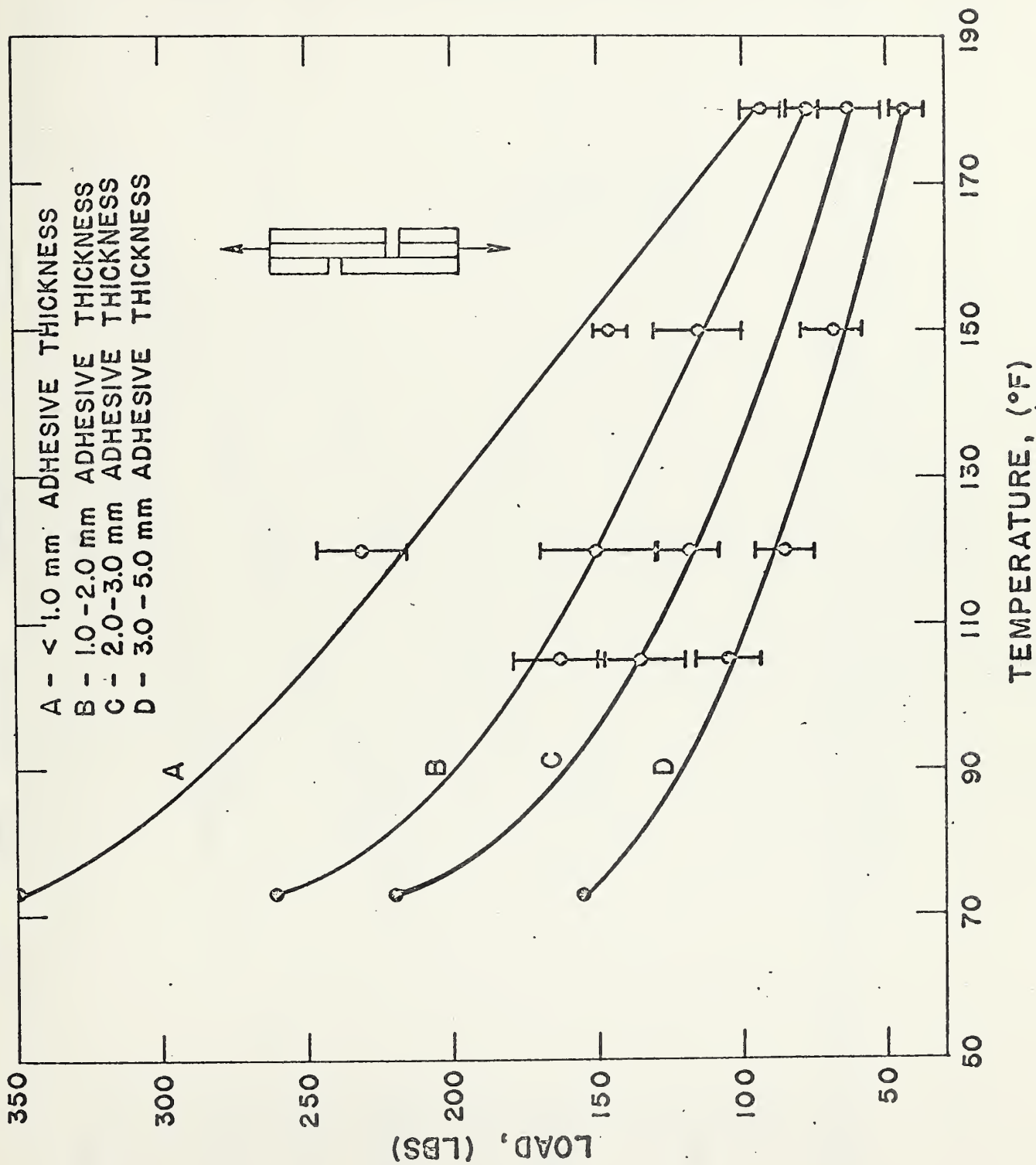


FIGURE 4

