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ABSTRACT

Five classes of adhesives were selected for evaluation at cryogenic temperatures on the basis of reported promising high lap-shear strengths at -65°F and 75°F. Lap-shear specimens were tested at -423°F, -320°F, -100°F, and 75°F utilizing epoxy-nylon adhesives (Metlbond 406, AF-40 and FM-1000), nitrile-phenolic adhesives (Metlbond 4041 and AF-32), epoxy-polyamide adhesives (Resiweld No. 4 and Narmco 3135), an epoxy-phenolic adhesive, (Metlbond 302-A), and a polyurethane adhesive (APCO 1219). Selection of adherends for testing was based on the anticipated use of these materials in future missiles and spacecraft, the prevalent use of some of these materials in the Atlas and Centaur, and the promising cryogenic properties of the base materials. The adherends utilized were: 0.020" EFH 301 CRES, 0.064" 2024-T3 bare aluminum, 0.020" A-110-AT titanium, 0.125" Conolon 506 (phenolic fiberglass laminate) and 0.125" Conolon 527 (polyester-fiberglass laminate). Butt-tensile tests were conducted with 3/4" round stock 321 stainless steel and AF-40 epoxy-nylon adhesive.

The epoxy-nylon adhesives resulted in the highest lap-shear strengths with all adherends over the entire temperature range of -423°F to 78°F. Values obtained at -423°F are more than 100% higher than any previous reported values for similar tests. The nitrile-phenolic adhesives gave excellent results over the temperature range of -320°F to 78°F but dropped off sharply at -423°F. The epoxy-phenolic adhesives gave uniform results over the complete temperature range. These results were significantly lower than the epoxy-nylon and nitrile-phenolic adhesives at -320°F, -100°F and 78°F. At -423°F the epoxy-phenolic is superior to the nitrile-phenolics. Room temperature cured adhesives are generally inferior to those that are heat cured. Of the three room temperature curing adhesives tested, the polyurethane gave higher lap-shear strengths than the epoxy-polyamides with aluminum adherends and approximately the same strengths with stainless steel adherends. All the adhesives tested had their highest lap-shear strengths at -100°F.

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CRYOGENIC ADHESIVE EVALUATION STUDY

J. Hertz

25 January 1961

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ENGINEERING DEPARTMENT

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CONVAIR (ASTRONAUTICS) DIVISION  
GENERAL DYNAMICS CORPORATION

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by

Paul W. Bergstedt  
Senior Engineering Metallurgist  
Materials Research Group, 592-1

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- ERR-AN-002...THE EFFECT OF CRYOGENIC TEMPERATURES ON THE MECHANICAL PROPERTIES OF HIGH STRENGTH SHEET ALLOYS (NONFERROUS ALLOYS)
- ERR-AN-003...THE EFFECT OF CRYOGENIC TEMPERATURES ON THE MECHANICAL PROPERTIES OF HIGH STRENGTH SHEET ALLOYS (COLD WORKED AUSTENITIC STAINLESS STEELS)
- \* ERR-AN-032...CRYOGENIC ADHESIVE EVALUATION STUDY
- ERR-AN-055...MEASUREMENT OF THE ELASTIC PROPERTIES OF 300 SERIES STAINLESS STEELS AT CRYOGENIC TEMPERATURES BY ULTRASONIC TECHNIQUES
- ERR-AN-057...A STUDY OF AUSTENITE DECOMPOSITION AT CRYOGENIC TEMPERATURES
- ERR-AN-067...A STUDY OF DEFORMATIONAL MECHANISMS IN DUCTILE CERAMICS
- ERR-AN-085...A STUDY OF THE EFFECTS OF NUCLEAR RADIATION ON HIGH-STRENGTH AEROSPACE VEHICLE MATERIALS AT THE BOILING POINT OF HYDROGEN (-423°F)

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ERH-AN-032  
Materials Research

**CRYOGENIC ADHESIVE EVALUATION STUDY**

J. Hertz

25 January 1961

ENGINEERING DEPARTMENT

This work was supported under Convair-Sponsored Research

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CONVAIR (ASTRONAUTICS) DIVISION  
GENERAL DYNAMICS CORPORATION

<u>CONTENTS</u>		<u>PAGE</u>
	LIST OF ILLUSTRATIONS	111
	LIST OF TABLES	v
	ABSTRACT	1
	FOREWARD	2
I	INTRODUCTION	3
II	EXPERIMENTAL PROCEDURE	4
III	DISCUSSION OF RESULTS	6
IV	SUMMARY	12
V	CONCLUSIONS	13
VI	FUTURE WORK SUGGESTED BY THIS PROGRAM	14
VII	ACKNOWLEDGMENT	15
VIII	BIBLIOGRAPHY	73
IX	DISTRIBUTION	74

## ILLUSTRATIONS

<b>No.</b>	<b>Title</b>	<b>Page</b>
1.	Lap-Shear, Butt-Tensile and Impact Specimens Utilized in REA 111-9106.	44
2.	Butt-Tensile Specimen Alignment Jig	45
3.	Advance Design Butt-Tensile Specimen Alignment Jig.	46
4.	Lap-Shear Strength of Metlbond 406 Adhesive Bonds vs. Temperature	47
5.	Lap-Shear Strength of AF-40 Adhesive Bonds vs. Temperature	48
6.	Lap-Shear Strength of FM-1000 Adhesive Bonds vs. Temperature	49
7.	Lap-Shear Strength of Metlbond 4041 Adhesive Bonds vs Temperature	50
8.	Lap-Shear Strength of AF-32 Adhesive Bonds vs. Temperature	51
9.	Lap-Shear Strength of Metlbond 302-A Adhesive Bonds vs. Temperature	52
10.	Lap-Shear Strength of Narmco 3135 Adhesive Bonds vs. Temperature	53
11.	Lap-Shear Strength of Resiweld No. 4 Adhesive Bonds vs. Temperature	54
12.	Lap-Shear Strength of APCO 1219 Adhesive Bonds vs. Temperature	55
13.	Lap-Shear Strength of Epoxy-Phenolic Adhesive Bonds vs. Temperature	56
14.	Lap-Shear Strength of Miscellaneous Adhesives Reported by North American Aviation Company.	57
15.	Lap-Shear Strength of Bloomingdale FM-47 Adhesive Bonds vs. Temperature	58
16.	Lap-Shear Strength of Bondmaster M24B Adhesive Bonds vs. Temperature	59
17.	Lap-Shear Strength of Swedlow 371-W Adhesive Bonds vs. Temperature	60
18.	Lap-Shear Strength of "3M" EC-1469 Adhesive Bonds vs. Temperature	61
19.	Lap-Shear Strength of Metlbond 4021 Adhesive Bonds vs. Temperature	62
20.	Lap-Shear Strength of Miscellaneous Adhesive Bonds vs. Temperature	63

ILLUSTRATIONS (CONT'D)

<u>No.</u>	<u>Title</u>	<u>Page</u>
21.	Strength of a Filled Epoxy Adhesive Bond vs. Temperature	64
22.	Strength of a Filled Epoxy Adhesive Bond vs. Temperature Prior to Testing at Room Temperature	65
23.	Results of Adhesive Optimization Tests Conducted at Denver Research Institute	66
24.	Results of Cure Cycle Optimization Tests Conducted at Denver Research Institute	67
25.	Results of Metal Surface Preparation Tests Conducted at Denver Research Institute	68
26.	Results of Teflon Etch Optimization Tests Conducted at Denver Research Institute	69
27.	Specimens of 0.020" A-110-AT Titanium Bonded with AF-40 Primed Adhesive System Before Bonding and After Being Pulled at 78°F, -100°F and -320°F.	70
28.	Cohesion and Adhesion Failure of Adhesive Bonds After Being Pulled at 78°F and -320°F.	71
29.	Effect of Surface Preparation and Overlap Length on Lap-Shear Strength of Metlbond 406.	72

## TABLES

<u>No.</u>	<u>Title</u>	<u>Page</u>
1.	Surface Preparations	16
2.	Adhesives Evaluated at Convair-Astronautics	17
3.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With Metlbond 406 Adhesive	18
4.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With AF-40 Adhesive	22
5.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With FM-1000 Adhesive	25
6.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With Metlbond 4041 Adhesive	27
7.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With AF-32 Adhesive	31
8.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With Metlbond 302-A Adhesive	33
9.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With Narmco 3135 Adhesive	35
10.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With Resiweld No. 4 Adhesive	38
11.	Results on Lap-Shear Tests on $\frac{1}{2}$ -Inch Overlap Specimens Bonded With APCO 1219 Adhesive	39
12.	Results on butt-Tensile Specimens Bonded With AF-40 Adhesive	41
13.	Adhesives Evaluated by NBS and FPL	42
14.	Adhesives Evaluated by Denver Research Institute	43

## ABSTRACT

Five classes of adhesives were selected for evaluation at cryogenic temperatures on the basis of reported promising high lap-shear strengths at  $-65^{\circ}\text{F}$  and  $75^{\circ}\text{F}$ . Lap-shear specimens were tested at  $-423^{\circ}\text{F}$ ,  $-320^{\circ}\text{F}$ ,  $-100^{\circ}\text{F}$ , and  $75^{\circ}\text{F}$  utilizing epoxy-nylon adhesives (Metlbond 406, AF-40 and FM-1000), nitrile-phenolic adhesives (Metlbond 4041 and AF-32), epoxy-polyamide adhesives (Resiveld No. 4 and Narmco 3135), an epoxy-phenolic adhesive, (Metlbond 302-A), and a polyurethane adhesive (APCO 1219). Selection of adherends for testing was based on the anticipated use of these materials in future missiles and spacecraft, the prevalent use of some of these materials in the Atlas and Centaur, and the promising cryogenic properties of the base materials. The adherends utilized were: 0.020" EFH 301 CRES, 0.064" 2024-T3 bare aluminum, 0.020" A-110-AT titanium, 0.125" Conolon 506 (phenolic-fiberglass laminate) and 0.125" Conolon 527 (polyester-fiberglass laminate). Butt-tensile tests were conducted with  $3/4$ " round stock 321 stainless steel and AF-40 epoxy-nylon adhesive.

The epoxy-nylon adhesives resulted in the highest lap-shear strengths with all adherends over the entire temperature range of  $-423^{\circ}\text{F}$  to  $78^{\circ}\text{F}$ . Values obtained at  $-423^{\circ}\text{F}$  are more than 100% higher than any previous reported values for similar tests. The nitrile-phenolic adhesives gave excellent results over the temperature range of  $-320^{\circ}\text{F}$  to  $78^{\circ}\text{F}$  but dropped off sharply at  $-423^{\circ}\text{F}$ . The epoxy-phenolic adhesives gave uniform results over the complete temperature range. These results were significantly lower than the epoxy-nylon and nitrile-phenolic adhesives at  $-320^{\circ}\text{F}$ ,  $-100^{\circ}\text{F}$  and  $78^{\circ}\text{F}$ . At  $-423^{\circ}\text{F}$  the epoxy-phenolic is superior to the nitrile-phenolics. Room temperature cured adhesives are generally inferior to those that are heat cured. Of the three room temperature curing adhesives tested, the polyurethane gave higher lap-shear strengths than the epoxy-polyamides with aluminum adherends and approximately the same strengths with stainless steel adherends. All the adhesives tested had their highest lap-shear strengths at  $-100^{\circ}\text{F}$ .

## FOREWARD

This report describes the experimental work performed under the REA 111-9106 entitled "Cryogenic Adhesive Evaluation Study". This included lap-shear and butt-tensile testing of adhesive bonds at the following test temperatures: 78°F, -100°F, -320°F, and -423°F. Nine adhesives were chosen on the basis of their reported strengths at -65°F and 75°F. Emphasis was placed on obtaining information on selected families of adhesives rather than on arriving at some optimum strength value. These adhesives were evaluated with the following adherends 301 and 321 stainless steels, 2024-T3 bare aluminum, A-110-AT titanium, phenolic-fiberglass laminate and polyester-fiberglass laminate.

An effort was made to avoid work previously accomplished by other agencies. Work performed by other agencies has been included to give an overall picture of the cryogenic properties of general classes of adhesives. The adhesives evaluated at Convair-Astronautics have much higher strength properties at cryogenic temperatures than any previously evaluated.

## ADHESIVES FOR CRYOGENIC APPLICATIONS

### INTRODUCTION:

Cryogenic fluids (liquefied gases) are finding increasing applications in the nuclear field, the electronics field and in the area of missile propellants. The use of cryogenic fluids in missiles for both fuels and oxidizers has resulted in numerous materials problems. Metals and non-metals which perform adequately at room temperature and at elevated temperatures often prove to be unsuitable at sub-zero temperatures due to severe embrittlement.

The extensive use of adhesive bonding in the Centaur, and the anticipated use of adhesives in future space vehicles such as the Saturn, RLV and Apollo has focused attention on the problems associated with bonded joints at cryogenic temperatures. Many of these problems are the result of stress concentrations and gradients developed within the adhesive bond. There are many causes for stress concentrations in adhesive joints, and many of these are aggravated by extreme sub-zero operational temperatures (as low as  $-423^{\circ}\text{F}$ ). Some of the principal causes for stress concentrations in adhesive joints are:

1. difference in thermal coefficient of linear expansion between adhesive and adherends,
2. shrinkage of adhesive in curing,
3. trapped gases or volatiles evolved during bonding,
4. differences in modulus of elasticity and shear strengths of adhesive and adherends,
5. differences in thermal conductivity of adhesive and adherends,
6. residual stresses in adherends as a result of the release of bonding pressure.

Operating at cryogenic temperatures intensifies the stress concentrations which arise as a result of differences in the physical and mechanical properties of adhesives and adherends. A low modulus adhesive at room temperature may readily relieve stress concentrations by deformation, however, at sub-zero temperatures the modulus of elasticity may increase to a point where the adhesive is no longer effective in relieving these concentrated stresses.

Advanced missiles and space vehicles will have many applications for metal-to-metal, metal-to-plastic, and plastic-to-plastic bonding materials which can withstand extreme low temperature conditions. Some of the foreseeable problem areas include: contractable plastic film fuel bags; bonding of foam and honeycomb insulation to missile skins; bonding of brackets, clips, spacers, etc. directly to or within cryogenic tanks; adhesive bonding of spiral wrapped (metal) cryogenic tanks; joining of tank sections with scarf or lap joints with or without doublers to provide joints of 100% tensile efficiency; bonding of composite plastic parts for antenna housings and instrument pods; bonding of dissimilar metals in an effort to prevent corrosion; etc.

The Materials Research Group at Convair-Astronautics has conducted an REA program entitled, "Cryogenic Adhesive Evaluation Study" during 1960 in order to obtain more useful information on adhesives at cryogenic temperatures. The primary objective of this study was to obtain mechanical property data on adhesive bonds

at cryogenic temperatures. It was intended to investigate basic classes of structural adhesives (epoxies, phenolics, polyurethanes, etc.) at these temperatures.

Prior to the work done at Convair-Astronautics on the evaluation of adhesives, there had been a minimum amount of work done by other agencies (most of it accomplished by the National Bureau of Standards). The data available is restricted mainly to lap-shear results on epoxy, phenolic and vinyl based adhesives utilizing stainless steel, aluminum and copper as the adherends. Some work has also been accomplished on butt-tensile and impact strengths of adhesives at cryogenic temperatures utilizing copper adherends. Since the adhesives evaluated were generally ones that had been designed for high-temperature applications, it was felt that an evaluation of adhesives carefully selected for their low-temperature properties rather than their high-temperature properties might result in better adhesives for cryogenic applications.

### EXPERIMENTAL PROCEDURE

This investigation was to include lap-shear, butt-tensile, impact and T-peel testing of metal-to-metal, plastic-to-metal and plastic-to-plastic adhesive bonds. Since the data obtained was to be used as a qualitative evaluation of adhesive systems rather than for design allowables, it was felt that the standard  $\frac{1}{2}$ "-overlap, lap-shear specimens (ASTM D1002-53T) would be the simplest and easiest to fabricate, and the results could readily be correlated with results obtained at other agencies. Most of the test data obtained was a result of lap-shear testing, although some butt-tensile testing was also accomplished. Impact specimens were bonded and awaiting test when the program was closed out for lack of funds (23 November 1960). Figure 1 is a photograph of the lap-shear, butt-tensile and impact specimens utilized in this REA program.

Lap-shear coupons were machined and fabricated from the following materials: 0.020" EFH 301 stainless steel, 0.064" 2024 T-3 bare aluminum, 0.020" A-110-AT titanium, 0.125" Conolon 506 (phenolic-fiberglass laminate) and 0.125" Conolon 527 (polyester-fiberglass laminate). The selection of these adherends was based on the expected use of these materials in future missiles and spacecraft, the prevalent use of some of these materials in the Atlas and Centaur and the excellent cryogenic properties of the materials. The lap-shear coupons were prepared by bonding  $\frac{1}{2}$ " x 1" strips to obtain an overall test specimen of  $7\frac{1}{2}$ " x 1". The ends of the specimens contain spotwelded doublers for reinforcement in the grip areas and  $\frac{1}{2}$ " holes for accommodating pin type grips.

Butt-tensile coupons were prepared from  $\frac{3}{4}$ " round stock 321 stainless steel. After bonding, the coupon had an overall length of 2" and a bond area of 0.442 sq. in. The coupons were threaded on each end with a  $\frac{3}{4}$ -10 thread.

Impact specimens were prepared from 321 stainless steel flat stock per Federal Test Method Standard No. 175 (Tentative Standard Method 1051.1-T). They consisted of two stainless steel blocks (a 1" x 1" x  $\frac{3}{8}$ " and a 1  $\frac{3}{4}$ " x 1" x  $\frac{3}{4}$ ") bonded together and resulting in 1 sq. in. of bonded area.

Surface preparation of all test coupons was standardized for each adherend (see Table 1). Priming and bonding were accomplished as per manufacturer's recommendations. In all cases the lap-shear specimens were loaded at a rate of 600-700 psi

of the shear area per minute (ASTM D 1002-53T). The butt-tensile coupons were also tested at the same rate. The testing was accomplished in the normal procedure utilizing a 50,000 lb. Baldwin-Emery SR-4 testing machine, Model FGT. The proper temperature was provided by means of a cryostat filled with either alcohol and dry ice (-100°F), liquid nitrogen (-320°F) or liquid hydrogen (-423°F) in which the sample was immersed during the test. Details of the experimental procedure including temperature measurement, liquid level indication and safety precautions are given in a paper by Christian and Watson which has been submitted for publication to the American Society for Testing Materials (Ref. 2).

Many of the specimens tested were prepared at no cost to Convair-Astronautics by the adhesive manufacturers per our recommendations and utilizing materials supplied by Convair. This resulted in the accomplishment of a much larger test program than could otherwise have been conducted with the funding provided for REA 111-9106.

The lap-shear coupons were bonded utilizing normal alignment fixtures. A special alignment jig was designed and fabricated for bonding of the butt-tensile specimens (see Figure 2). This consisted of two solid blocks made of 321 stainless steel (each having a drilled and threaded hole) and a set of alignment pins. The blocks were gang drilled so that they would be perfectly aligned. In bonding, the coupons were screwed into the alignment fixture, the adhesive was applied and the pressure was applied to the top plate by the heated platens of a hydraulic press. Bonding of butt-tensile coupons in this manner proved to be very slow and resulted in a wide variation of glue line thicknesses. A second alignment jig (see Figure 3) has been designed and fabricated which can handle ten butt-tensile coupons in one operation. This fixture incorporates the use of flanged sleeves having internal threads. The specimens are screwed into the sleeves and are held in place by a gusset plate which is bolted to the bottom plate with Allen-head screws. Coupons are also screwed into the top plate, adhesive is applied to the specimens, and the top and bottom plates are then brought into contact with each other utilizing the alignment pins to insure proper alignment. Pressure and heat is applied by using the heated platens of a hydraulic press. By surface grinding the test coupons after they have been installed in the alignment fixture and prior to the bonding operation it is possible to obtain ten test coupons having practically identical adhesive thicknesses.

Prior to test the lap-shear coupons were measured optically to determine the length and width of the overlap area. Thickness measurements of the adherends and the bonded area for each specimen were made with a micrometer to determine the adhesive thickness of each test coupon. After testing all specimens that failed in the bonded area rather than in the adherends were visually examined. The type and extent of each type of failure was noted. For this report a cohesion failure is defined as one that occurs within the adhesive, while an adhesion failure is defined as one that occurs in a weak boundary layer. In the cases where a primer was utilized and failure occurred between adhesive and primer, the failure is considered to be one of adhesion. In cases where the adhesive is a composite of fiberglass coated with resin, and failure occurs between the resin and the fiberglass the failure is considered to be one of cohesion.

Nine adhesives were chosen for testing in this program. This included six adhesives which required pressure and heat for curing, and three adhesives which cured at room temperature and contact pressure. The basis for choosing the particular adhesives was the fact that they had higher reported strengths at -65°F than at room temperature. The commercial designation of the adhesives, the adhesive types, the suppliers and the methods of application are listed in Table 2.

## DISCUSSION OF RESULTS

All the results obtained on  $\frac{1}{2}$ "- overlap, lap-shear specimens are reported in Tables 3-11 and are plotted in Figures 4-12. Results obtained on butt-tensile coupons are reported in Table 12.

