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Report No.
D180-24744-1

FINAL REPORT
Environmental Exposure on Thermoplastic Adhesives
Contract N00019-77-C-0340

By

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Prepared For
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FOREWARD

This report summarizes the work performed by the Boeing Aerospace Company during the period of 20 May 1977 to 20 April 1978 for Naval Air Systems Command, United States Department of the Navy under Contract N00019-77-C-0340, entitled "Environmental Exposure Study on Thermoplastic Adhesives". Mr. J. J. Gurtowski (AIR 52032C) was the Program Monitor.

This program was conducted by the Boeing Military Airplane Development Organization of the Boeing Aerospace Company, Seattle, Washington; Mr. John Hoggatt was Program Manager and Mr. Sylvester Hill was principal investigator.

Messrs E. House, R. Hodges, W. Dumars, and Ms J. Jaquist and V. Monroe were major contributors to the program.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES (CONT.)

LIST OF TABLES

1.0 INTRODUCTION AND SUMMARY

In aerospace applications, the predominance of adhesive bonding is performed with epoxy based adhesives because of their demonstrated capabilities. However, there are several unfavorable aspects of bonding with thermosets such as epoxies, that can be avoided by the use of thermoplastic adhesives. For example, thermoplastic adhesives, unlike epoxies, have infinite shelf life, are inexpensive, absorb less moisture than epoxies, and permit post forming of bonded parts. Recognizing these desirable attributes, previous NAVAIR programs (Ref. 1 and 2) were conducted which showed that thermoplastic adhesives offer the integrity and endurance required of a structural adhesive. Processing parameters were established, surface preparations defined, and a limited amount of mechanical properties data obtained. The purpose of the program reported on herein, was to establish basic mechanical properties and determine the environmental resistance of two thermoplastic polymer systems, RADEL 5000 and PKXA manufactured by Union Carbide Company. Both polymers were evaluated as adhesives on stainless steel and titanium adherends while the PKXA system was also evaluated using fiberglass reinforced polyimide composite adherends.

Task I of this program was devoted to selecting the adhesives, primers, surface preparation processes, and to performing environmental exposure studies. Both polymers selected, RADEL 5000 and PKXA are modified polysulfones. To minimize the risk of adhesive primer degradation during the 600°F + processing, a polyimide adhesive primer (BR-34 by American Cyanamid) was applied to the adherends and then overcoated with a thin coat of RADEL 5000 or PKXA primer depending on which adhesive film was used in the joint. Surface preparation consisted of chromic acid anodizing for titanium, sulfuric acid anodizing for stainless steel, and vacuum blast plus solvent wipe for the polyimide composite adherends. Selection of adhesive primers and surface preparation treatments was based on room temperature and 300°F lap shear tests as well as crack extension determinations after condensing humidity (120°F) exposure of 200 hours.

To ascertain the thermal stability and environmental resistance of the selected adhesive systems, specimens were isothermally aged at elevated temperatures and other specimens were exposed to fluids commonly used in aircraft operations. After exposures ranging from 7 to 90 days duration, the following factors were assessed:

- Effects of fluid exposure on lap-shear strength retention at -65, +70, +180, +300°F.
- Effects of prolonged thermal exposure on lap-shear strength retention at -65, +70, +180, +300°F.
- Effects of prolonged fluid and thermal exposure on adhesive bondline crack propagation at +70°F.

While some of the exposures caused a reduction in properties, both the RADEL 5000 and PKXA are considered to have adequate environmental resistance except that RADEL 5000 is degraded when exposed to MIL-H-83306 hydraulic fluid.