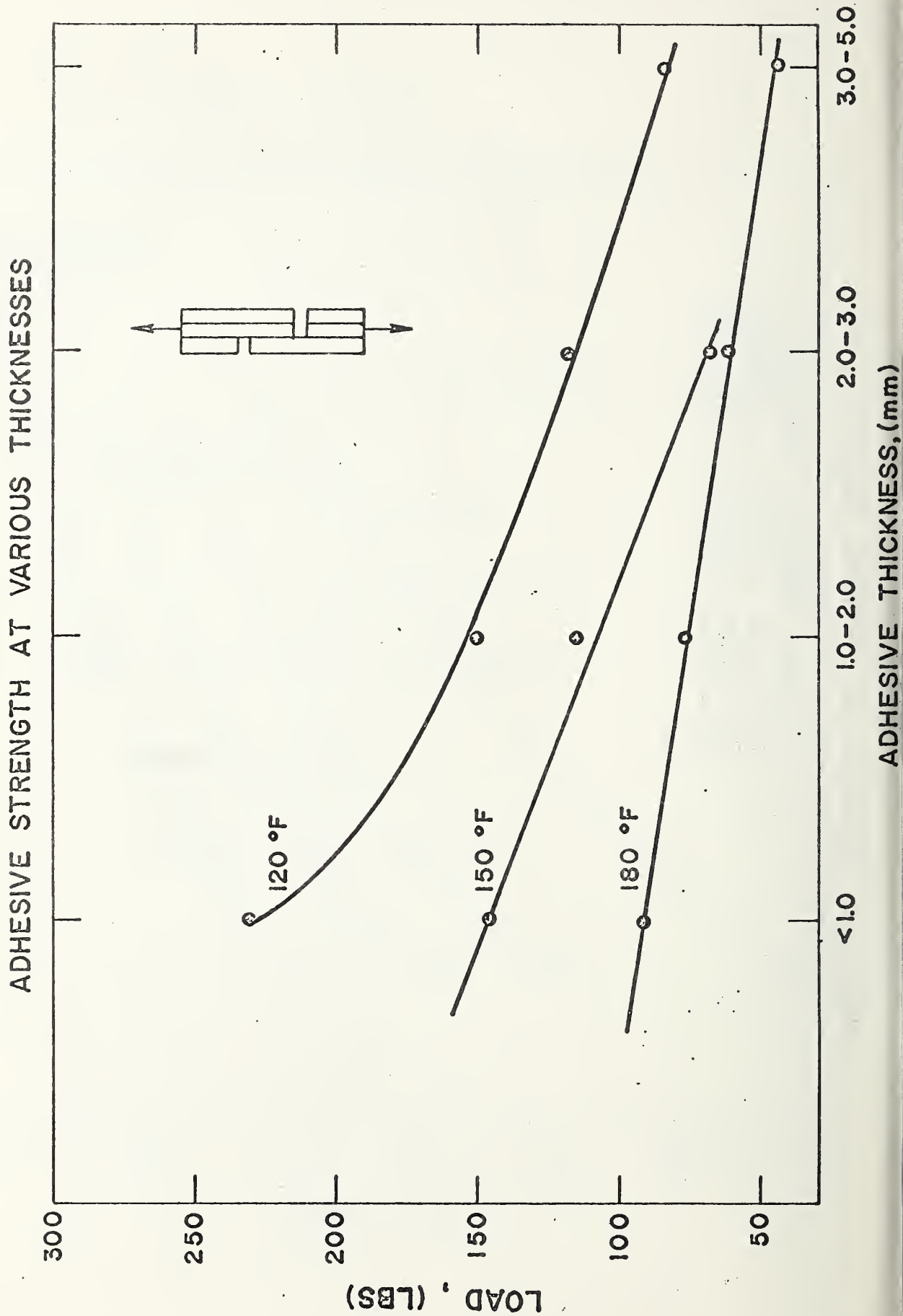


FIGURE 5