Results of this program indicate that, of the adhesives investigated, the epoxy-nylon class of adhesives (Metlbond 406, FM-1000 and AF-40) are most suitable for applications over the temperature range of  $-423^{\circ}\text{F}$  to  $78^{\circ}\text{F}$ . The nitrile-phenolic adhesives tested (Metlbond 4041 and AF-32) gave excellent results over the temperature range of  $-320^{\circ}\text{F}$  to  $78^{\circ}\text{F}$ , but fell off sharply when tested at  $-423^{\circ}\text{F}$ .

The only other heat cured adhesive investigated was an epoxy-phenolic resin impregnated on fiberglass cloth (Metlbond 302-A) and this resulted in almost constant strengths over the complete test range of  $-423^{\circ}\text{F}$  to  $78^{\circ}\text{F}$ . Figure 13 is a plot of some of the results obtained by the National Bureau of Standards (Ref. 3) when they evaluated two epoxy-phenolic adhesives of the same general class (Epon 422 and Metlbond 302) utilizing stainless steel adherends. In general the values obtained by the National Bureau of Standards in conjunction with the Forest Products Laboratory result in a curve parallel to the values obtained at CVA (see Figures 9 and 13). The values obtained by NBS and FPL are approximately 200-300 psi higher than those obtained at CVA. This may be attributed to two causes: (1) The etch cycles used were different and (2) the specimens used by NBS and FPL were made with 0.063" thick adherends while those used at CVA were made with 0.020" thick adherends. Epstein (ref. 4) has stated that the strength of a bonded joint would be expected to increase with increase in adherend thickness. He stated that in a simple overlap specimen where both adherends are the same the stress concentration (n) is equal to  $\frac{1}{k} \coth \frac{1}{k}$  where  $\frac{1}{k} = \frac{Gd}{2Ead}$

In this formula: L = overlap length, in.

G = adhesive shear modulus, psi

E = adherend modulus of elasticity, psi

s = adherend thickness, in.

d = adhesive thickness, in.

From the above equation it is apparent that the stress concentration in the joint will increase with increase in the adhesive shear modulus or with increase in overlap length, while the stress concentration will decrease with increase in modulus of elasticity of the adherends, thickness of the adherends, or thickness of the adhesive film. Since the adhesive thickness, the overlap area, the shear modulus of the adhesive and the modulus of elasticity of the adherends were essentially the same for both the NBS and the current tests, it appears that the difference in adherend thickness is the primary cause for differences in the end results. No tests were conducted with Metlbond 302-A and aluminum adherends since much of this work had already been accomplished by another agency (see Figure 14). The values reported by North American Aviation Co. (see Ref. 5), for 2024 T-3 aluminum bonded with Metlbond 302 if plotted in Figure 9 would result in a parallel curve between the stainless steel and titanium curves.

Since there are many applications in the manufacturing of missiles where room temperature curing, contact pressure adhesives are preferable to the heat cured, pressure type adhesive, the current investigation included three of the room temperature curing adhesives. Two of these belong to the epoxy-polyamide class (Narmco 3135 and Resiweld No. 4). In general these adhesives indicate little or no change in strength properties over the temperature range of  $-423^{\circ}\text{F}$  to  $78^{\circ}\text{F}$  (see Figures 10 and 11). The values obtained with Resiweld No. 4 are slightly higher than those obtained with Narmco 3135 since a more rigid formulation was used with the Resiweld No. 4. The values reported by North American Aviation Co. (see Figure 14) for an epoxy-polyamide adhesive drop off more rapidly at low temperatures than do the ones obtained in this study.

The third room temperature curing, contact pressure adhesive investigated in this program was a polyurethane (APCO 1219). The results obtained with this adhesive indicate an extreme increase in strength when going from  $78^{\circ}\text{F}$  to  $-100^{\circ}\text{F}$ , and then a gradual decrease in strength at  $-320^{\circ}\text{F}$  and  $-423^{\circ}\text{F}$ . In general the polyurethane appeared better at the lower temperatures than the epoxy-polyamides with aluminum adherends and gave approximately the same results with stainless steel adherends. However, a wider variation in results was noticed with the polyurethanes. This may have been caused by the normal parameters which adversely affect polyurethanes, i.e.: humidity and temperature during cure.

Other classes of heat cured adhesives have been investigated by the National Bureau of Standards and other agencies, but in all cases the results obtained were much poorer than the low temperature strength values of the adhesives tested at CVA. The adhesives tested by Frost (Ref. 3) are listed in Table 13, and the results are plotted in Figures 13 and 15-20. The nitrile-phenolic adhesives tested (Metlbond 4021 and AF 5930) had high lap-shear strengths at room temperature but dropped off very sharply at low temperatures (see Figures 19 and 20). Their poor low temperature properties as compared to the strengths obtained on Metlbond 4041 and AF-32 tested at CVA may be attributed to the formulation of the adhesives. Superior low temperature properties in nitrile-phenolics may be obtained by utilizing lower molecular weight resins and by increasing the percentage of rubber in the adhesive formulation. This, however, is usually accompanied by lower strengths at higher temperatures ( $78^{\circ}\text{F}$  to  $500^{\circ}\text{F}$ ).

Vinyl-phenolics have been investigated at NBS (FM-47, 371-W, EC-1471 and Redux 775) and the results are plotted in Figures 15, 17 and 20. The general trend in all the adhesives of this class which were evaluated are high lap-shear strengths at room temperature, sharp drops in strength at  $-100^{\circ}\text{F}$  and then a gradual leveling off in strength at  $-320^{\circ}\text{F}$  and  $-423^{\circ}\text{F}$ . A rubber-epoxide-phenolic adhesive (Bondmaster M24B) which was investigated (see Figure 17) gave similar results, but at all temperatures the shear strengths were lower than those obtained with the vinyl-phenolics.

A filled epoxy (EC-1469) was investigated at NBS and resulted in almost uniform strength values over the complete temperature range of  $-423^{\circ}\text{F}$  to  $78^{\circ}\text{F}$  (see Figure 18). The strength values fall between those obtained at CVA for epoxy-phenolics and those obtained for room temperature curing epoxy-polyamides. Similar results were reported by North American Aviation Co. for an epoxy adhesive (see Figure 14). Previous work at NBS reported by McClintock and Hiza (Ref. 6) on a filled epoxy adhesive system with copper adherends (see Figure 21) shows a sharp decrease in lap-shear strength at  $75^{\circ}\text{K}$  ( $-320^{\circ}\text{F}$ ) and  $20^{\circ}\text{K}$  ( $-423^{\circ}\text{F}$ ) from the strength obtained at  $300^{\circ}\text{K}$  ( $80^{\circ}\text{F}$ ). However, three new factors come into play:

- (1) The lap-shear specimens used were such that no tearing forces would be encountered
- (2) stress concentrations differ as a result of the use of copper adherends
- (3) the adherend thickness was extremely thick (approximately 3/8" to 1/2".)

Data reported by North American Aviation Co. on a neoprene-phenolic adhesive supported on Nylon tape, Metlbond MN3C (see Figure 14), shows uniform results over the temperature range of -320°F to 75°F. The results are very similar to those obtained with Metlbond 302. Data reported by Narmco Resin & Coatings on Metlbond 408, a vinyl modified version of Metlbond 406, indicates a sharp reduction in low temperature strength for the Metlbond 408 when compared to the low temperature values of Metlbond 406 (2800 psi at -320°F compared to 6020 psi obtained with Metlbond 406 at -320°F)

It has been demonstrated by McClintock and Hiza at NBS that thermal cycling of adhesive bonds from room temperature to -320°F results in a reduction in final strength when the specimens are subsequently pulled at room temperature and compared to specimens which have not been subject to the thermal cycle. This work was accomplished on a filled epoxy (asbestos and alumina filler) cured with diethylaminopropylamine (see Figure 22). The explanation set forth to account for this behavior is that the difference in thermal conductivity between adhesive and adherends produces additional transient stresses (above those present from other sources) during thermal cycling. It was postulated that the adhesive will lag behind the metal in most changes of temperature because of its lower thermal conductivity, and this lag coupled with the contact resistance present at the interface may result in stresses sufficient to cause local failures of the bond. Since the adhesive normally has a greater expansivity than the adherends, it would be expected that the adhesive temperature lag would help to prevent such local failures. When equilibrium occurs these transient stresses would disappear. It was also postulated that the residual effect on tensile strength might have been caused in the same way, except that the strength reduction is due to local failures within the adhesive (since the adhesive is a heterogeneous mixture of two components of different expansivities).

Work accomplished at Massachusetts Institute of Technology indicated that there was no reduction in room temperature strength properties of Bakelite BJ -16320 (vinyl-phenolic) bonds when tested at room temperature after thermal cycling in liquid helium. Since the work by McClintock and Hiza was accomplished utilizing thick glue lines (approximately 10-30 mils), it could be expected that transient thermal stresses encountered would be greater than those expected with very thin glue lines, since the temperature lag in thick glue lines would be greater than the lag in thin glue lines. Work accomplished by Frost (Figures 13, 15-20) indicated no difference in results between specimens pulled after shock cooling and those pulled after gradual cooling. As a result of these previous accomplishments the work accomplished at CVA was all done after shock cooling, since this method is much faster, cheaper, and appears to result in values equivalent to those obtained by testing after gradual cooling.

It is imperative that all information on tests conducted by outside sources be evaluated before any valid comparison of test results can be accomplished. This is very apparent when viewing the results published by Eppinger and Love (Ref. 7).

The adhesives tested by Eppinger and Love are listed in Table 14 and the results they obtained are plotted in Figures 23-26. The values obtained are extremely high when they are considered only as lap-shear strengths, and the use of adhesives such as the epoxies, nitrile-phenolics and neoprene-phenolics might be chosen for other applications where they might prove to be inadequate. The results obtained are based on a double overlap configuration utilizing Teflon and 52100 steel as the adherends. The resulting stress concentrations under these conditions would be much less than those obtained with single overlap joints and metal adherends. The reduction in stress concentrations in the joint is a result of the removal of eccentricity in loading due to specimen configuration, and the utilization of a very low modulus adherend which more closely matches the modulus of the adhesive. No direct comparison can be made between the data obtained by Eppinger and Love and that obtained at CVA. Some semblance of relative adhesive bond strength can be attained when considering that AF-13 is reported having a strength value of 6700 psi (see Figure 23), and tests at  $-67^{\circ}\text{F}$  and  $75^{\circ}\text{F}$  on aluminum to aluminum simple overlap joints are reported by Minnesota Mining & Manufacturing Co. to give strengths of 2810 psi. Similarly, all the other adhesives when checked in simple overlap with metal-to-metal bonds result in much lower values at room temperature than those obtained at  $-320^{\circ}\text{F}$  by Eppinger and Love.

Adhesive bond thickness was determined for each specimen, but no emphasis was placed on trying to determine the effect of adhesive thickness on overlap strength. However, there appeared to be quite a variation in adhesive thickness in some of the adhesives tested. No general trend is evident when comparing these individual specimens, but this may be a result of the small number of specimens at each thickness level for each adherend-adhesive system. Reinhart (Ref. 8) has stated that for the same difference in thermal expansions of adhesive and adherend, thinner adhesive layers will experience less thermal stress. Frost has indicated that the difference in strengths of Metlbond 302 and Epon 422 (see Figure 13) can be attributed to this factor since the Metlbond 302 had an approximate thickness of 5 mils and the Epon 422 had an approximate thickness of 8 mils, and since both adhesives are essentially the same. However, when examining individual test specimens with other adhesive systems tested at NBS and FPL no general trend could be noted for strength versus adhesive thickness. Epstein mentions that the strength of adhesives which do not liberate gases during cure show little dependence upon glue line thickness. He states, however, that glue lines should not exceed 10 mils in order to obtain optimum strength. McClintock and Hiza have shown that there is no appreciable change in strength when glue lines have varied over the thickness range of 10-30 mils. It appears likely that strength variation with thickness may be dependent on the particular adhesive-adherend system, and further effort in this area may be fruitful.

The type of failure for each bond was noted, but here again no definite trend was established for strength versus percent cohesive failure. In theory it would be expected that complete cohesive failure would result in the highest strength values. Figure 27 is a photograph of a series of 0.020" A-110-AT titanium bonded with AF-40 primed adhesive system before bonding and after being pulled at  $78^{\circ}\text{F}$ ,  $-100^{\circ}\text{F}$ , and  $-320^{\circ}\text{F}$ . It can be seen that specimen C, pulled at  $-100^{\circ}\text{F}$ , broke completely in the base metal. Specimen B, pulled at  $78^{\circ}\text{F}$ , broke partially in adhesion and partially in cohesion, while specimen D, pulled at  $-320^{\circ}\text{F}$ , broke completely in adhesion. Figure 28 shows specimens B & D at a magnification of 1.0. All adhesives which failed in adhesion at  $-100^{\circ}\text{F}$  or  $-320^{\circ}\text{F}$  also failed in adhesion at  $-423^{\circ}\text{F}$ . The only adhesives which resulted in some cohesive failure at  $-423^{\circ}\text{F}$  belonged to the epoxy-nylon class (Metlbond 406 and FM-1000).

The effects of a primer on the final strength versus temperature curve for an epoxy-nylon system was investigated, and the results are plotted in figure 5. Specimens of 0.020" EFH 301 CRES were bonded with AF-40. Some were bonded after priming with EC-1956 and some were bonded without primer. The primed specimens showed much higher strengths at 78°F, approximately the same strength as the unprimed specimens at -100° and -320°F, and much lower strengths at -423°F. Since the epoxy-nylon resin system is presently not available in solution, the primer generally utilized is a phenolic-nitrile solution or an epoxy. In either case the resultant -423°F strength properties of the primer would be considerably lower than that of the epoxy-nylon resin system.

An effort was made to minimize the effects of etch on the final strength properties of the joints. In all cases the etch cycle was standardized for each adherend system. No effort was made to optimize the etch cycle, but instead it was decided to standardize on etchants which could be expected to result in high strengths. A phosphate etch which normally results in optimum strengths at high temperatures was also investigated since it is often used in preference to chromate etch systems, and since it results in a surface film. As expected the tests (see Figure 29) show that at 78°F the phosphate etch results in slightly higher strengths than the saturated chromate etch, while at cryogenic temperatures the phosphate etch proved very inferior. This may be explained by considering the phosphate film acting as a weak boundary layer or as a weak primer at low temperatures. The effects of etchants and surface preparation on final strength properties of overlap joints have many times been investigated at room temperature and at high temperatures, but little has been accomplished in this area for low temperature applications. Figures 24 and 25 indicate the magnitude of strength variations which might be expected as a result of choice of surface preparation. Although usual chromate etchants for stainless steel are much more dilute, initial evaluations with a saturated chromate solution resulted in high lap-shear strengths and it was then used for the entire evaluation study. It was not the intention of this program to arrive at an optimum system, but rather to come up with relative values for various adhesive classes. In this light no emphasis was placed on evaluating surface preparations. However, in any final design where design allowables are a requirement, it would be essential to properly evaluate surface preparation.

It is expected that the strength values obtained for FM-1000 bonds are not truly indicative of what might truly be expected. This is a result of the initial bonding of the specimens. The bonded specimens as received from the Bloomingdale Rubber Company showed the adherends to be at an angle to each other with a resulting sloped glue line. This can be expected to cause added stress concentrations and result in lower strength values. The large variations in strengths which were encountered with the FM-1000 bonds most likely are a result of the poor initial bonds.

In many cases specimens failed in the doublers at low temperatures. This was particularly true for the aluminum specimens, and may be a result of two factors: (1) poor spot welding of aluminum in the shop and (2) high stress concentrations around the aluminum spot welds at extreme sub-zero temperatures. Since in all cases, the adhesives chosen had their highest strengths at -100°F, most of the doubler failures resulted at this temperature.

Only the tensile strengths of specimens bonded with AF-40 after priming with EC-1956 were determined. There appears to be no logical explanation at this time for the wide variation in strength properties which was noted at each test temperature (see Table 12).

The strengths obtained on heat cured adhesive bonds when tested at -423°F gave the highest strengths with stainless steel adherends. Aluminum adherends gave the next highest strengths, and titanium adherends resulted in the weakest bonds. This trend was not apparent at all temperatures. At all temperatures, the strengths obtained with room temperature curing adhesives were highest for aluminum adherends, next highest with stainless steel adherends and lowest with titanium adherends.

As expected, the limiting lap-shear value for plastic laminate-to-plastic laminate adhesive joints is the interlaminar strength of the plastic. In cases where the adhesive utilized had poorer low temperature strength than the resin of the plastic laminate, some cohesion and adhesion type failures were encountered. The testing indicated that there was no need for testing of plastic laminate-to-metal adhesive bonds, since the proper choice of adhesive would insure failure in the laminate (weakest link) rather than in the adhesive.

## SUMMARY

This study has shown that the most promising group of adhesives for cryogenic applications (-423°F to 78°F) belong to the epoxy-nylon class. It should be kept in mind, however, that the results obtained are qualitative, and indicate the relative strengths of various chosen classes of adhesives. These values are not intended for use as design allowables.

The nitrile-phenolics tested gave high lap-shear strengths over the temperature range of -320°F to 78°F, but dropped off sharply at -423°F. They were superior to nitrile-phenolics tested previously at other facilities, and this may be accounted for by considering the formulation variations which can be encountered. The epoxy-phenolic tested had uniform strength properties over the complete range of temperatures. It was inferior to the epoxy-nylon and nitrile-phenolic adhesives at all temperatures except at -423°F where it was better than the nitrile-phenolics. The results obtained with the epoxy-phenolic correlated well with work accomplished at other agencies.

The room temperature cured adhesives were generally inferior to the heat cured adhesives. Of these, the polyurethane adhesive tested resulted in higher lap-shear strengths than the epoxy-polyamide adhesives over the complete temperature range with aluminum adherends, and approximately the same strengths with stainless steel adherends.

All the heat cured adhesives evaluated at -423°F resulted in highest strength values with stainless steel adherends, lower strengths with aluminum adherends and still lower strengths with titanium adherends. The room temperature cured adhesives at all test temperatures resulted in highest strengths with aluminum adherends, slightly lower strengths with stainless steel adherends and still lower strengths with titanium adherends. The limiting lap-shear strength with reinforced plastic adherends was the interlaminar strength of the plastic, and this type of testing has proved only useful in weeding out very poor adhesives.

The use of a primer with an epoxy-nylon adhesive resulted in higher strengths at 78°F, approximately the same strength at -100°F and -320°F, and lower strengths at -423°F. It was also found that a phosphate etch, with results in a surface film, drastically reduces low temperature lap-shear strength of adhesive joints.

This study has indicated no correlation between adhesive thickness and lap-shear strength. It has also failed to indicate a correlation between percent cohesive failure and lap-shear strength.

## - CONCLUSIONS

1. The epoxy-nylon adhesives tested (Metlbond 406, AF-40 and FM-1000) resulted in the highest lap-shear strengths at low temperatures (-100°F, -320°F and -423°F).
2. The nitrile-phenolic adhesives tested (Metlbond 4041 and AF-32) resulted in very high lap-shear strengths over the temperature range of -320°F to 78°F, but their strengths fell off sharply at -423°F.
3. The epoxy-phenolic adhesive tested had poorer lap-shear strength than the epoxy-nylon and phenolic-nitrile adhesives over the temperature range of -423°F to 78°F with the exception that it was superior to the phenolic-nitriles at -423°F.
4. The room temperature curing adhesives had much lower lap-shear strengths at 78°F than the heat cured adhesives, but in general they maintained a higher percentage of their strength at low temperatures.
5. The polyurethane adhesive tested resulted in higher lap-shear strengths than the epoxy-polyamide adhesives over the temperature range of -423°F to 78°F with aluminum adherends, and approximately the same strengths with stainless steel adherends.
6. The limiting lap-shear value for plastic laminate-to-plastic laminate adhesive bonds is the interlaminar strength of the plastic, and this type of testing could only be used to weed out very poor adhesives.
7. All the adhesives tested had their highest lap-shear strengths when tested at -100°F.
8. Results indicate no correlation between adhesive thickness and lap-shear strength.
9. Results indicate no correlation between percent cohesive failure and lap-shear strength.
10. The use of a primer with an epoxy-nylon adhesive results in higher lap-shear strengths over the temperature range of -320°F to 78°F, but results in lower lap-shear strengths at -423°F.
11. A phosphate etch which results in a surface film drastically reduces low temperature lap-shear strength of adhesive joints.
12. All the heat cured adhesives tested at -423°F gave the highest lap-shear strengths with stainless steel adherends, lower strength with aluminum adherends and still lower strengths with titanium adherends. No definite trend was established at the other test temperatures.
13. The room temperature cured adhesives at all test temperatures resulted in highest lap-shear strengths with aluminum adherends, slightly lower strengths with stainless steel adherends and lowest strengths with the titanium adherends.