The objective of Task II of this program was to determine basic mechanical properties of the adhesive systems selected in Task I, on stainless steel and titanium adherends. The properties evaluated were metal-to-metal climbing drum peel strength, creep, and fatigue life. Peel strengths were in the range of 30-40 pounds per inch width at 70 and 250°F which is adequate for structural bonding. The static creep characteristics of the RADEL 5000 and PKXA were determined at 75 and 250°F under various loads up to 80% of ultimate. Some of these conditions were purposely made more severe than those required in the applicable military specifications to better match anticipated applications. Both RADEL 5000 and PKXA exhibited good creep resistance at relatively high stress levels (50-70% of ultimate). Tension-tention (R=.1) fatigue life determinations were conducted at 70°F and load levels ranging from 30 to 80% of ultimate. It is considered that adequate fatigue resistance was exhibited under these conditions by both the RADEL 5000 and PKXA adhesives.

In Task III, the data from Tasks I and II was analyzed and an assessment made regarding the suitability of the adhesive systems for use in aerospace applications. The data indicates that the PKXA adhesive is suitable for structural aerospace applications. The RADEL 5000 is also suitable for structural aerospace applications, but should not be used in applications where prolonged exposure to phosphate ester type hydraulic fluids (MIL-H-83306) is a possibility. Also, the large scatter in mechanical properties test data, indicates that improvements in the adhesive primer system should be made.

The final task (IV) in this program consisted of preparing interim technical reports and a final report.

2.0 STUDY PROGRAM

The objectives of this program were:

(1) to determine the environmental resistance and thermal stability of two thermoplastic adhesives on titanium, stainless steel and composite adherends,

(2) to evaluate selected basic mechanical properties of the adhesives, and

(3) to assess the suitability of the thermoplastic adhesive systems for use in structural aerospace applications.

The program was divided into three principal areas: Task I - Environmental Resistance Evaluations; Task II - Mechanical Properties Determination; and Task III - Data Assessment.

2.1 Task I - Environmental Exposure

2.1.1 Preliminary Investigation

Under previous contracts (References 1 and 2) in which thermoplastic adhesives were evaluated, the adherends were primed with an epoxy corrosion inhibitor primer (BR-127) from American Cyanamide to increase the wettability of the faying surfaces. Due to concern about the stability of the epoxy primer when bonding the thermoplastic adhesives at high temperatures (650 - 750^{0}F), BR-34 polyimide primer was selected for evaluating the PKXA and R-5000 thermoplastic adhesives on this program. The BR-34 was selected because it has been commercially available for a number of years and a large data base has been developed on it throughout the aerospace industry.

2.1.1.1 Adhesive Selection

The P-1700 and PKXA polysulfone resins were evaluated as adhesives under previous Navy contracts (References 1 and 2). PKXA adhesively bonded joints were resistant to most of the common laboratory solvents and shear strengths equivalent to epoxy adhesives were obtained when lap shear specimens were tested after exposure to different environments. Based upon this data, the PKXA resin was selected for further evaluation.

RADEL 5000 polyphenylsulfone was selected as the second resin because of its potential high usage temperature and resistance to chlorinated solvents.

2.1.1.2 Primer Selection

Adhesive primer selection was conducted on specimens that had been surface treated using the methods outlined in Section 2.1.1.3. Three primers were evaluated: a 5% solution of the bonding resin R-5000 in dimethylformamide (DMF) or PKXA in methylene chloride (CH_2Cl_2), BR-34 polyimide, and BR-34 coated with a dilute solution of the bonding resin. Test results from these bonded panels showed that all of the primers were adequate but scatter in shear data was less when the panels were primed with BR-34 and subsequently overcoated with a thin coat of the bonding resin. See Tables I and II for a summary of the test data.

After carefully analyzing the data data, failed specimens, and data scatter, the BR-34 primer coated with a dilute solution of the bonding resin was selected as the primer system for this program. Primer application was performed in accordance with the following procedure:

> The BR-34 primer was sprayed on the adherends, air dried 2 hours and then forced air dried in an oven at $350^{\circ}F$ for one hour. The rate of rise in oven temperature from RT to $350^{\circ}F$ was $3-5^{\circ}F/min$. The dilute solution of the resins (PKXA or R-5000) were brushed on the adherends or over the BR-34 as the priming method diluted and dried in an oven at $250^{\circ}F$ for one hour using the $3-5^{\circ}F$/per minute rate of rise.

2.1.1.3 Surface Preparation

The comparative test methods used to evaluate the candidate bonding surface preparations were the crack extension and standard lap shear tests (Fig. 1 and 2). The crack extension method was chosen because of its sensitivity to surface conditions. The standard lap shear test was selected because the test data obtained are easily compared to available shear data of other adhesive systems.

Surface preparation methods evaluated for steel, titanium, and composites were:

- Steel (17-7PH)
  - Sulfuric Acid Anodize
- Titanium (6Al-4V)
  - Chromic Acid Anodize
  - Pasa Gel
  - Phosphate Fluoride

Composite (Polyimide/Glass)
- Scotchbrite + Solvent
- Vacuum Blast + Solvent
- Peel Ply

After priming, bonding and evaluating the failed specimens and test data, chromic acid anodize, sulfuric acid anodize and vacuum blast plus solvent wipe were selected as the surface preparation methods for processing titanium, steel and composite adherends respectively for the remainder of the program. The adherends were primed using the priming methods outlined in section 2.1.1.2.

2.1.1.4 Adhesive Film Preparation

The PKXA and R-5000 adhesive films were fabricated by coating glass fabric (112 E-glass/A-1100) stretched over an aluminum frame (Figure 3) with a 20-25% solution of PKXA/CH_2Cl_2 or R-5000/DMF until the film was 8-12 mils thick. After achieving the desired thickness, the film was dried at 210-215^{O}F for PKXA and 310-315^{O}F for R-5000 until it reached constant weight. This drying cycle was to remove all traces of the solvent.

2.1.1.5 Specimen Fabrication and Testing

Panel Fabrication

Titanium and steel crack extension panels were fabricated from .050" to 6.0" x 6.0" sheet material. Standard five finger lap shear panels were used for the lap shear specimens.

Standard 4.0" x 6.0 x .1" composite lap shear panels were cut from a large polyimide laminate (Hexcel's F-174-1-7781).

Bonding

The primed adherends were laid up on bonding fixtures with adhesive film cut to allow approximately 1/8 inch excess material around the lap joint. The bonding tool assembly was enveloped bagged and full vacuum was applied on the part. The assemblies were bonded as follows:

R-5000 - Heat from RT - 600^{O}F at 10^{O}/min under full vacuum, apply 50 psi, hold part temperature at 600^{O}F for 15 minutes, cool to 100^{O}F under pressure and vacuum.

PKXA - Heat from RT - 475^{O}F at 3-5^{O}/min under full vacuum and 15 psi, then apply full vacuum and raise temperature to 575^{O}F, hold at 575^{O}F for 30 min. and cool to RT under pressure and vacuum.

Test Results

Titanium and Steel

Lap shear and crack extension specimens were fabricated and tested using each of the primer systems and surface treatments (2.1.1.2 and 2.1.1.3). Structural bonds were achieved with all three of the primer systems. Test results are presented in Tables I through IV and observations pertaining to the adhesive primer and surface preparation evaluations follows.

PKXA Adhesive Bonds

Chromic acid anodized titanium lap shear specimens with BR-34, BR-34 + PKXA, and PKXA primers failed with average lap shear strengths of 3160 and 2140; 4380 and 2680; 3230 and 2390 psi at RT and 300^{O}F, respectively. The failure mode for the RT specimens was 80-95% cohesive within the thermoplastic adhesive film. The failure mode of the 300^{O}F specimens was 95-100% cohesive within the film adhesive. The titanium crack extension specimens with BR-34, BR-34 + PKXA, and PKXA specimens exhibited crack growths of .05, 0, .4 inch after 200 hours in condensing humidity. Specimens with PKXA primer failed adhesively.