### Future Work Suggested By This Program

1. Testing is required on the butt-tensile, impact and T-peel strengths of the classes of adhesives investigated. This is needed to get a complete picture of the strengths of adhesives at cryogenic temperatures.
2. The mechanical and physical properties of both the adhesives and the adherends should be obtained in order to determine why some adhesives are better than others at cryogenic temperatures. This would require stress-strain relationships, thermal expansion properties and thermal conductivity properties for the adhesives and the adherends over the complete temperature range.
3. The adhesives should be investigated to determine why some in one class have better low temperature properties than others in the same general class. This would require infra-red spectroscopy and solvent swelling type evaluations.
4. The effects of thermal cycling on the room temperature strengths of adhesive bonds should be evaluated for each class of adhesives. In cases where there is a marked effect, the relationship between number of cycles and final strength should be investigated.
5. The effect of adhesive thickness on low temperature strength should be fully investigated.
6. The optimization of surface preparations should be investigated with aluminum, titanium, and stainless steel adherends.
7. The addition of fillers and plasticizers to room temperature curing adhesives should be evaluated.
8. The effects of composite environments such as space effects and cryogenic temperatures should be investigated.

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## TABLE I

### Surface Preparations

#### Stainless Steel and Titanium Adherends

Wipe specimens with trichloroethylene. Immerse specimen for 10 minutes at 150°F in the following solution:

100 gms. hydrochloric acid (conc.)  
4 gms. hydrogen peroxide (30%)  
20 gms. formalin (40%)  
90 gms. water

Rinse specimens in cold tap water. Follow this with a distilled water rinse and air dry. This is followed by a 5-10 minute etch at 140-160°F in the following solution:

100 gms. sulfuric acid (conc.)  
10 gms. sodium dichromate  
30 gms. water

Repeat rinse and drying procedures as above.

#### Aluminum Adherends

Wipe specimens with trichloroethylene. Immerse specimens for 10 minutes at 150°F in the following solution:

30 gms. water  
10 gms. sulfuric acid (conc.)  
4 gms. sodium dichromate

Rinse specimens in cold tap water. Follow this with a distilled water rinse and air dry.

#### Plastic Laminate Adherends

Sand lightly and follow with a trichloroethylene wipe.

TABLE 2

Adhesives evaluated at Convair-Astronautics

COMMERCIAL DESIGNATION	ADHESIVE TYPE	SUPPLIER	CURING TEMPERATURE & PRESSURE
Metlbond 406	Epoxy-nylon (unsupported tape)	Narmco Resins & Coatings Co.	Heat to 350°F in 1 hr. Hold at 350°F and 25 psi for 1 hr. Cool in press to 275°F and remove.
AF-40	Epoxy-nylon (unsupported tape)	Minnesota Mining & Manufacturing Company	Dry primer, EC-1956, for ½ hr. at 78°F and ½ hr. at 250°F. Joint-heated at 10°/min. to 350°F. Cured at 350°F and 50 psi for 1 hr.
FM-1000	Epoxy-nylon (unsupported tape)	Bloomingsdale Rubber Company	Heat to 350°F in 30 min. Cure for 1 hr. at 350°F and 25 psi.
Metlbond 302-A	Modified epoxy- phenolic supported on glass cloth	Narmco Resins & Coatings Company	Cure at 350°F and 25 psi for 2 hr.
AF-32	Nitrile-modified phenolic (unsupported tape)	Minnesota Mining & Manufacturing Company	Dry primer, EC-1660 ½ hr. at 78°F plus ½ hr. at 350°F. Same cure cycle as AF-40 with 100 psi.
Metlbond 4041	Nitrile-modified phenolic (unsupported tape)	Narmco Resins & Coatings Company	Primed with 4041 Type II and dried at 78°F for 1 hr. Stage at 250°F for 15 min. and cure for 1 hr. at 350°F and 100 psi.
APCO 1219	Polyurethane	Applied Plastics Co.	Room temperature, contact pressure, 24 hr.
Resiveld No. 4	Epoxy-polyamide	H. B. Fuller Co.	Room temperature, contact pressure, 24 hr.
Narmco 3135	Epoxy-polyamide	Narmco Resins & Coatings Company	Room temperature, contact pressure, 24 hr.

TABLE 3

Results On Lap-Shear Tests On  $\frac{1}{4}$ -Inch Overlap  
Specimens Bonded With Metlbond 406 Adhesive

ADHERENDS: .020" EFH 301 CRES

Temperature	Lap	Type of Failure		Adhesive	Remarks
	Strength psi	Cohesive, %	Adhesive, %	Thickness Mils	
78°F	5250	60	40	5.3	-
	5860	70	30	3.1	-
	5990	80	20	4.1	-
	5960	80	20	1.3	-
	<u>6000</u>	75	25	3.6	-
Average	5810				
-100°F	8160	80	20	2.0	-
	8550	85	15	2.6	-
	8400	80	20	1.8	-
	7770+	-	-	2.4	Broke in doublers
	<u>6050</u>	80	20	6.0	-
Average	7790+				
-320°F	7040	85	15	0.7	-
	6110	100	0	2.5	-
	4920	100	0	3.3	-
	5790	75	25	2.0	-
	<u>6250</u>	95	5	6.2	-
Average	6020				
-423°F	4380	100	0	1.9	-
	6770	100	0	1.7	-
	7590+	-	-	2.2	Broke in doublers
	7800+	-	-	1.9	Broke in doublers
	<u>7100</u>	100	0	5.3	-
Average	6730+				

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM

78°F	6030+	-	-	2.0	Broke in doublers
	5830+	-	-	2.0	Broke in doublers
	4910	25	75	1.7	-
	<u>6390+</u>	-	-	2.5	Broke in doublers
Average	5790+				

**TABLE 3 (cont'd)**

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
<b>ADHERENDS: .064" 2024 T-3 BARE ALUMINUM (cont'd)</b>					
-100°F	5050	-	-	2.0	Broke in doublers
	5230	-	-	1.9	Broke in doublers
	5890	-	-	3.0	Broke in doublers
	6380	-	-	2.5	Broke in doublers
	<u>4790</u>	-	-	2.8	Broke in doublers
Average	5470+				
-320°F	5950	90	10	2.2	-
	4930	50	50	3.0	-
	4600	0	100	3.2	-
	4900	10	90	5.0	-
	<u>4880</u>	10	90	3.0	-
Average	5050				
-423°F	3960	15	85	4.0	-
	4970	0	100	3.5	-
	4800	0	100	4.0	-
	4660	0	100	4.6	-
	<u>4500</u>	0	100	4.8	-
Average	4580				
<b>ADHERENDS: 020" A-110-AT TITANIUM</b>					
78°F	4260	50	50	4.8	-
	4430	60	40	2.5	-
	4750	65	35	3.5	-
	3920	50	50	3.5	-
	<u>3460</u>	60	40	2.0	-
Average	4160				
-100°F	6500+	-	-	3.2	Broke in doublers
	4240	70	30	6.0	-
	4410	60	40	6.0	-
	8080	85	15	2.0	-
	<u>7340</u>	80	20	2.8	-
Average	6110+				
-320°F	5750	100	0	3.2	-
	5930	100	0	3.2	-
	5380	100	0	3.2	-
	4870	100	0	2.0	-
	<u>4210</u>	90	10	4.5	-
Average	5230				

TABLE 3 (cont'd)

<u>Temperature</u>	<u>Lap Strength</u> psi	<u>Type of Failure</u>		<u>Adhesive Thickness</u> Mils	<u>Remarks</u>
		<u>Cohesive,%</u>	<u>Adhesive,%</u>		
<u>ADHERENDS: 020" A-110-AT TITANIUM (cont'd)</u>					
-423°F	3200	100	0	3.7	-
	3520	100	0	5.0	-
	3390	100	0	4.5	-
	2920	100	0	5.3	-
	<u>4160</u>	100	0	3.0	-
Average	3440				
<u>ADHERENDS: .125" CONOLON 506 (PHENOLIC - FIBERGLASS LAMINATES)</u>					
78°F	2220	40	-	-	60% interlaminar failure
	2350	-	-	-	100% interlaminar failure
	2300	-	-	-	100% interlaminar failure
	2190	-	-	-	100% interlaminar failure
	<u>2550</u>	-	-	-	100% interlaminar failure
Average	2300				
-100°F	2670	-	-	-	100% interlaminar failure
	2640	-	-	-	100% interlaminar failure
	2590	-	-	-	100% interlaminar failure
	2540	-	-	-	100% interlaminar failure
	<u>2470</u>	-	-	-	100% interlaminar failure
Average	2570				
-320°F	2350	-	-	-	100% interlaminar failure
	2350	-	-	-	100% interlaminar failure
	2610	-	-	-	100% interlaminar failure
	2520	-	-	-	100% interlaminar failure
	<u>2480</u>	-	-	-	100% interlaminar failure
Average	2460				
-423°F	2580	-	-	-	100% interlaminar failure
	2310	-	-	-	100% interlaminar failure
	2310	-	-	-	100% interlaminar failure
	2590	-	-	-	100% interlaminar failure
	<u>2510</u>	-	-	-	100% interlaminar failure
Average	2460				

TABLE 3 (cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
<u>ADHERENDS: 125" CONOLON 527 (POLYESTER-FIBERGLASS LAMINATES)</u>					
78°F	2280	0	100	-	-
	2240	0	100	-	-
	2190	0	100	-	-
	2020	0	100	-	-
	<u>2250</u>	0	100	-	-
Average	2200				
<hr/>					
-100°F	1650	0	100	-	-
	1580	0	100	-	-
	1590	0	100	-	-
	1370	0	100	-	-
	<u>1490</u>	0	100	-	-
Average	1540				
<hr/>					
-320°F	1440	0	100	-	-
	1220	0	100	-	-
	1440	0	100	-	-
	1410	0	100	-	-
	<u>1070</u>	0	100	-	-
Average	1320				
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-423°F	1180	0	100	-	-
	1210	0	100	-	-
	1170	0	100	-	-
	1460	0	100	-	-
	<u>1380</u>	0	100	-	-
Average	1280				
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**TABLE 4**

**Results On Lap-Shear Tests On 1/2-Inch Overlap  
Specimens Bonded With AF-40 Adhesive**

**ADHERENDS: .020" EFH 301 CRES**

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	3120	45	55	4.0	-
	3260	50	50	4.0	-
	3210	45	55	4.0	-
	3080	45	55	3.5	-
	<u>2590</u>	45	55	3.0	-
Average	3050				
-100°F	8980+	-	-	5.0	Broke in doublers
	7670	15	85	4.5	-
	9010	20	80	3.0	-
	8750+	-	-	4.0	Broke in doublers
	<u>8350+</u>	-	-	3.5	Broke in doublers
Average	8550				
-320°F	4730	0	100	5.0	-
	5110	0	100	5.0	-
	5380	0	100	4.5	-
	5930	0	100	4.0	-
	<u>6230</u>	0	100	3.0	-
Average	5480				
-423°F	4310	0	100	5.0	-
	4550	0	100	5.0	-
	4190	0	100	4.5	-
	4610	0	100	4.0	-
	7220	0	100	3.0	-
	<u>6290</u>	0	100	-	-
Average	5200				
<b><u>ADHERENDS: .020" EFH 301 CRES PRIMED WITH EC-1956</u></b>					
78°F	5120	40	60	4.5	-
	5090	45	55	4.0	-
	4460	20	80	4.5	-
	4560	55	45	4.0	-
	<u>4810</u>	50	50	4.0	-
Average	4810				

TABLE 4 (Cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-100°F	8690+	-	-	4.5	Broke in doublers
	8560+	-	-	4.0	Broke in doublers
	8820+	-	-	4.5	Broke in doublers
	9020+	-	-	4.0	Broke in doublers
	<u>8490+</u>	-	-	4.0	Broke in doublers
Average	8720+				
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-320°F	4920	0	100	3.5	-
	5660	0	100	3.5	-
	5260	0	100	4.7	-
	5370	0	100	4.5	-
	<u>5190</u>	0	100	4.5	-
Average	5280				
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-423°F	3740	0	100	4.0	-
	3770	0	100	4.0	-
	2360	0	100	4.0	-
	3050	0	100	4.0	-
	<u>2580</u>	0	100	4.0	-
Average	3100				
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<u>ADHERENDS: .064" 2024 T-3 BARE ALUMINUM PRIMED WITH EC-1956</u>					
78°F	3770	20	80	7.0	-
	4310	20	80	7.0	-
	4620	15	85	4.7	-
	4180	45	55	6.0	-
	<u>3960</u>	45	55	7.0	-
Average	4170				
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-100°F	5870+	-	-	5.0	Broke in doublers
	5040+	-	-	6.5	Broke in doublers
	6210+	-	-	4.5	Broke in doublers
	5230+	-	-	4.0	Broke in doublers
	<u>5410+</u>	-	-	4.0	Broke in doublers
Average	5550+				
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-320°F	2710	0	100	4.0	-
	3380	0	100	5.0	-
	2760	0	100	7.0	-
	4520	0	100	5.0	-
	<u>3140</u>	0	100	6.0	-
Average	3300				

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM PRIMED WITH EC-1956 (Cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-423°F	2860	0	100	6.0	-
	2310	0	100	7.0	-
	2340	0	100	7.0	-
	2560	0	100	4.5	-
	<u>2470</u>	0	100	4.0	-
Average	2510				

ADHERENDS: .020" A-110-AT TITANIUM PRIMED WITH EC-1956

78°F	5010	70	30	5.0	-
	4180	60	40	6.0	-
	4870	45	55	5.0	-
	<u>3810</u>	50	50	5.0	-
Average	4470				

-100°F	8150+	-	-	4.0	Broke in doublers
	7940+	-	-	4.0	Broke in base material
	7490+	-	-	4.0	Broke in doublers
	<u>8530+</u>	-	-	3.5	Broke in doublers
Average	8030+				

-320°F	4410	0	100	3.5	-
	4780	0	100	4.0	-
	3700	0	100	3.5	-
	3910	0	100	4.0	-
	<u>3130</u>	0	100	4.0	-
Average	4000				

-423°F	2330	0	100	5.0	-
	2500	0	100	5.0	-
	1910	0	100	4.0	-
	2860	0	100	4.5	-
	<u>1560</u>	0	100	5.0	-
Average	2230				

**TABLE 5**

Results On Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With FM-1000 Adhesive

ADHERENTS: .020" EFH 301 CRES

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	3950	50	50	Film thickness varied from 1.0 to 10.0 mils	
	3780	80	20	Film thickness varied from 1.0 to 10.0 mils	
	3400	40	60	Film thickness varied from 1.0 to 10.0 mils	
	3060	65	35	Film thickness varied from 1.0 to 10.0 mils	
	<u>4450</u>	55	45	Film thickness varied from 1.0 to 10.0 mils	
Average	3730				
-100°F	7370+	-	-	2.0-8.0	Broke in doublers
	7620	80	20	1.0-8.0	-
	7320	85	15	1.5-9.0	-
	6940	90	10	1.0-5.0	-
	<u>8010</u>	85	15	1.0-10.0	-
Average	7450+				
-320°F	4630	50	50	1.0-10.0	-
	3860	50	50	1.0- 9.0	-
	4510	25	75	1.0- 9.0	-
	2940	50	50	1.0-10.0	-
	<u>5610</u>	35	65	1.0- 9.4	-
Average	4310				
-423°F	2780	30	70	2.0-12.0	-
	3890	10	90	1.0- 8.0	-
	2970	15	85	2.0-10.0	-
	2600	35	65	1.0-10-0	-
	<u>6520</u>	40	60	1.0-3.5	-
Average	3750				

TABLE 5 (Cont'd)

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	5030	100	0	1.5	-
	6230	85	15	0.7	-
	6850	75	25	1.0	-
	6120	75	25	1.0	-
	<u>6310</u>	80	20	1.0	-
Average	6110				
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-100°F	5230+	-	-	-	Broke in doublers
	5240+	-	-	-	Broke in doublers
	4960+	-	-	-	Broke in doublers
	5560+	-	-	-	Broke in doublers
	<u>5050+</u>	-	-	-	Broke in doublers
Average	5210+				
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-320°F	4980	50	50	2.3	-
	4020	80	20	2.0	-
	4090	60	40	2.3	-
	2360	90	10	1.5	-
	<u>3480</u>	60	40	1.7	-
Average	3790				
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-423°F	3330	60	40	1.8	-
	3500	75	25	2.0	-
	3260	50	50	2.5	-
	3600	50	50	2.0	-
	<u>3160</u>	50	50	2.0	-
Average	3370				

**TABLE 6**

Results on Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With Metlbond 4041 Adhesive

ADHERENDS: .020" EFH 301 CRES PRIMED WITH 4041 TYPE II

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	2640	75	25	4.0	-
	2290	20	80	5.0	-
	2240	70	30	6.0	-
	2820	40	60	5.3	-
	<u>2910</u>	15	85	5.0	-
	Average	2580			
-100°F	9360	0	100	5.5	-
	7200	0	100	6.5	-
	7770	0	100	6.0	-
	9410	0	100	5.0	-
	<u>9750+</u>	-	-	4.0	Broke in doublers
	Average	8700+			
-320°F	5010	0	100	5.0	-
	6460	0	100	5.0	-
	5900	0	100	4.0	-
	5050	0	100	6.0	-
	<u>6620</u>	0	100	6.0	-
	Average	5810			
-423°F	2100	0	100	6.5	-
	2010	0	100	5.5	-
	2020	0	100	5.0	-
	2400	0	100	4.5	-
	<u>1820</u>	0	100	6.0	-
	Average	2070			

TABLE 6 (Cont'd)

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM PRIMED WITH 4041 TYPE II

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive.%	Adhesive.%		
78°F	3300	75	25	3.5	-
	3220	75	25	4.5	-
	1950	85	15	6.0	-
	3080	80	20	5.5	-
	<u>3250</u>	70	30	4.5	-
Average	2960				
<hr/>					
-100°F	5050	60	40	4.8	-
	5090	60	40	5.0	-
	5230	60	40	4.5	-
	4810	60	40	5.5	-
	<u>4920</u>	60	40	4.5	-
Average	5020				
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-320°F	4380	0	100	4.0	-
	4220	0	100	4.5	-
	4130	3	97	4.0	-
	4440	5	95	4.0	-
	<u>4820</u>	3	97	4.8	-
Average	4400				
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-423°F	1720	0	100	4.0	-
	1840	0	100	4.3	-
	1660	0	100	4.0	-
	1700	0	100	4.5	-
	<u>1620</u>	0	100	5.0	-
Average	1710				

ADHERENDS: .020" A-110-AT TITANIUM PRIMED WITH 4041 TYPE II

78°F	1510	20	80	5.5	-
	1410	15	85	7.0	-
	1530	25	75	4.5	-
	1880	35	65	5.0	-
	<u>1630</u>	35	65	4.8	-
Average	1590				

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**TABLE 6 (Cont'd)**

**ADHERENDS: .020" AT-110-AT TITANIUM PRIMED WITH 4041 TYPE II (Cont'd)**

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-100°F	6320	25	75	4.5	Failure between film & primer
	7370	50	50	5.0	Failure between film & primer
	4770	30	70	4.0	Failure between film & primer
	5150	25	75	6.0	Failure between film & primer
	<u>7480</u>	45	55	4.8	Failure between film & primer
Average	6220				
-320°F	2860	0	100	6.0	Failure between film & primer
	5950	0	100	4.3	Failure between film & primer
	1990	0	100	6.8	Failure between film & primer
	2720	0	100	4.5	Failure between film & primer
	<u>4940</u>	0	100	4.3	Failure between film & primer
Average	3690				
-423°F	1360	0	100	5.0	Failure between film & primer
	1320	0	100	4.3	Failure between film & primer
	1400	0	100	6.3	Failure between film & primer
	1600	0	100	5.5	Failure between film & primer
	<u>1310</u>	0	100	5.0	Failure between film & primer
Average	1420				

**ADHERENDS: .125" CONOLON 506 (PHENOLIC-FIBERGLASS LAMINATE)**

78°F	1980	0	0	-	100% interlaminar failure
	1810	0	0	-	100% interlaminar failure
	1790	0	0	-	100% interlaminar failure
	2300	0	100	-	-
	<u>2140</u>	0	100	-	-
Average	2060				
-100°F	1480	0	50	-	50% interlaminar failure
	1560	0	0	-	100% interlaminar failure
	2640	0	65	-	35% interlaminar failure
	2630	0	100	-	-
	<u>1580</u>	0	20	-	80% interlaminar failure
Average	1980				

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FIGURE 6 (Cont'd)

ADHERENDS: .125" CONOLON 506 (PHENOLIC-FIBERGLASS LAMINATE) (Cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-320°F	1300	0	100	-	-
	960	0	100	-	-
	1990	0	100	-	-
	1840	0	100	-	-
	<u>1240</u>	0	100	-	-
Average	1470				
<hr/>					
-423°F	2200	0	100	-	-
	2120	0	100	-	-
	1800	0	100	-	-
	970	0	100	-	-
	<u>1840</u>	0	100	-	-
Average	1790				

TABLE 7

Results On Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With AF-32 Adhesive