Pasa gel/titanium lap shear specimens with BR-34, BR-34 + PKXA, and PKXA primers failed in average lap shear strengths of 1920 and 1705; 3230 and 2390; 5175 and 3808 psi at RT and 300^{O}F, respectively. The failure modes of the specimens with BR-34 primer were 75-80% adhesive at the primer-metal interface. The failure modes of the specimens with BR-34/PKXA and PKXA primers were 80-90% cohesive at the metal-primer interface. The pasa gel/titanium crack extension specimens with the BR-34, BR-34/PKXA, and PKXA primers failed with crack growths of .21, .18, and .32 inch after 200 hours of exposure in condensing humidity. All specimens failed adhesively at the primer-metal interface.

Titanium lap shear specimens with phosphate fluoride surface treatment and BR-34, BR-34/PKXA, and PKXA primers exhibited average lap shear strengths of 2780 and 2105;

3960 and 2745; 2985 and 1900 psi when tested at RT and 300°F, respectively. The failure modes of the RT shear specimens were 80-85% cohesive within the film adhesive, while the 300°F specimens were 90-95% cohesive within the film adhesive. Phosphate fluoride surfaces treated crack extension specimens with BR-34, BR-34/PKXA, and PKXA primers exhibited crack growths of .17, .23, and .28 inch after 200 hours in condensing humidity. The failures were adhesively at the metal-primer interface.

Anodized steel lap shear specimens with BR-34, BR-34/PKXA, and PKXA primers failed at 3310 and 2820; 4345 and 2375; 2575 and 2035 psi at RT and 300°F, respectively. The failure modes were 85-90% cohesive for the BR-34 and BR-34/PKXA primed specimens. The PKXA primed specimens exhibited 70-80% cohesive failure. The anodized steel crack extension specimens exhibited very little crack growth when exposed to humidity environments.

R-5000 Adhesive Bonds

Chromic acid anodized titanium lap shear specimens with BR-34, BR-34 + R-5000 primers failed with average shear strengths of 3325 and 2460; 3685 and 2830; 4380 and 2680 psi at RT and 300°F, respectively. The failure modes for these specimens were 90-95% cohesive within the adhesive film. The anodized titanium crack extension specimens exhibited crack growths of .03, 0, .07 inch after 200 hours in condensing humidity for the BR-34, BR-34/R-5000, R-5000 primer systems, respectively.

Pasa gel surface-treated titanium lap shear specimens with BR-34, BR-34/R-5000, and R-5000 primers failed with average lap shear strengths of 3860 and 2300; 4080 and 2900; 3190 and 2760 psi at RT and 300°F, respectively. The failure modes for these specimens were 90-95% cohesive within the film adhesive. The pasa gel titanium crack extension specimens with BR-34, BR-34/R-5000, and R-5000 exhibited crack growths of .04, .01, .05 inch after humidity exposure.

Titanium lap shear specimens with phosphate fluoride surface treatment and primed with BR-34, BR-34/R-5000 primers failed with average shear strengths of 3780 and 2680; 4120 and 2785; 3365 and 2635 psi when tested at RT and 300°F, respectively. The failure modes of the lap shear specimens were 90-95% cohesive within the adhesive film. Crack extension specimens with phosphate fluoride surface treatment and the three priming systems exhibited crack growths of 0 to .05 inch after 200 hours in humidity.

Anodized steel lap shear specimens with BR-34, BR-34/R-5000, and R-5000 primers failed in shear strengths of 3460 and 2162; 4080 and 3205; 2965 and 2205 psi at RT and 300°F, respectively. BR-34 and R-5000 primed specimens failed 80-85% cohesively within the adhesive. BR-34/R-5000 primed specimens failed 90-95% cohesively. Crack growths of .07, .02, and .07 inch were measured for the anodized steel crack extension specimens. The failure modes were cohesive.

Composites

The composite panels for the fabrication of the lap shear specimens were fabricated from Hexcel's F-174-1-7781 (BMS 8-144) polyimide prepreg. The prepreg is 181 style fiberglass impregnated with a B-staged, fire-resistant, thermosetting polyimide resin which has good serviceability at 450°F. This polyimide was chosen because of its in-house specification coverage and availability.

During this investigation for surface preparation, three methods were selected and evaluated for the polyimide composite panels. These methods were Scotchbrite followed by solvent wipe, vacuum blast followed by solvent wipe, and the use of Style 71789 peel ply from Burlington Industries.

The surface-treated polyimide composite panels were primed with BR-34 primer and cured for 2 hours at 350°F. A thin coat of PKXA or R-5000 polymer solution (5%) was brushed over the polyimide primer, air dried for a minimum of two hours, and oven dried for one hour at 250°F. Lap shear assemblies were fabricated from the primed panels and the film adhesives using the established bonding cycles (2.1.1.5) for the adhesives. The bonded assemblies were cut into lap shear specimens and tested. The test results show that the best surface preparation method for bonding the polyimide composite with PKXA and R-5000 adhesives was vacuum blast followed by solvent wipe. These data are reported in Tables I and II for the PKXA and R-5000 adhesives, respectively. The vacuum blast followed by solvent wipe was selected as the surface treatment for the polyimide composites.

2.1.2 Environmental Stability

This portion of the program was designed to determine the long-term stability of the thermoplastic adhesive systems under adverse environments. These environments included thermal aging at 180 and 300°F as well as room temperature immersion in selected fluids likely to be encountered in aircraft flight/maintenance operations. The environmental aging solutions selected were:

(1) Deicing fluid (#146 ethylene glycol base)
(2) JP-4 fuel
(3) Hydraulic fluids
    MIL-H-83306 (phosphate ester type)
    MIL-H-5606 (standard petroleum base)
(4) Synthetic lubricant (MIL-L-7808)
(5) Salt water (5% NaCl)
(6) Caustic (Ph9)
(7) Cleaning solvent, organic (Stoddard)
(8) Naptha
(9) AMLgard MIL-C-85054 corrosion inhibitor

The solutions for the environmental test program were selected in cooperation with NAVAIR Project Monitor. AMLgard (MIL-C-85054) was included because of its wide use in aircraft systems for corrosion inhibiting. Naphtha was selected because it is an excellent solvent which is used frequently for removal of MIL-C-85404-type materials. The MIL-H-5606 hydraulic fluid was chosen to compare its effect on bonded specimens with the phosphate ester-type hydraulic fluids (MIL-H-83306). The remaining solutions are commonly encountered during an aircrafts service life.

Both unstressed lap shear specimens (Fig. 2) and stressed crack extension specimens (Fig. 1) were exposed in the selected environments for periods ranging from seven to ninety days. Crack growth was measured after the exposure to assess effect of the exposure on the stressed bond joint.

After the lap shear specimens had been exposed, they were tested at -65, RT, 180 and 300°F for residual strength. The residual strength was then compared to that of unexposed control specimens for determining strength loss due to the exposure.

The adhesive, primer and surface preparations evaluated were those selected during the preliminary investigation portion of this program. They included: PKXA adhesive film on BR-34/PKXA primed titanium, stainless steel and polyimide/fiberglass composite adherends; and, RADEL 5000 adhesive film on BR-34/RADEL 5000 primed titanium and stainless steel specimens.

The exposure test matrix is shown in Table V and effects of the environmental exposures are discussed in 2.1.2.1 through 2.1.2.4. Test results are presented in the Appendix (Tables A-1 through A-5).

2.1.2.1 Thermal Stability

Unstressed lap shear specimens were thermally aged at 180°F and 300°F for periods of 28 days and 90 days. All five adhesive/adherend combinations were exposed; PKXA/Composite, PKXA/steel, PKXA/titanium, R-5000/steel, and R-5000/titanium. The percentage reduction in strength at -65, RT, 180 and 300°F after exposure is shown in Table VI while the control and residual strengths obtained are plotted in Figures 4 through 8. The only bond joints consistently showing reduced properties after thermal aging were the PKXA/composite bonds.

Excluding the PKXA/composite specimens and considering the number of specimens tested, the thermal aging did not appreciably affect the elevated temperature (180 and 300°F) properties of either PKXA or R-5000. The same findings hold for the -65°F and RT properties except for a 25% reduction in the PKXA/Ti bond strength at -65°F after 90 days at 300°F and a 27% reduction in the R-5000/Ti bond strength at RT after aging 28 days at 300°F. A study of the data indicates that these strength reductions do not fit any particular trend.