ADHERENDS: .020" EFH 301 CRES PRIMED WITH EC-1660

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	3260	0	100	6.5	85% Failure between film & primer
	3650	0	100	6.0	100% Failure between film & primer
	4190	0	100	6.0	100% Failure between film & primer
	<u>3480</u>	0	100	6.0	85% Failure between film & primer
Average	3650				
-100°F	7910+	-	-	5.0	Broke in doublers
	8310+	-	-	6.0	Broke in doublers
	8810+	-	-	6.0	Broke in doublers
	<u>8920</u>	0	100	5.0	100% Failure between film & primer
Average	8490+				
-320°F	2760	0	100	6.0	100% Failure between film & primer
	3800	0	100	6.5	100% Failure between film & primer
	3940	0	100	6.5	100% Failure between film & primer
	<u>3890</u>	0	100	6.5	100% Failure between film & primer
Average	3600				
-423°F	2530	0	100	6.0	100% Failure between film & primer
	2880	0	100	5.5	100% Failure between film & primer
	2840	0	100	6.0	100% Failure between film & primer
	<u>2740</u>	0	100	6.0	100% Failure between film & primer
Average	2750				

**TABLE 8**

Results On Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With Metlbond 302-A Adhesive

ADHERENDS: .020" EFH 301 CRES

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	2370	100	0	5.0	Failure between 112 cloth & adhesive
	2680	100	0	5.5	Failure between 112 cloth & adhesive
	2390	100	0	5.0	Failure between 112 cloth & adhesive
	2340	100	0	6.0	Failure between 112 cloth & adhesive
	<u>2650</u>	100	0	5.0	Failure between 112 cloth & adhesive
Average	2490				
-100°F	2830	100	0	5.0	Failure between 112 cloth & adhesive
	2430	100	0	4.5	Failure between 112 cloth & adhesive
	2990	100	0	4.5	Failure between 112 cloth & adhesive
	2770	100	0	5.0	Failure between 112 cloth & adhesive
	<u>2890</u>	100	0	5.0	Failure between 112 cloth & adhesive
Average	2780				
-320°F	2820	100	0	5.0	Failure between 112 cloth & adhesive
	2650	100	0	5.5	Failure between 112 cloth & adhesive
	2700	100	0	5.0	Failure between 112 cloth & adhesive
	2640	100	0	5.3	Failure between 112 cloth & adhesive
	<u>3210</u>	100	0	5.0	Failure between 112 cloth & adhesive
Average	2800				
-423°F	2380	100	0	6.0	Failure between 112 cloth & adhesive
	2550	100	0	5.7	Failure between 112 cloth & adhesive
	2780	100	0	5.0	Failure between 112 cloth & adhesive
	<u>3030</u>	100	0	4.8	Failure between 112 cloth & adhesive
	Average	2690			

ADHERENDS: .020" A-110-AT TITANIUM

78°F	1120	100	0	4.0	Failure between 112 cloth & adhesive
	710	100	0	5.0	Failure between 112 cloth & adhesive
	920	100	0	8.0	Failure between 112 cloth & adhesive
	680	100	0	6.0	Failure between 112 cloth & adhesive
	<u>590</u>	100	0	5.5	Failure between 112 cloth & adhesive
Average	800				

TABLE 8 (Cont'd)

ADHERENDS: .020" A-110-AT TITANIUM (Cont'd)

Temperature	Lap	Type of Failure		Adhesive	Remarks
	Strength psi	Cohesive, %	Adhesive, %	Thickness Mils	
-100°F	1440	100	0	5.0	Failure between 112 cloth & adhesive
	1110	100	0	5.0	Failure between 112 cloth & adhesive
	1350	100	0	5.5	Failure between 112 cloth & adhesive
	1670	100	0	4.5	Failure between 112 cloth & adhesive
	<u>1630</u>	100	0	6.5	Failure between 112 cloth & adhesive
Average	1440				
-320°F	1870	100	0	3.5	Failure between 112 cloth & adhesive
	1820	100	0	5.0	Failure between 112 cloth & adhesive
	1790	100	0	4.1	Failure between 112 cloth & adhesive
	2090	100	0	4.0	Failure between 112 cloth & adhesive
	<u>2020</u>	100	0	4.2	Failure between 112 cloth & adhesive
Average	1820				
-423°F	1570	100	0	5.5	Failure between 112 cloth & adhesive
	1490	100	0	5.7	Failure between 112 cloth & adhesive
	1540	100	0	5.0	Failure between 112 cloth & adhesive
	1650	100	0	4.8	Failure between 112 cloth & adhesive
	<u>1570</u>	100	0	4.7	Failure between 112 cloth & adhesive
Average	1560				

ADHERENDS: .125" COLOLON 506 (PHENOLIC-FIBERGLASS LAMINATE)

78°F	1720	35	0	-	65% interlaminar failure
	1720	35	0	-	65% interlaminar failure
	1720	30	0	-	70% interlaminar failure
	1510	20	0	-	80% interlaminar failure
	<u>1560</u>	5	0	-	95% interlaminar failure
Average	1650				
-100°F	1650	10	0	-	95% interlaminar failure
	1960	10	0	-	90% interlaminar failure
	2170	75	0	-	25% interlaminar failure
	2360	70	0	-	30% interlaminar failure
	<u>2230</u>	80	0	-	20% interlaminar failure
Average	2070				

**TABLE 8 (Cont'd)**

**ADHERENDS: .125" CONOLON 506 (PHENOLIC-FIBERGLASS LAMINATE) (CONT'D)**

Temperature	Lap Thickness psi	Type of Failure Cohesive, %	Adhesive, %	Adhesive Thickness Mils	Remarks
-320°F	2450	80	0	-	20% interlaminar failure
	2300	40	0	-	60% interlaminar failure
	2460	5	0	-	95% interlaminar failure
	2090	60	0	-	40% interlaminar failure
	<u>2500</u>	70	0	-	30% interlaminar failure
Average	2360				
-423°F	2240	0	0	-	100% interlaminar failure
	2080	0	0	-	100% interlaminar failure
	2620	10	0	-	90% interlaminar failure
	2150	5	0	-	95% interlaminar failure
	<u>2380</u>	15	0	-	85% interlaminar failure
Average	2290				

**TABLE 9**

Results On Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With Narmco 3135 Adhesive

ADHERENDS: .020" EFH 301 CRES

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	1750	0	100	0.2	-
	920	0	100	0.1	-
	1070	0	100	0.4	-
	1450	0	100	1.6	-
	<u>1350</u>	0	100	0.1	-
Average	1310				
-100°F	1730	40	60	1.1	-
	2010	0	100	1.1	-
	1650	0	100	0.4	-
	2200	0	100	0.8	-
	<u>1580</u>	20	80	1.9	-
Average	1830				
-320°F	1600	25	75	0.8	-
	980	0	100	4.4	-
	1820	15	85	1.2	-
	1270	0	100	0.7	-
	<u>1100</u>	10	90	0.9	-
Average	1350				
-423°F	1250	15	85	0.7	-
	890	0	100	0.7	-
	750	0	100	1.2	-
	830	0	100	1.2	-
	<u>980</u>	0	100	1.1	-
Average	940				
<u>ADHERENDS: .020" A-110-AT TITANIUM</u>					
78°F	940	0	100	1.0	-
	1270	0	100	0.5	-
	900	0	100	1.0	-
	1490	25	75	1.0	-
	<u>540</u>	0	100	0.5	-
Average	1040				

TABLE 9 (Cont'd)

ADHERENDS: .020" A-110-AT TITANIUM (Cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-100°F	2110	15	85	0.5	-
	1660	0	100	1.5	-
	1730	35	65	1.0	-
	1920	15	85	1.0	-
	<u>1540</u>	35	65	1.0	-
Average	1790				
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-320°F	910	0	100	0.5	-
	920	0	100	1.0	-
	1170	25	75	1.0	-
	<u>1080</u>	35	65	1.0	-
	Average	1020			
<hr/>					
-423°F	990	25	75	1.0	-
	900	10	90	1.5	-
	1140	10	90	1.0	-
	1050	0	100	1.0	-
	<u>1040</u>	20	80	1.0	-
Average	1020				

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM

78°F	2320	0	100	1.5	-
	1610	0	100	1.5	-
	2270	20	80	-	-
	2250	10	90	2.0	-
	<u>2450</u>	15	85	1.5	-
Average	2180				
<hr/>					
-100°F	1740	20	80	-	-
	2190	10	90	2.0	-
	1660	35	65	-	-
	1820	40	60	1.0	-
	<u>1860</u>	40	60	1.0	-
Average	1850				
<hr/>					
-320°F	1630	50	50	1.7	-
	2120	50	50	1.0	-
	2020	30	70	-	-
	1490	0	100	1.4	-
	<u>1560</u>	15	85	1.4	-
Average	1760				

TABLE 9 (Cont'd)

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM(Cont'd)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive,%	Adhesive,%		
-423°F	1590	75	25	3.0	-
	1570	40	60	3.5	-
	1730	55	45	4.5	-
	1590	65	35	3.0	-
	<u>1740</u>	40	60	2.5	-
Average	1640				

**TABLE 10**

Results On Lap-Shear Tests On  $\frac{1}{2}$ -Inch Overlap  
Specimens Bonded With Resiweld No. 4 Adhesive

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive.%	Adhesive.%		
78°F	2610	90	10	0.3	-
	2140	75	25	0.2	-
	2660	15	85	0.1	-
	2670	90	10	0.5	-
	2210	20	80	0.5	-
	<u>2450</u>	100	0	0.4	-
Average	2460				
<hr/>					
-100°F	2160	0	100	0.3	-
	2770	30	70	0.5	-
	2660	75	25	0.6	-
	2480	0	100	0.3	-
	<u>1820</u>	20	80	0.2	-
	Average	2380			
<hr/>					
-320°F	2180	0	100	0.2	-
	1760	0	100	0.4	-
	1870	100	0	0.7	-
	<u>1800</u>	100	0	0.2	-
	Average	1900			
<hr/>					
-423°F	2100	0	100	1.0	-
	2010	75	25	1.0	-
	2060	0	100	1.0	-
	2050	0	100	1.0	-
	<u>1830</u>	0	100	1.0	-
	Average	2010			

**TABLE 11**

Results On Lap-Shear Tests On  $\frac{1}{8}$ -Inch Overlap  
Specimens Bonded With APCO 1219 Adhesive

**ADHERENDS: .020" EFH 301 CRCS**

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	550	0	100	0.6	-
	640	0	100	0.2	-
	820	45	55	0.3	-
	1120	90	10	0.3	-
	<u>1090</u>	0	100	0.2	-
Average	840				
<hr/>					
-100°F	2410	0	100	0.6	-
	2400	0	100	0.3	-
	2490	0	100	0.3	-
	2180	0	100	0.4	-
	<u>1550</u>	15	85	0.3	-
Average	2210				
<hr/>					
-320°F	570	0	100	0.4	-
	1930	0	100	0.5	-
	1900	20	80	0.4	-
	2420	20	80	0.2	-
	<u>1870</u>	30	70	0.4	-
Average	1640				
<hr/>					
-423°F	1900	30	70	0.5	-
	750	40	60	0.3	-
	1140	0	100	0.3	-
	840	10	90	0.2	-
	<u>490</u>	15	85	0.3	-
Average	1020				

**ADHERENDS: .064" 2024 T-3 BARE ALUMINUM**

78°F	1210	-	-	2 1.0
	1350	-	-	2 1.0
	1410	-	-	2 1.0
	1260	-	-	2 1.0
	<u>1320</u>	-	-	2 1.0
Average	1310			

TABLE 11 (Cont'd)

ADHERENDS: .064" 2024 T-3 BARE ALUMINUM (CONT'D)

Temperature	Lap Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
-100°F	3170	-	-	41.0	-
	3760	-	-	41.0	-
	2660	-	-	41.0	-
	2500	-	-	41.0	-
	<u>3410</u>	-	-	41.0	-
Average	3100				
<hr/>					
-320°F	2370	-	-	41.0	-
	3160	-	-	41.0	-
	3200	-	-	41.0	-
	2970	-	-	41.0	-
	<u>3220</u>	-	-	41.0	-
Average	2980				
<hr/>					
-423°F	2430	-	-	41.0	-
	2590	-	-	41.0	-
	2510	-	-	41.0	-
	2970	-	-	41.0	-
	<u>3040</u>	-	-	41.0	-
Average	2710				

**TABLE 12**

**Results On Butt-Tensile Specimens  
Bonded With AF-40 Adhesive**

**ADHERENDS: 3/4" ROUND STOCK 321 STAINLESS STEEL**

Temperature	Tensile Strength psi	Type of Failure		Adhesive Thickness Mils	Remarks
		Cohesive, %	Adhesive, %		
78°F	4750	40	60	10.0	-
	3350	65	35	10.0	-
	2890	25	75	18.0	-
	<u>5390</u>	40	60	7.0	-
Average	4100				
-100°F	9910	100	0	4.0	-
	7780	100	0	6.0	-
	18420	50	50	2.5	-
	19250	70	30	7.0	-
	<u>19460</u>	60	40	9.0	-
Average	14960				
-320°F	27940	100	0	3.0	-
	11090	75	25	13.0	-
	15250	90	10	20.0	-
	15340	90	10	6.5	-
	<u>16340</u>	90	10	9.0	-
Average	17190				
-423°F	13800	-	-	15.0	Type of failure not recorded
	11670	-	-	13.0	Type of failure not recorded
	15340	85	15	7.0	-
	15390	97	3	6.0	-
	<u>25340</u>	100	0	7.0	-
Average	16310				

**TABLE 13****Adhesives Evaluated by NBS & FPL**

<u>Commercial Designation</u>	<u>Adhesive Type</u>	<u>Supplier</u>
Bondmaster M24B	Rubber-Epoxide Phenolic	Rubber and Asbestos Corporation
Swedlow 371W	Vinyl and Phenolic Resins in Organic Solvent	Swedlow Plastics Company
EC-1469	Liquid Epoxide with Filler	Minnesota Mining and Manufacturing Company
Metlbond 4021	Nitrile Rubber-Phenolic Primer and Film	Narmco Resins and Coatings Company
FM-47	Vinyl-Phenolic Primer with Fiber Glass Film	Bloomingtondale Rubber Company
Redux 775	Film of Phenol Resin Solution and Vinyl Polymer Powder	Ciba Company, Inc.
AF-5930	Nitrile Rubber-Phenolic Primer and Film	Minnesota Mining and Manufacturing Company
EC-1471	Vinyl-Phenolic Liquid Resins	Minnesota Mining and Manufacturing Company
EPON 422	Epoxy-Phenolic Resins Supported on Glass Cloth	Shell Chemical Corporation
Metlbond 302	Epoxy-Phenolic Resins Supported on Glass Cloth	Narmco Resins and Coatings Company

TABLE 14

Adhesives Evaluated by Denver Research Institute

<u>Commercial Designation</u>	<u>Adhesive Type</u>	<u>Supplier</u>
Bondmaster M-653	Epoxy	Rubber and Asbestos Corporation
Bondmaster M-648	Epoxy	Rubber and Asbestos Corporation
Armstrong A-4	Filled Epoxy	Armstrong Products Company
Metlbond MN3C	Neoprene-Phenolic on Nylon Tape	Narmco Resins and Coatings Company
Scotchweld AF-13	Nitrile-Phenolic (Unsupported Film)	Minnesota Mining and Manufacturing Company
Scotchweld 583	Nitrile-Phenolic (Unsupported Film)	Minnesota Mining and Manufacturing Company

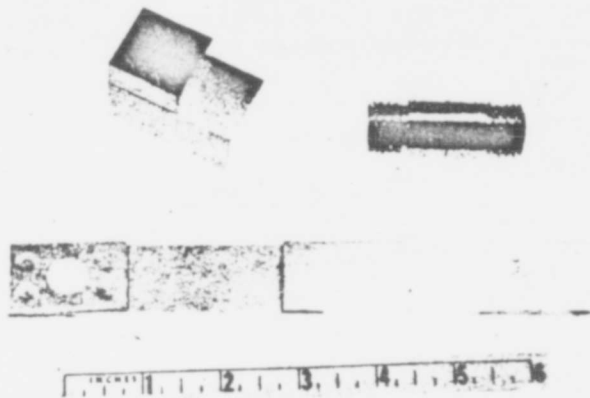


Figure 1. Lap-Shear, Butt-Tensile and Impact Specimens Utilized in REA 111-9106

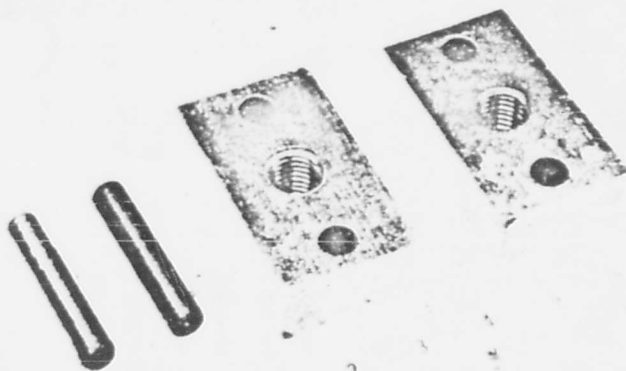


Figure 2. Butt-Tensile Specimen Alignment Jig

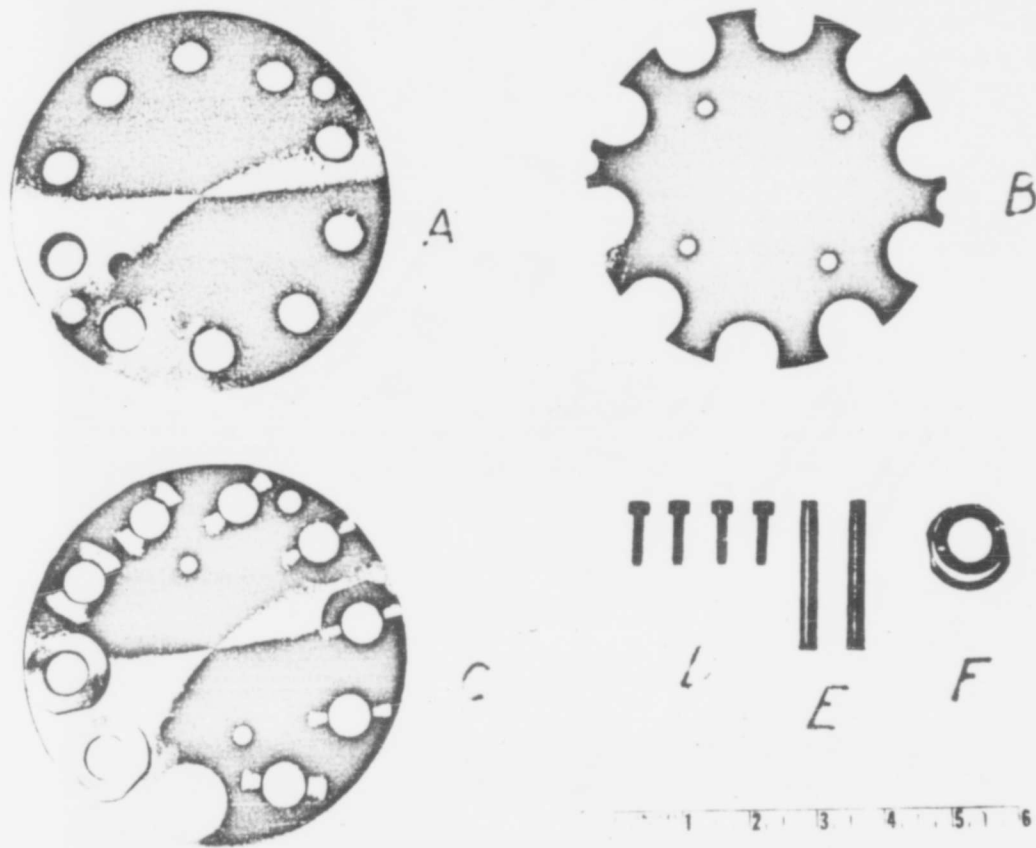


Figure 3. Advance Design Butt-Tensile Specimen Alignment Jig - consists of upper plate (A), gusset plate (B), lower plate (C), screws (D), alignment pins (E) and internally threaded flanged sleeve (F).

NOTE: ALL POINTS ARE AVERAGES OF FIVE SPECIMENS UNLESS OTHERWISE NOTED.

- ADHERENDS:
- .020" EFH 301 CRES
  - .064" 2024 T-3 BARE ALUMINUM
  - △ .020" A-110-AT TITANIUM
  - D .125" CONOLON 506 (PHENOLIC-FIBERGLASS LAMINATE)
  - .125" CONOLON 527 (POLYESTER)

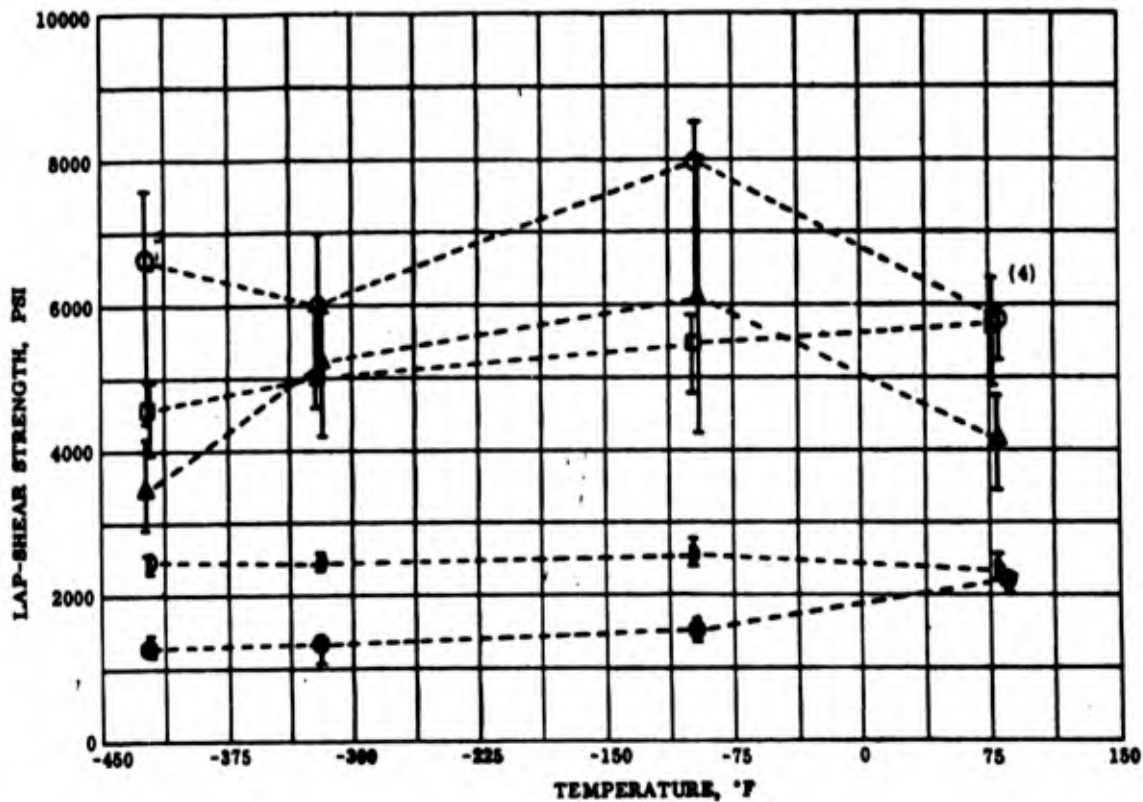


Fig. 4. Lap-Shear Strength of Metlbond 406 Adhesive Bonds vs. Temperature

NOTE: PRIMER WAS EC-1976  
 ALL POINTS ARE AVERAGES OF FIVE  
 SPECIMENS UNLESS OTHERWISE NOTED

ADHERENDS:  
 ○ .020" EFH 301 CRES PRIMED  
 ◊ .020" EFH 301 CRES UNPRIMED  
 □ .063" 2024 T-3 BARE Al PRIMED  
 ▲ .020" A-110-AT TITANIUM PRIMED

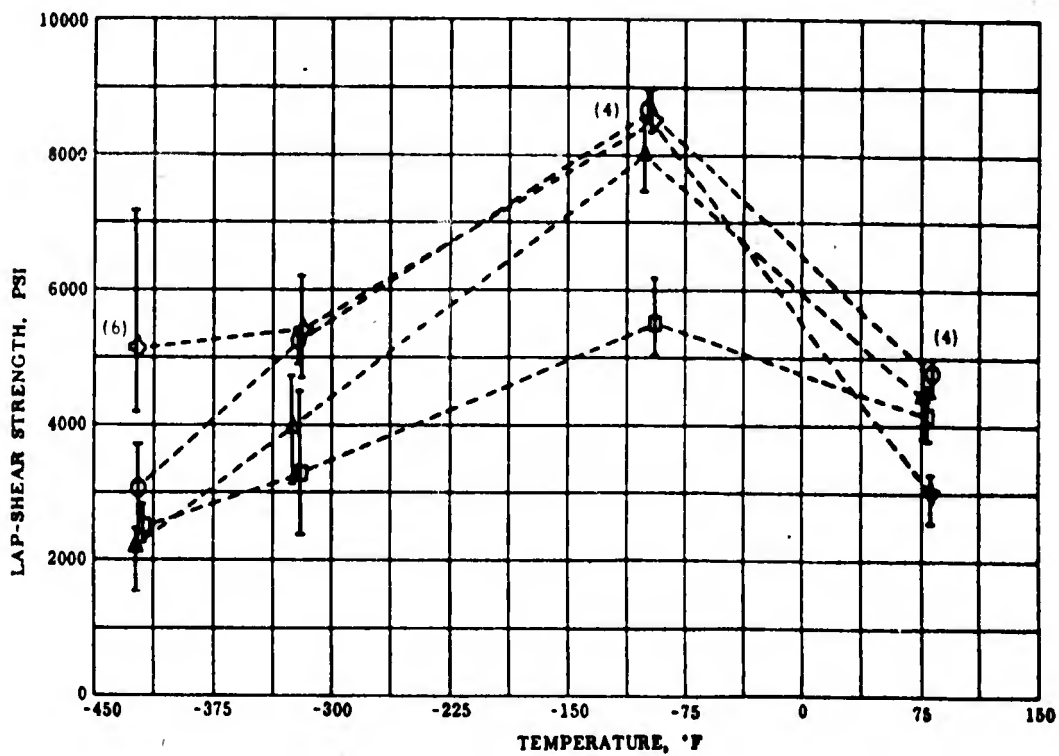


Fig. 5. Lap-Shear Strength of AF-40 Adhesive Bonds vs. Temperature

ADHERENDS:  
 ○ .030" 57H 501 CRES  
 □ .064" 2024 T-3 BARE ALUMINUM  
 NOTE: ALL POINTS ARE AVERAGES  
 OF FIVE SPECIMENS

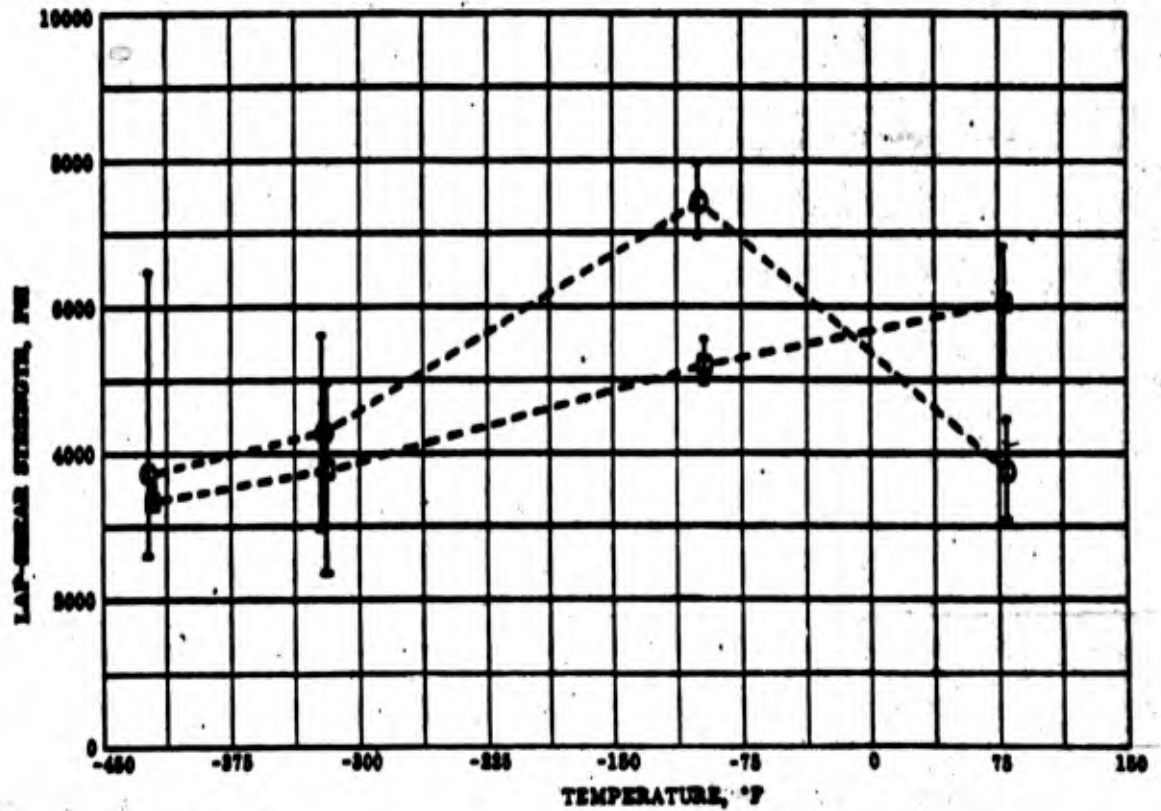


Fig. 6. Lap-Shear Strength of FM-1000 Adhesive Bonds vs. Temperature

NOTE: STAINLESS STEEL, ALUMINUM AND  
TITANIUM ADHERENDS WERE PRIMED  
WITH METLBOND 4041 TYPE D  
ALL POINTS ARE AVERAGES OF FIVE  
SPECIMENS

ADHERENDS:  
○ .020" EFH 301 CRES  
□ .064" 2024-T-3 BARE ALUMINUM  
△ .020" A-110-AT TITANIUM  
◇ 1/2" CONOLON 506 (PHENOLIC-  
FIBERGLASS LAMINATE)

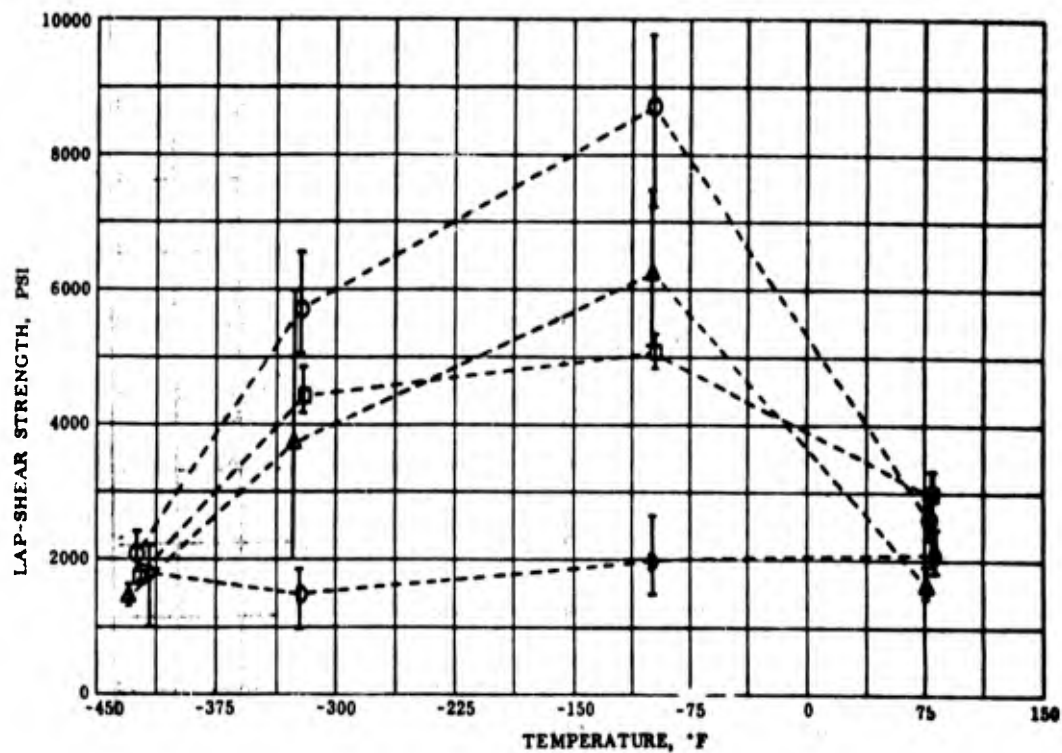


Fig. 7. Lap-Shear Strength of Metlbond 4041 Adhesive Bonds vs. Temperature

A

ADHERENDS:  
○ .020" EFH 301 CRES PRIMED WITH  
EC-1640

NOTE: ALL POINTS ARE AVERAGES OF  
FOUR SPECIMENS UNLESS OTHER-  
WISE NOTED

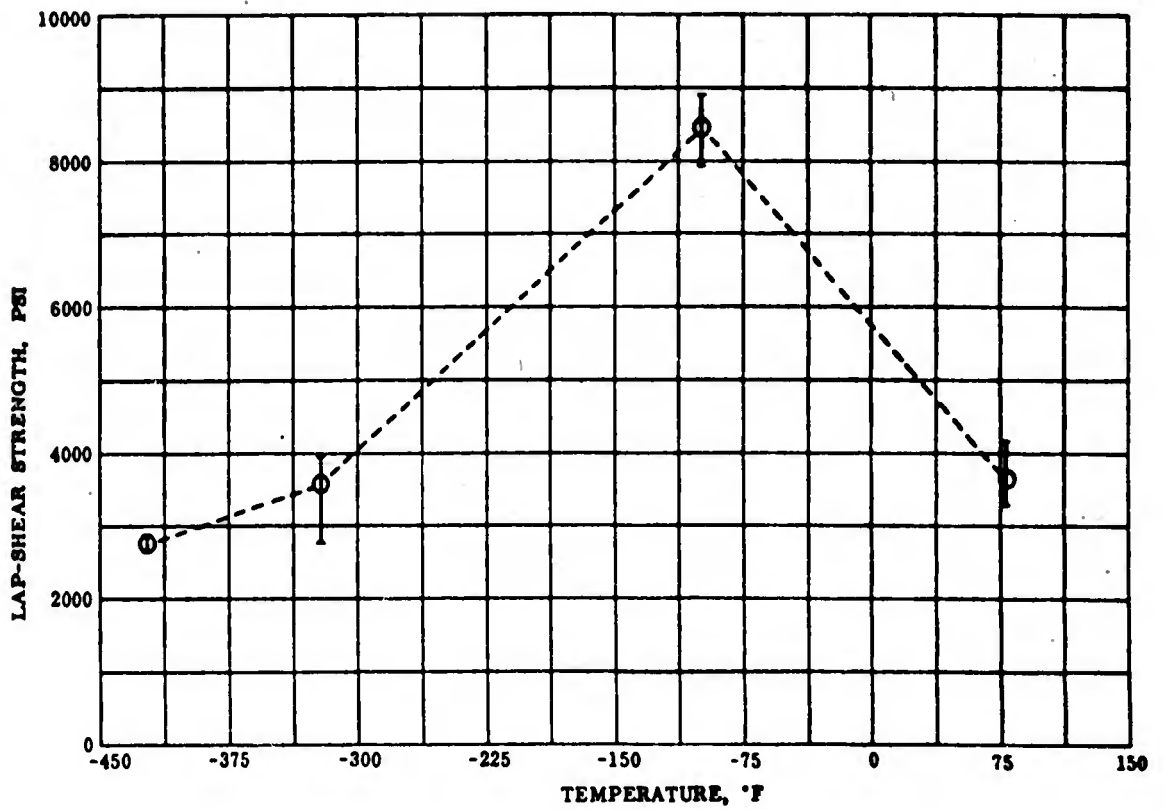


Fig. 8. Lap-Shear Strength of AF-32 Adhesive Bonds vs. Temperature

NOTED: ALL POINTS ARE AVERAGES OF FIVE SPECIMENS UNLESS OTHERWISE NOTED

ADHERENDS:  
○ .020" EFH 301 CRES  
△ .020" A-110-AT TITANIUM  
D .125" CONOLON 506 (PHENOLIC-FIBERGLASS LAMINATE)

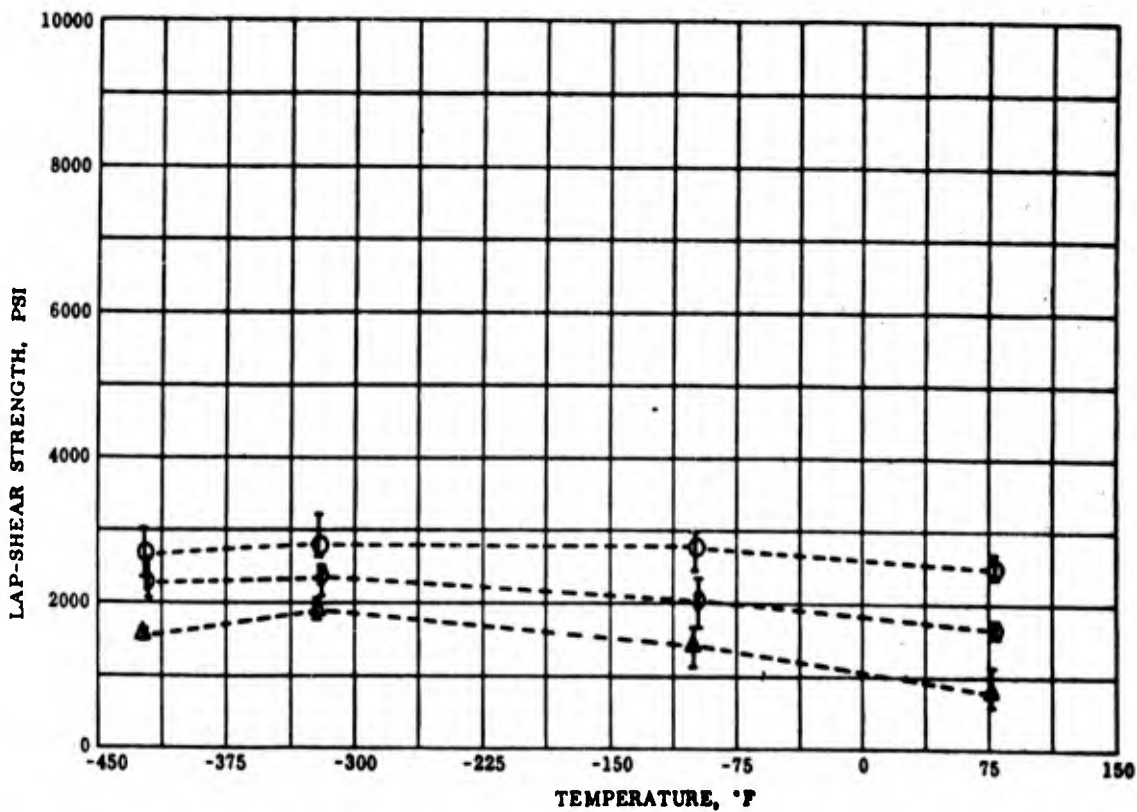


Fig. 9. Lap-Shear Strength of Metlbond 302-A Adhesive Bonds vs. Temperature

NOTE: ALL POINTS ARE AVERAGES OF FIVE SPECIMENS UNLESS OTHERWISE NOTED

ADHERENDS:  
○ .020" EFH 301 CRES  
□ .064" 2024 T-3 BARE ALUMINUM  
△ .020" A-110-AT TITANIUM

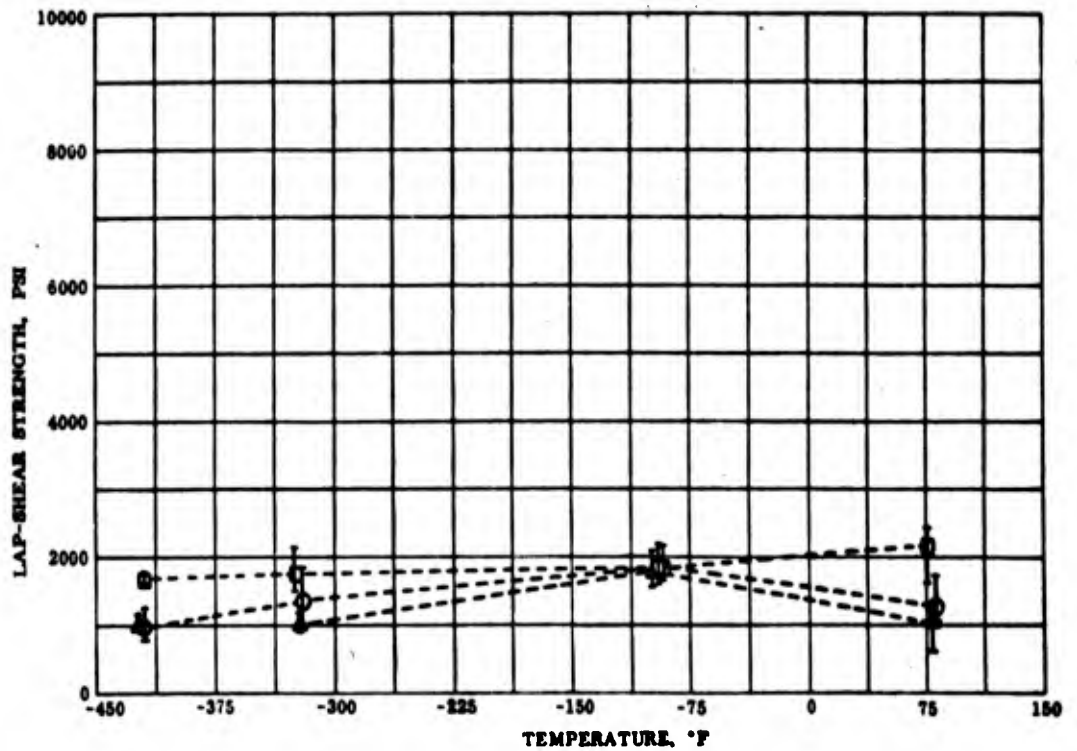


Fig. 10. Lap-Shear Strength of Narmco 3135 Adhesive Bonds vs. Temperature

ADHEREND  
□ .064" 2024 T-3 BARE ALUMINUM

NOTE: ALL POINTS ARE AVERAGES  
OF FIVE SPECIMENS

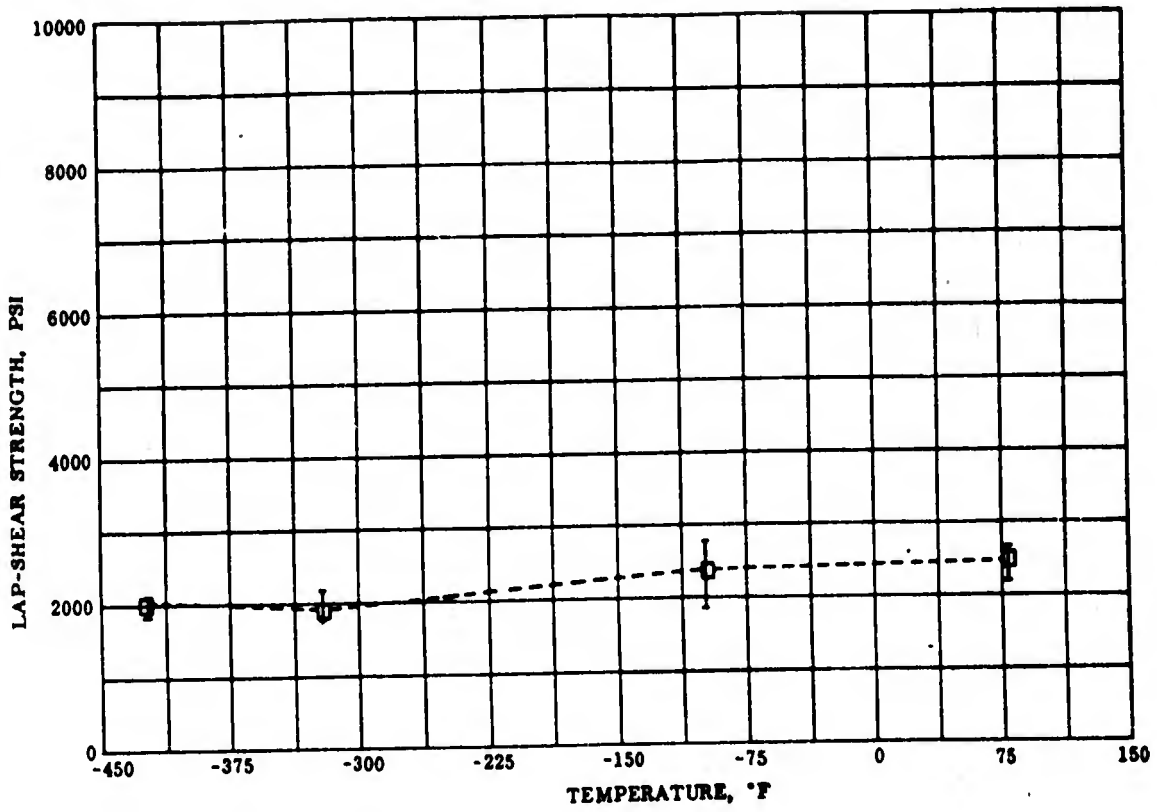


Fig. 11. Lap-Shear Strength of Resiweld No. 4 Adhesive Bonds vs. Temperature

ADHERENDS:

○ .020" EFH 301 CRES

□ .064" 2024 T-3 BARE ALUMINUM

NOTE: ALL POINTS ARE AVERAGES OF FIVE SPECIMENS

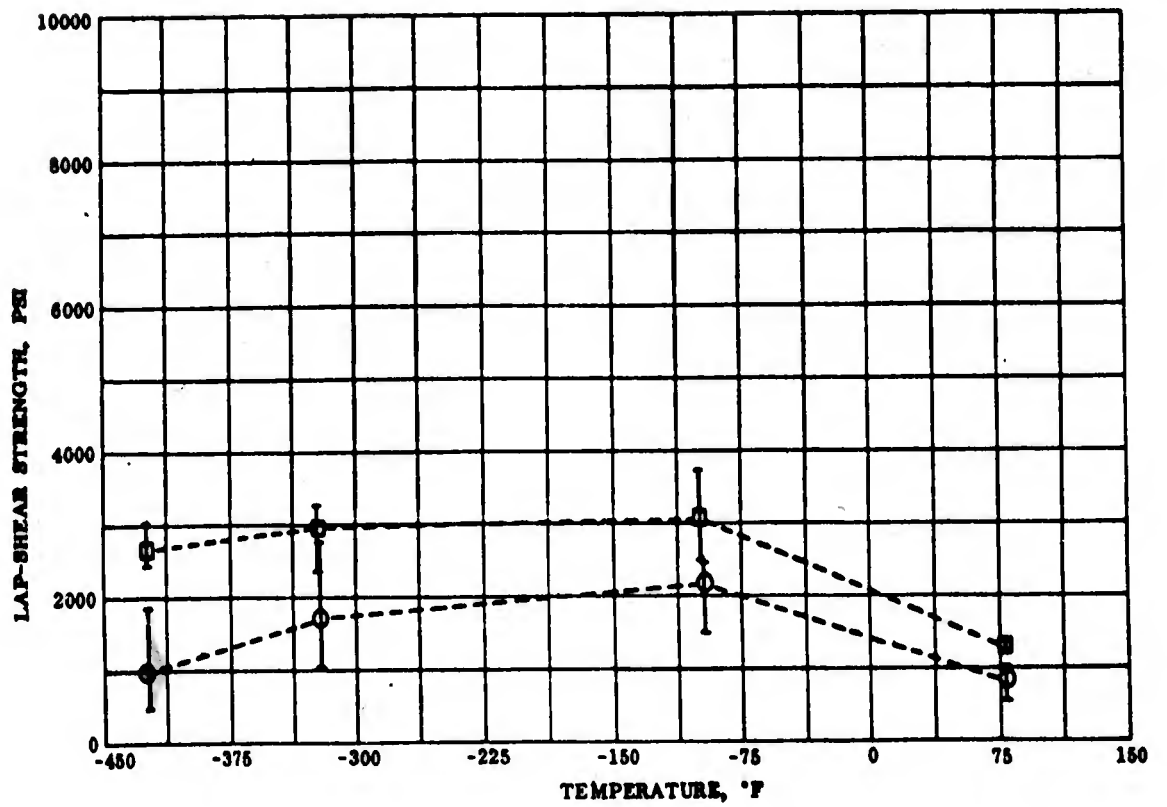


Fig. 12. Lap-Shear Strength of APCO 1219 Adhesive Bonds vs. Temperature

ADHERENDS: .063" TYPE 301, 1/2H, 2B FINISH STAINLESS STEEL  
 ▲ EPON 422, FOREST PRODUCTS LABORATORY, 6 SPEC.  
 ○ EPON 422, NES GRADUAL COOLING, 5 SPEC.  
 △ METLBOND 302, FOREST PRODUCTS LABORATORY, 6 SPEC.  
 □ METLBOND 302, NBS GRADUAL COOLING, 3 SPEC.

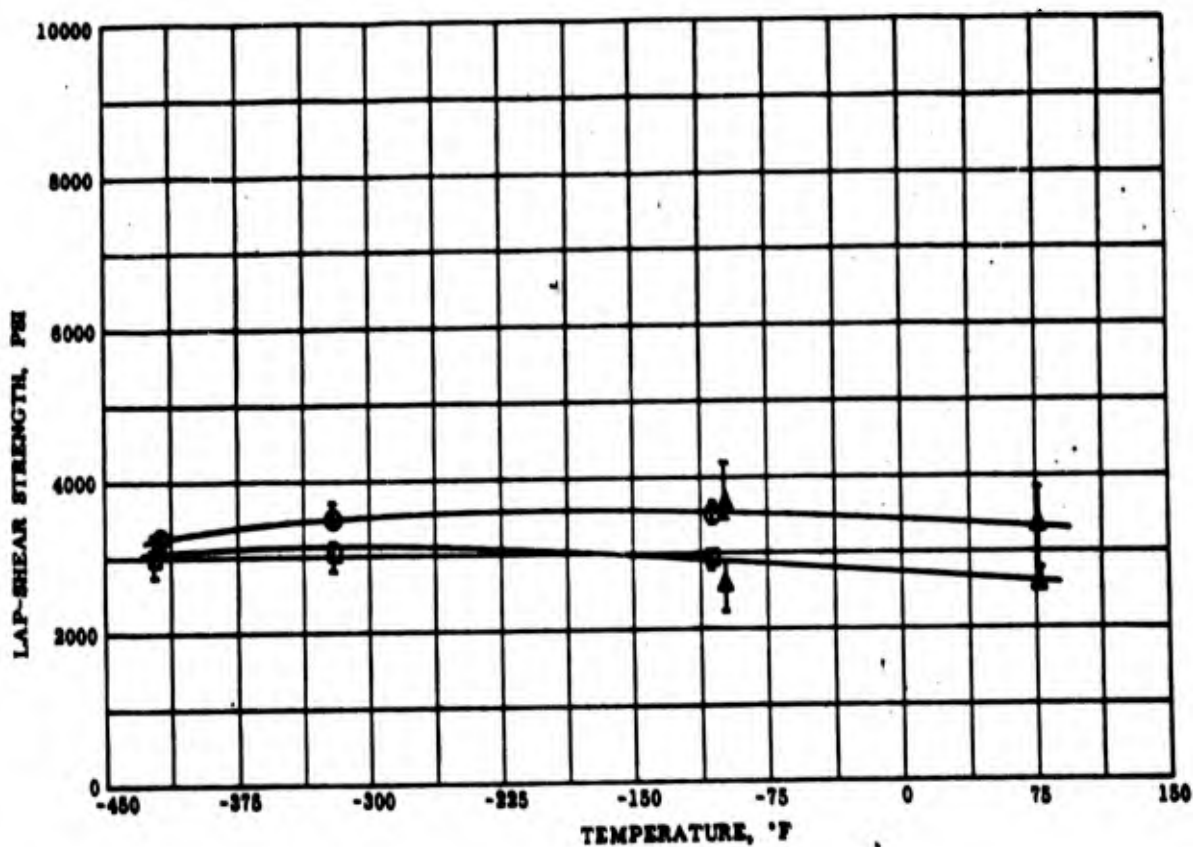


Fig. 13. Lap-Shear Strength of Epoxy-Phenolic Adhesive Bonds vs. Temperature

ADHERENDS: 2024 T-3 ALUMINUM  
 ○ METLBOND 302  
 □ EPOXY ADHESIVE, UNPRIMED (BONDMASTER M611)  
 △ METLBOND MN3C  
 ◊ EPOXY ADHESIVE, PRIMED (BONDMASTER M611/602)  
 ○ EPOXY-POLYAMIDE (EPON 828 VERSAMID 115, 50:50)

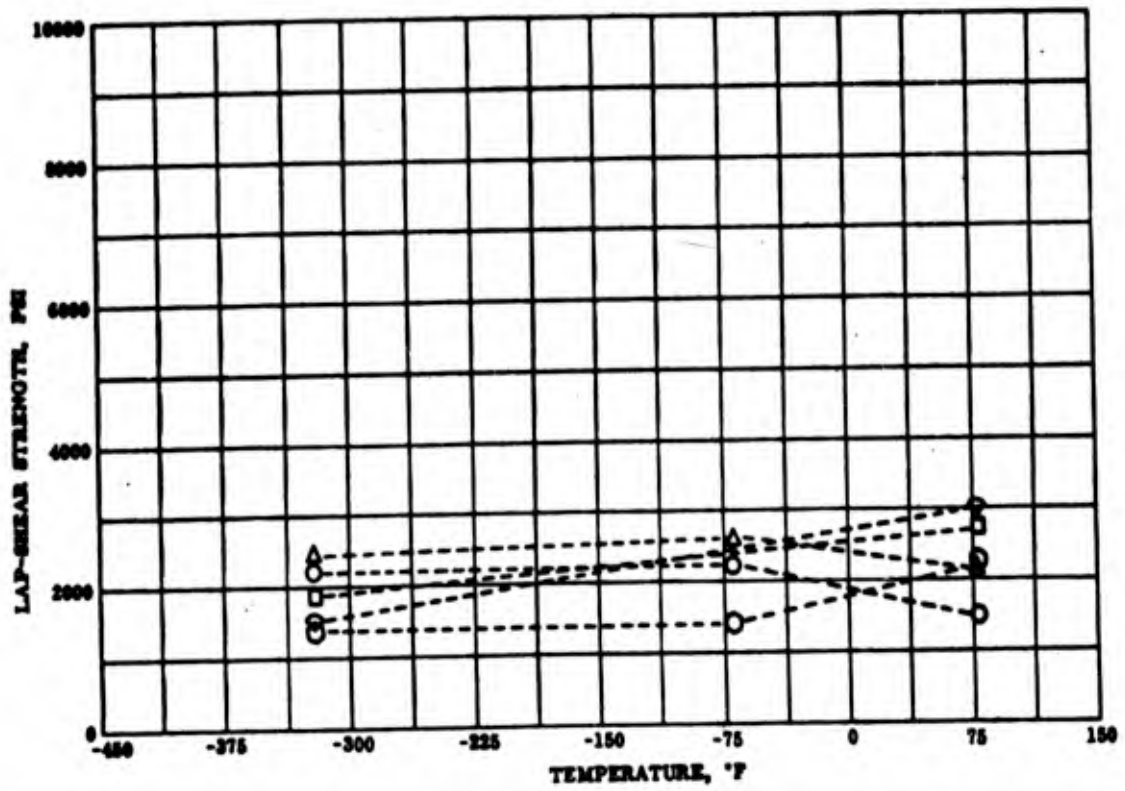


Fig. 14. La-Shear Strength of Miscellaneous Adhesives Reported by North American Aviation Company

ADHERENDS: .063" CLAD 2024 T-3 ALUMINUM  
 ■ FOREST PRODUCTS LABORATORY, 6 SPEC.  
 ▲ NATIONAL BUREAU OF STANDARDS, GRADUAL COOLING, 5 SPEC.  
 ○ NATIONAL BUREAU OF STANDARDS, SHOCK COOLING, 3 SPEC.

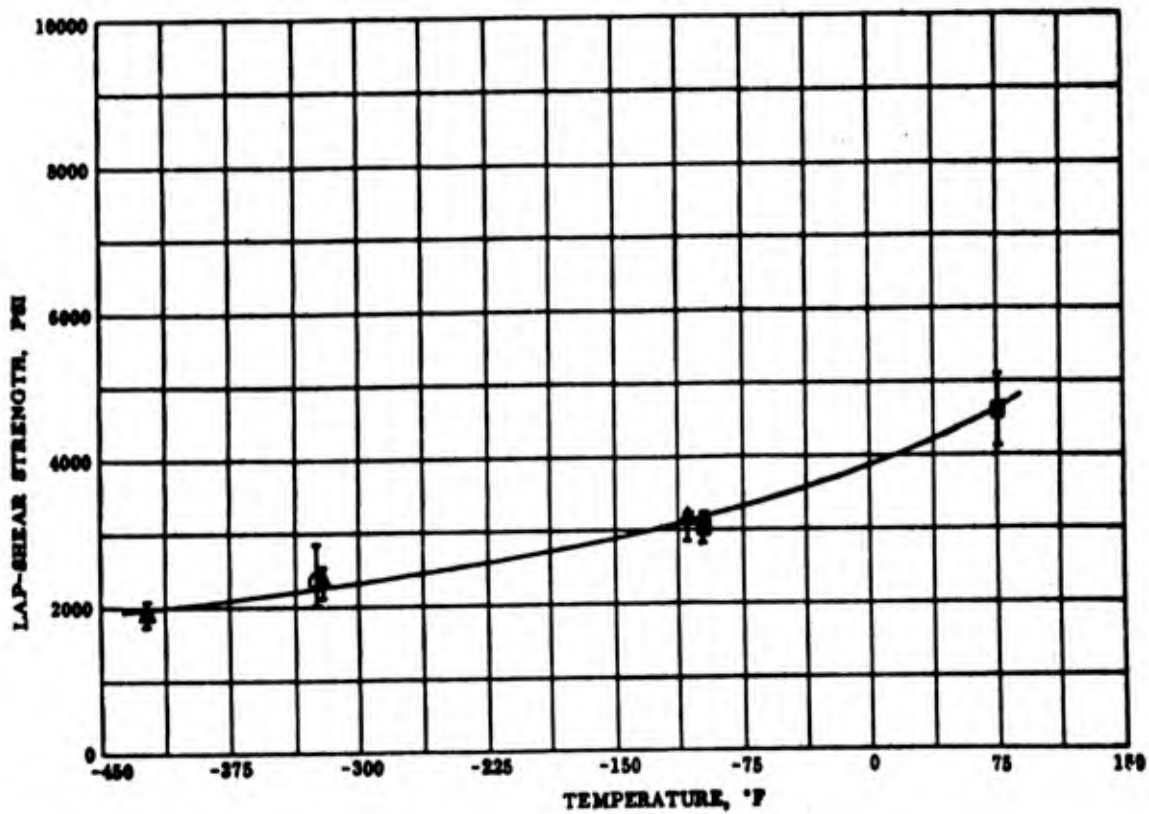


Fig. 15. Lap-Shear Strength of Bloomingdale FM-47 Adhesive Bonds vs. Temperature

ALUMINUM ADHERENDS: CLAD 2024 T-3 ALUMINUM (.063")  
 ▲ FOREST PRODUCTS LABORATORY, 6 SPEC.  
 ○ NATIONAL BUREAU OF STANDARDS, GRADUAL COOLING TESTS, 58 SPEC.  
 □ NATIONAL BUREAU OF STANDARDS, SHOCK COOLING TESTS, 3 SPEC.

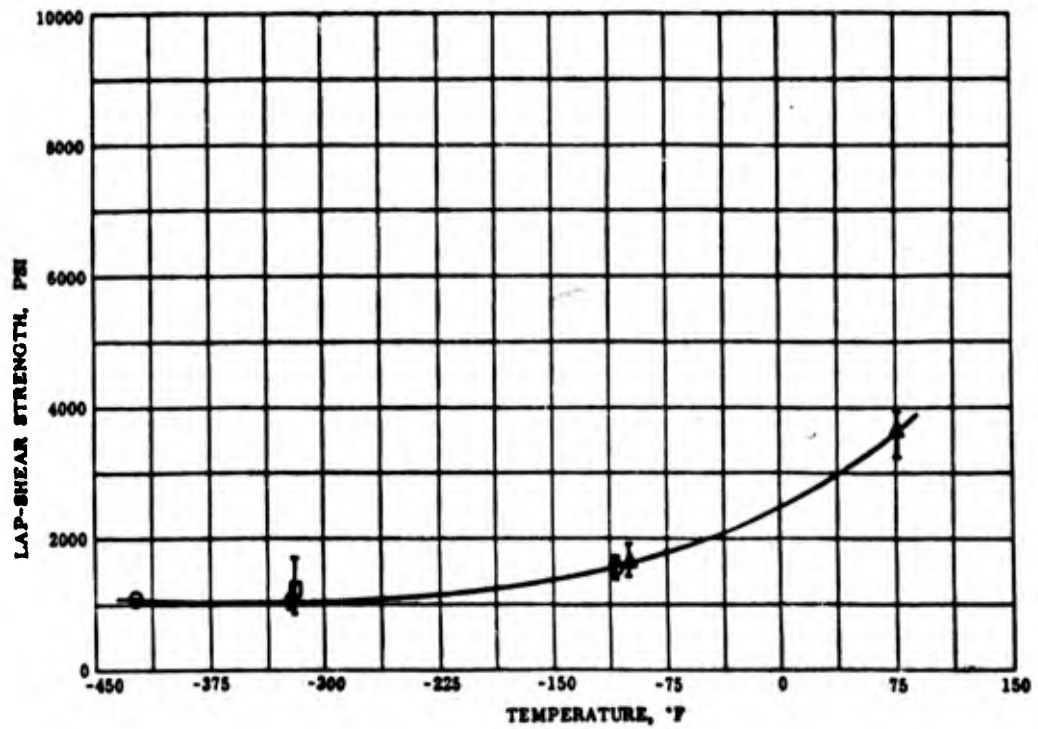


Fig. 16. Lap-Shear Strength of Bondmaster M24B Adhesive Bonds vs. Temperature

ADHERENDS: .063" CLAD 2024 T3 ALUMINUM  
 ▲ FOREST PRODUCTS LABORATORY, 6 SPEC.  
 ○ NATIONAL BUREAU OF STANDARDS, GRADUAL COOLING, 8 SPEC.  
 □ NATIONAL BUREAU OF STANDARDS, SHOCK COOLING, 3 SPEC.

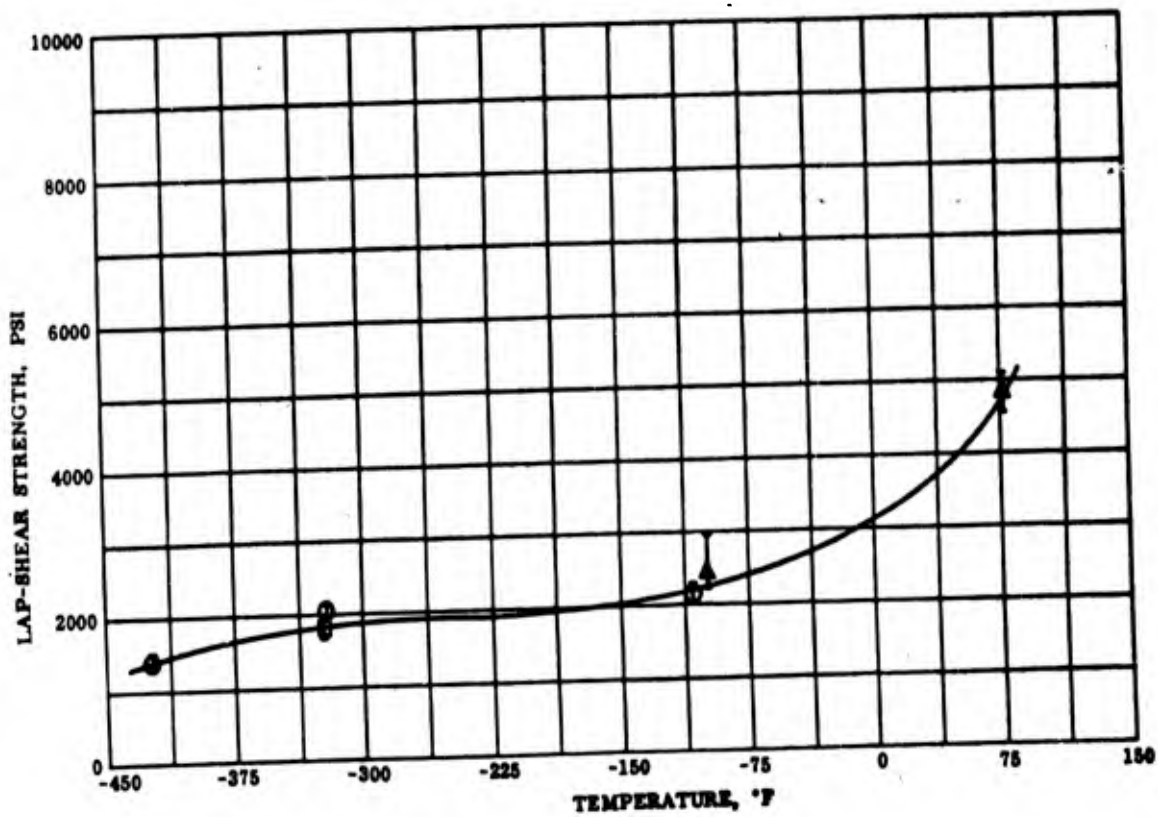


Fig. 17. Lap-Shear Strength of Swedlow 371-W Adhesive Bonds vs. Temperature

ADHREND 1007 CLAD 2024 T-3 ALUMINUM  
 ■ FOREST PRODUCTS LABORATORY 6 SPEC.  
 ▲ NATIONAL BUREAU OF STANDARDS GRADUAL COOLING 7 SPEC.  
 UNLESS OTHERWISE NOTED.  
 ○ NATIONAL BUREAU OF STANDARDS SHOCK COOLING 8 SPEC.

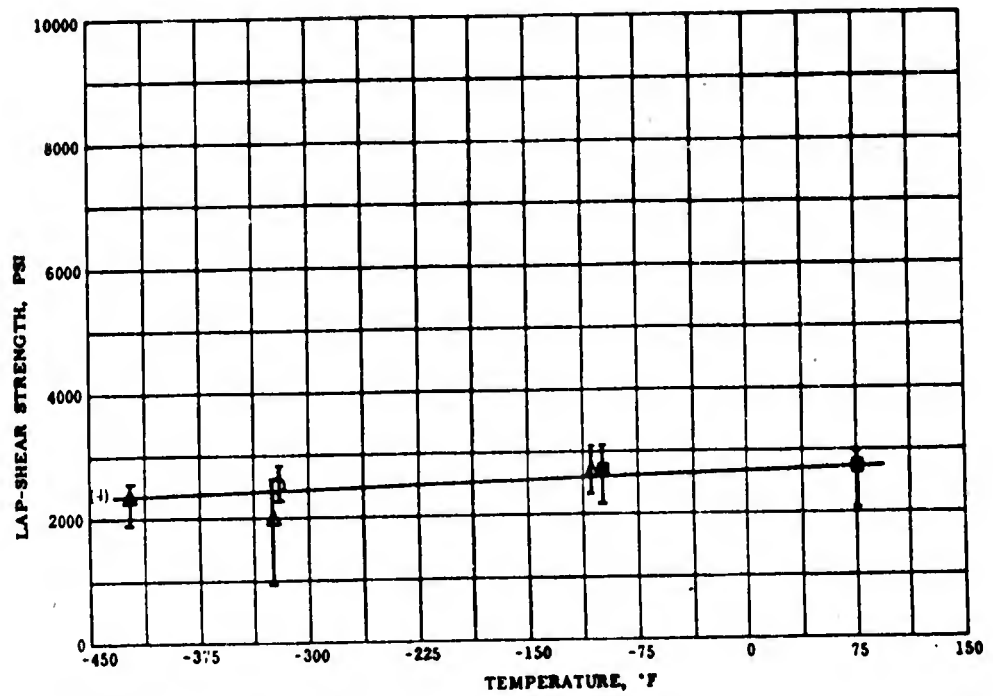


Fig. 18. Lap-Shear Strength of "3M" EC-1469 Adhesive Bonds vs. Temperature

ADHERENDS: .063" CLAD 2024 T-3 ALUMINUM  
 ■ FOREST PRODUCTS LABORATORY, 6 SPEC.  
 △ NATIONAL BUREAU OF STANDARDS, GRADUAL COOLING, 6 SPEC.,  
 UNLESS OTHERWISE SPECIFIED.  
 ○ NATIONAL BUREAU OF STANDARDS, SHOCK COOLING, 3 SPEC.

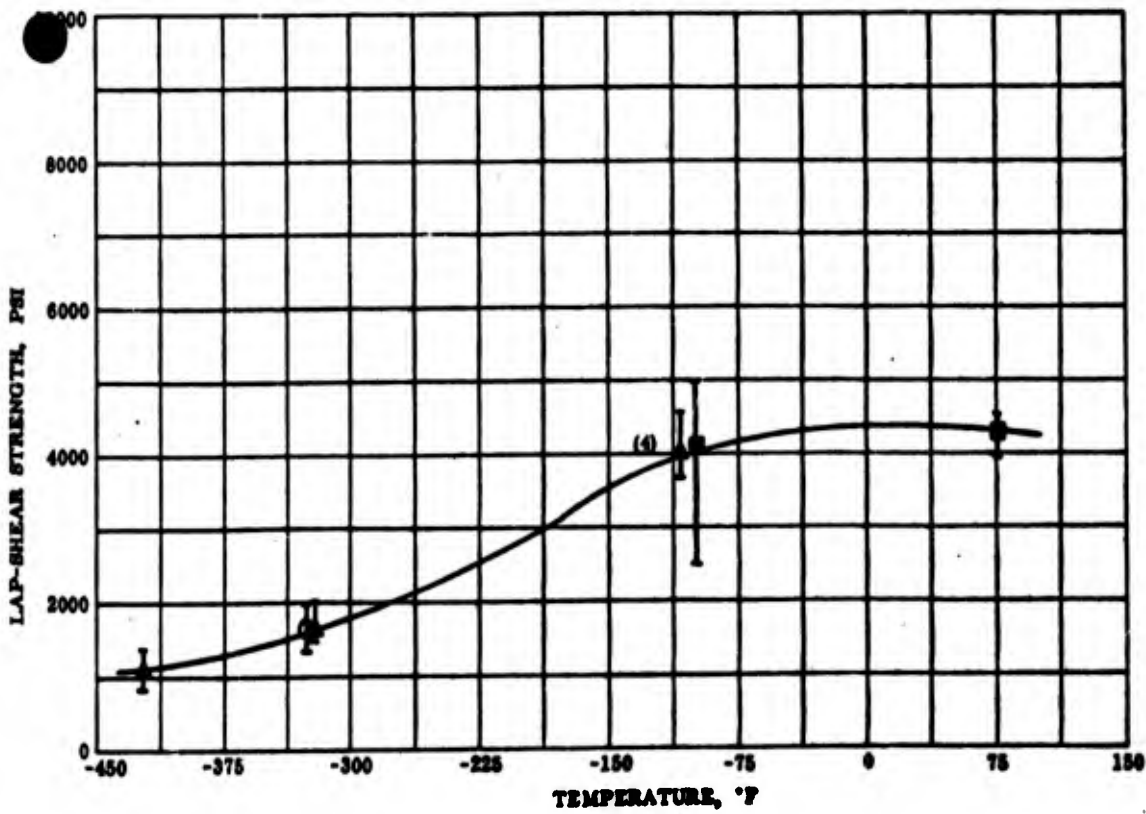


Fig. 19. Lap-Shear Strength of Metlbond 4021 Adhesive Bonds vs. Temperature

ADHERENDS: .063" CLAD 2024 T-3 ALUMINUM

- EC-1471, FOREST PRODUCTS LABORATORY, 2 SPEC.
- ▲ EC-1471, NBS, GRADUAL COOLING, 5 SPEC.
- ▲ AF-5930, FOREST PRODUCTS LABORATORY, 2 SPEC.
- ◇ AF-5930, NBS, GRADUAL COOLING, 5 SPEC.
- REDUX 775, FOREST PRODUCTS LABORATORY, 2 SPEC.
- REDUX 775, NBS, GRADUAL COOLING, 5 SPEC.

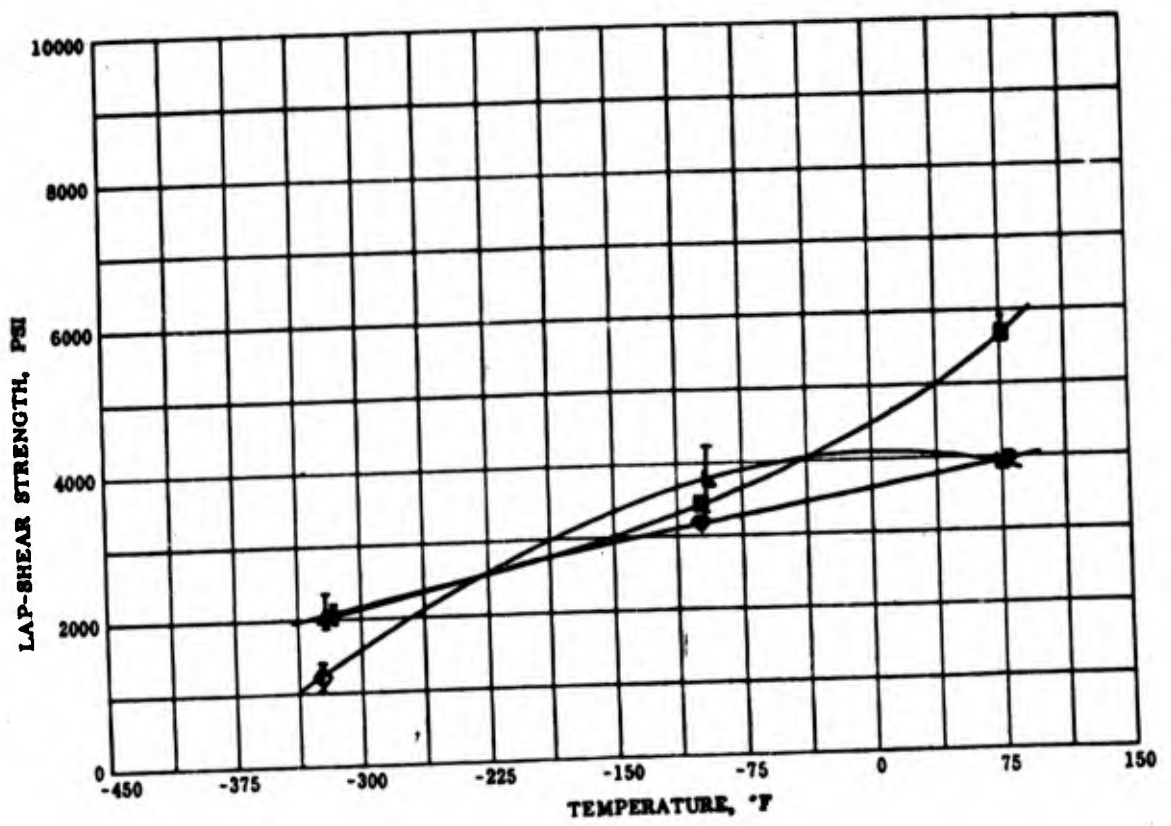


Fig. 20. Lap-Shear Strength of Miscellaneous Adhesive Bonds vs. Temperature

ADHERENDS: COMMERCIAL ELECTROLYTIC TROUGH PITCH COPPER  
ADHESIVE: EPOXY RESIN PASTE CONTAINING APPROXIMATELY 80%  
FILLER (PREDOMINANTLY ALUMINA AND ASBESTOS)  
AND CURED WITH DIETHYLAMINOPROPYLAMINE

- NATIONAL BUREAU OF STANDARDS, TENSILE SPECIMENS
- NATIONAL BUREAU OF STANDARDS, SHEAR SPECIMENS

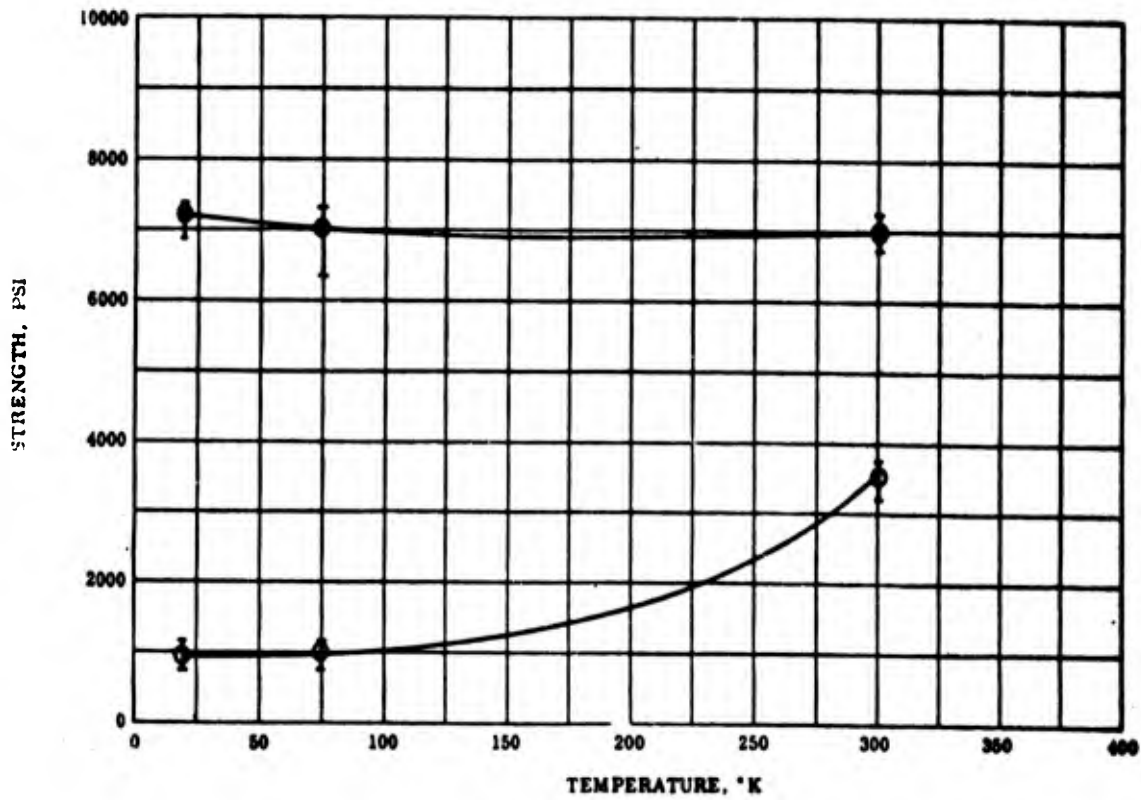


Fig. 21. Strength of a Filled Epoxy Adhesive Bond vs. Temperature

ADHERENDS: COMMERCIAL ELECTROLYTIC TOUGH PITCH COPPER.  
 ADHESIVE: EPOXY RESIN PASTE CONTAINING APPROXIMATELY 55%  
 FILLER (PREDOMINANTLY ALUMINA AND ASBESTOS)  
 AND CURED WITH DIETHYLAMINOPROPYLAMINE

- NATIONAL BUREAU OF STANDARDS, TENSILE SPECIMENS
- NATIONAL BUREAU OF STANDARDS, SHEAR SPECIMENS

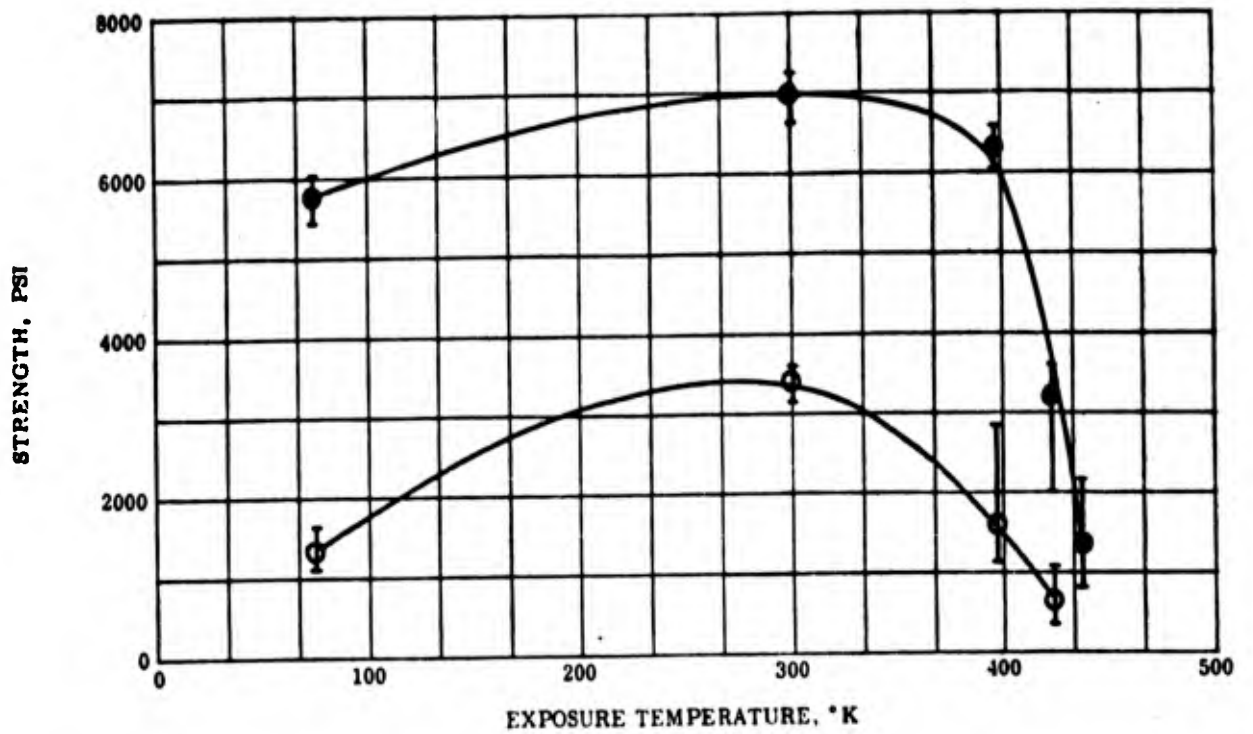


Fig. 22. Strength of Filled Epoxy Adhesive Bond vs. Temperature Prior to Testing at Room Temperature

ADHERENDS: TEFLON AND 52100 STEEL  
TEMPERATURE: -320° F

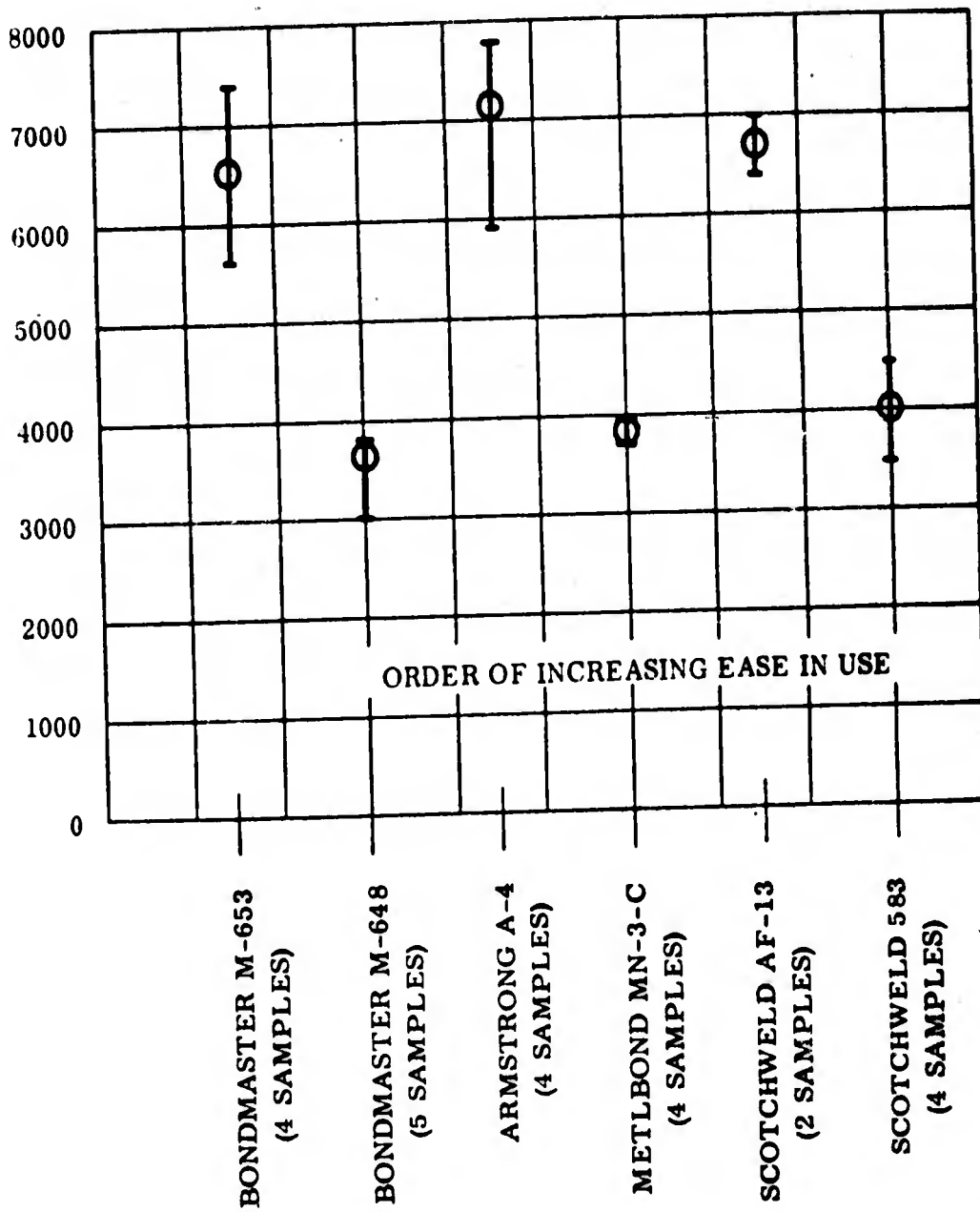


Fig. 23. Results of Adhesive Optimization Tests Conducted at Denver Research Institute

ADHERENDS: TEFLON AND 52100 STEEL  
ADHESIVE: SCOTCHWELD AF-13  
TEMPERATURE: -320° F

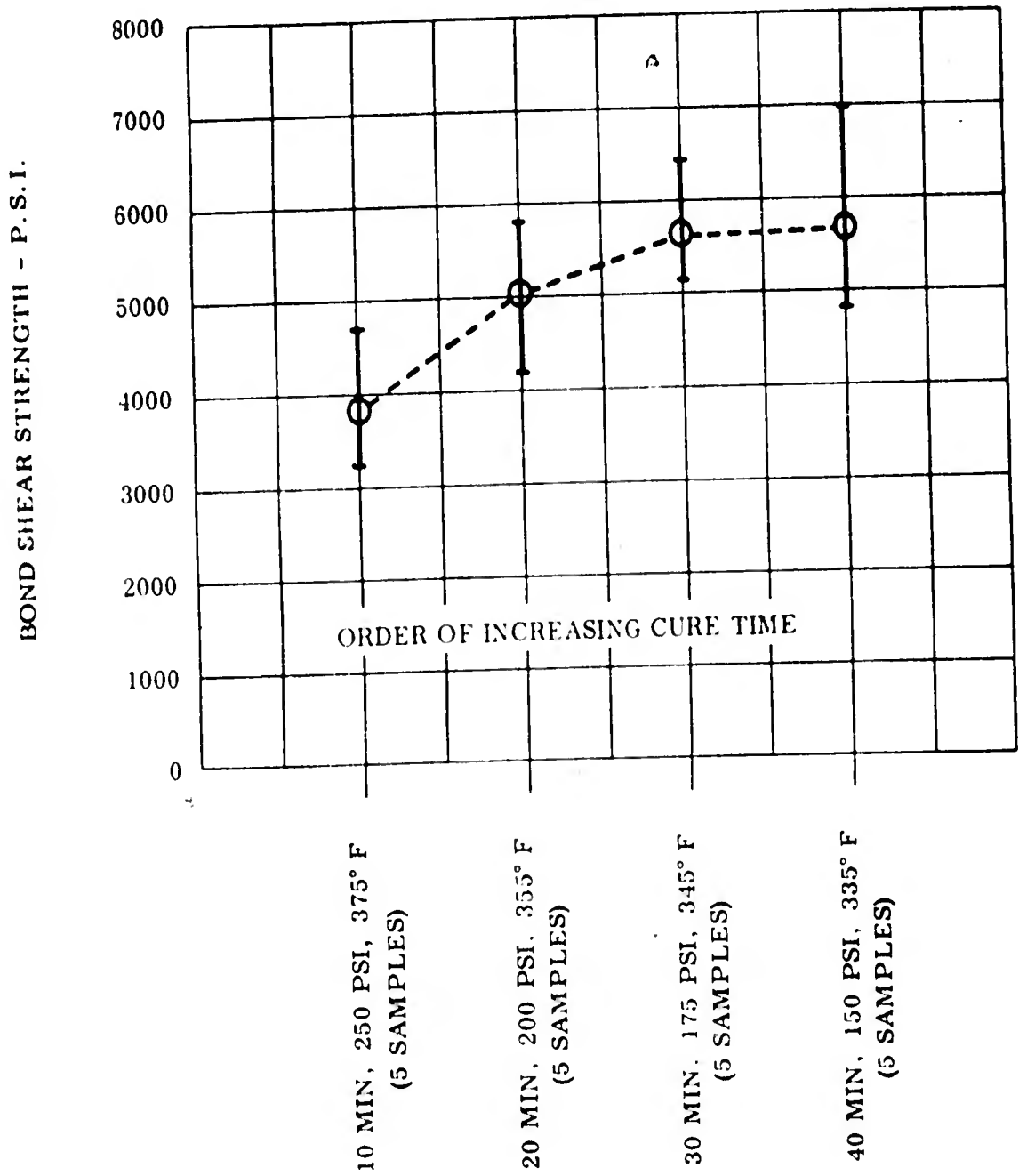


Fig. 24. Results of Cure Cycle Optimization Tests Conducted at Denver Research Institute

ADHERENDS: TEFLON AND 52100 STEEL  
ADHESIVE: SCOTCHWELD AF-13  
TEMPERATURE: -320°F

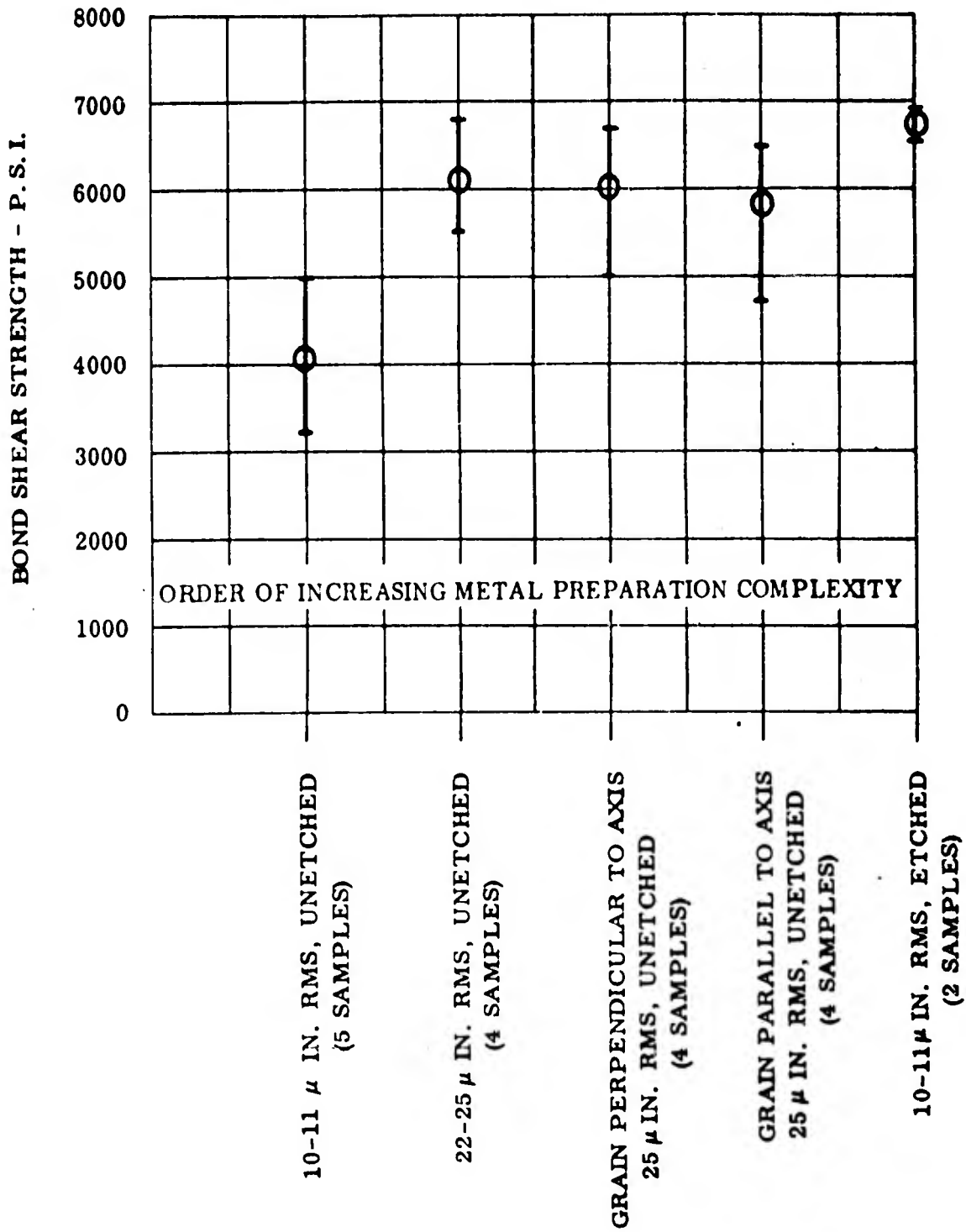


Fig. 25. Results of Metal Surface Preparation Tests Conducted at Denver Research Institute

ADHERENDS: TEFLON AND 52100 STEEL  
ADHESIVE: SCOTCHWELD AF-13  
TEMPERATURE: -320° F

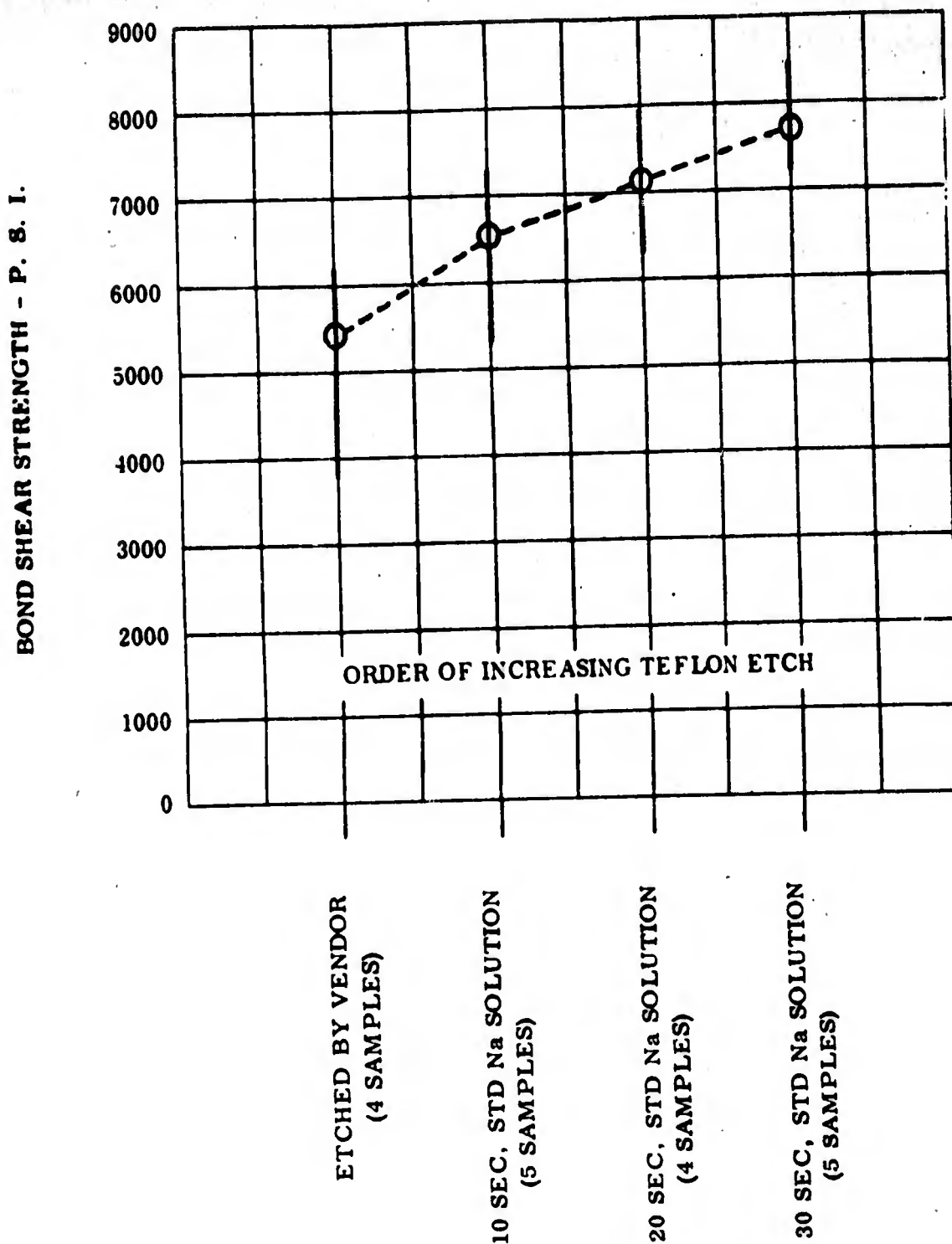


Fig. 26. Results of Teflon Etch Optimization Tests Conducted at Denver Research Institute.



Figure 27. Specimens of 0.020" A-110-AT Titanium Bonded With AF-40 Primed Adhesive System Before Pulling and After Being Pulled at 78° F, -100° F and -320° F.

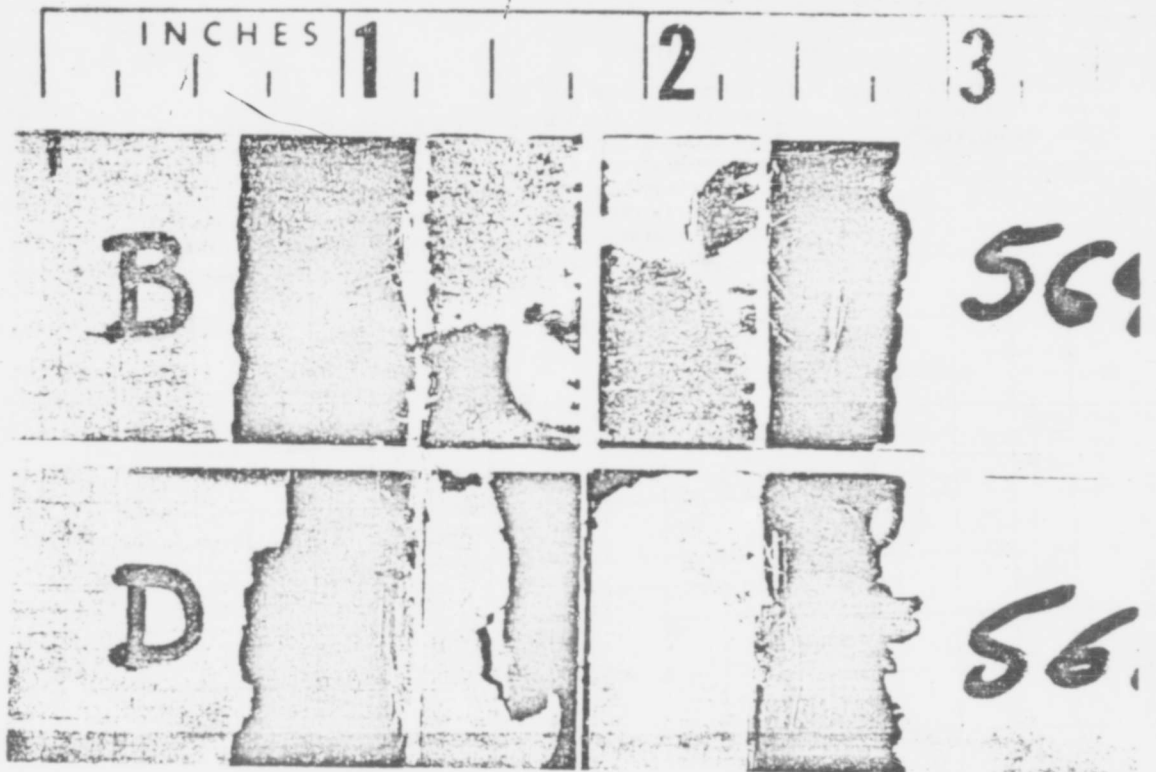


Figure 28. Cohesion and Adhesion Failure of Adhesive Bonds After Being Pulled at 78° F and -320° F.

NOTE: ALL POINTS ARE AVERAGES  
OF FOUR VALUES UNLESS  
OTHERWISE NOTED.

ADHERENDS: .020" EFH 301 CRES

- NARMCO MATERIALS DIVISION, 1/2" OVERLAPS, PHOSPHATE ETCH, 2 SPEC.
- NARMCO MATERIALS DIVISION, 1/2" OVERLAPS, CVA ETCH
- △ NARMCO MATERIALS DIVISION, 3/4" OVERLAPS, PHOSPHATE ETCH
- ◇ NARMCO MATERIALS DIVISION, 3/4" OVERLAPS, CVA ETCH
- CONVAIR-ASTRONAUTICS, 1/2" OVERLAPS, CVA ETCH, 5 SPEC.
- ▲ CONVAIR-ASTRONAUTICS, 3/4" OVERLAPS, PHOSPHATE ETCH

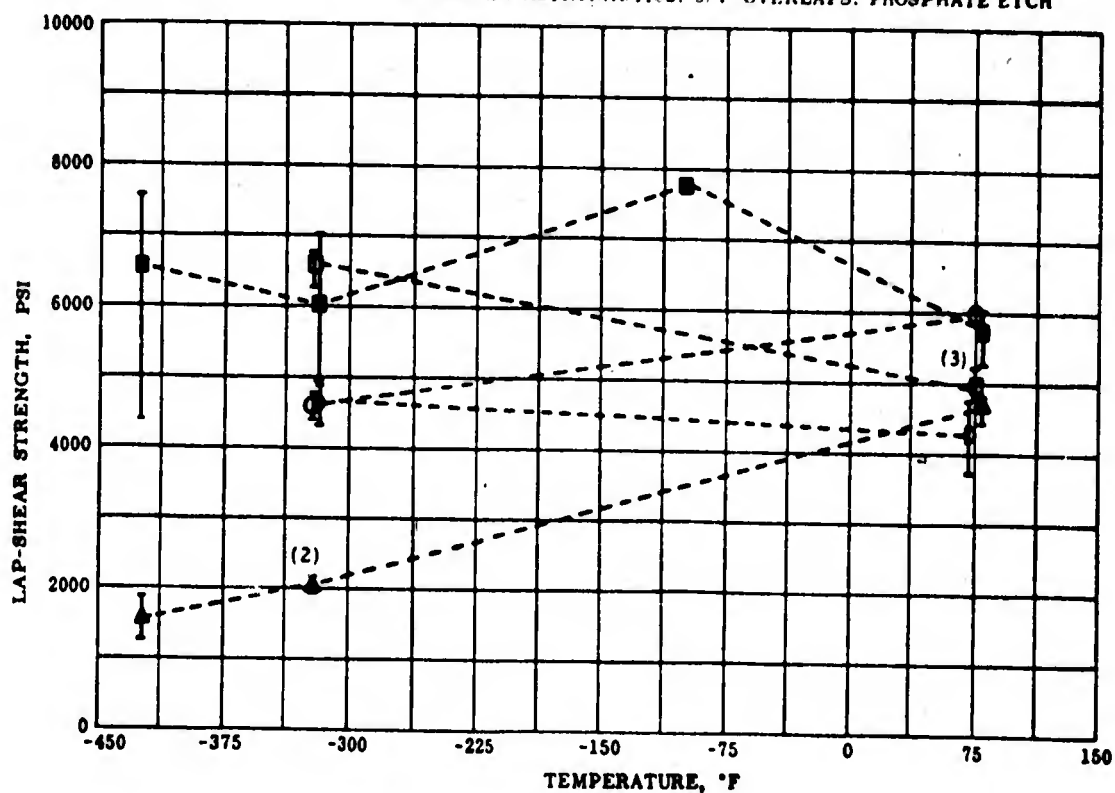


Fig. 29. Effect of Surface Preparation and Overlap Length on Lap-Shear Strength of Metlbond 406

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