Crack extension specimens were also exposed at 180 and 300°F for 28 and 90 days as shown in Table V. The only crack growth (0.03") occured in a PKXA/steel specimen after 90 days at 300°F. From these results, it is apparent that the thermal aging had no effect on the PKXA or R-5000 steel or titanium bonds as measured by crack extension specimens. Crack extension data is presented in Tables A-1 through A-4 in the Appendix.

2.1.2.2 Hydraulic Fluids and Lubricant Exposure

Lap shear and crack extension specimens from all five adherend/adhesive combinations were exposed to MIL-H-83306 (phosphate ester type hydraulic fluid), MIL-H-5606 (petroleum base hydraulic fluid), and MIL-L-7808 (synthetic lubricant) for different aging periods. These periods range from 7 and 28 days for the petroleum base hydraulic fluid to 90 days for the phosphate ester type hydraulic fluid and the synthetic lubricant.

MIL-H-83306

Unstressed lap shear specimens and stressed crack extension specimens were aged for 90 days in MIL-H-83306. Test results show that the phosphate ester type hydraulic fluid had a detrimental effect on the R-5000 bonded titanium and steel specimens. The reduction in shear strength for the steel bonded specimens ranged from 78% at RT to 96% at 300°F. The range for the titanium bonded specimens was in the same order of magnitude. See Figures 9 and 10 for a graphical presentation of these data.

Lap shear specimens bonded with PKXA showed a decrease in shear strength after environmental exposure. The strength reductions of the steel specimens were 7% at 300 to 16% at RT. Titanium bonded specimens displayed a change in shear strength from +14% at RT to -22% at 300°F. These data are provided in Figures 11 and 12.

Composite lap shear specimens which were bonded with PKXA and exposed in MIL-H-83306 for 90 days exhibited a 19% reduction in RT shear strength and a 5% reduction in shear strength at 300°F. These specimens failed in the polyimide/glass laminates rather than in the adhesive and may not represent a true reduction in shear strength due to the environment. The composite data are presented graphically in Figure 13.

The crack extension specimens which were bonded with R-5000 and PKXA adhesives were exposed in MIL-H-83306 for 90 days. At the completion of the 90 day exposure, the R-5000 bonded titanium and steel specimens had delaminated while the PKXA bonded steel and titanium specimens exhibited crack growths of .03" and .24", respectively.

MIL-H-5606

The unstressed R-5000/steel and titanium lap shear specimens showed a decrease in lap shear strength after environmental exposure in MIL-H-5606 for 7 and 28 days. The room temperature steel lap shear strengths were reduced by 14% and 21% after 7 and 28 days in solution while the titanium specimens showed strength reductions of 18% and 28% under the same conditions. Steel lap shear specimens exposed in the fluid environment and tested at 300°F showed strength losses of 31% and 20% for 7 and 28 day exposure, respectively. A 25% and 31% reduction in shear strength was noted for titanium specimens that were aged in the same environment and tested after 7 and 28 days.

The unstressed steel lap shear specimens that were bonded with PKXA and exposed to MIL-H-5606 solution for 7 and 28 days showed an increase in shear strength when tested at RT and a 6% to 8% loss in shear strength when tested at 300°F. PKXA/titanium lap shear specimens that were aged under the same conditions and tested at RT show an increase in shear strength after 7 and 28 days aging. Titanium specimens tested at 300°F showed in increase in shear strength after 7 days aging and a 13% decrease after 28 day aging. Figures 11 and 12 are plots of these data.

The PKXA/composite lap shear specimens that were aged in the MIL-H-5606 fluid exhibited some loss in shear strength after exposure to the hydraulic fluid. The failures were in the composite adherends and do not accurately reflect the strength of the adhesive. The data is presented in Figure 13.

The steel/R-5000 crack extension specimens that were aged in MIL-H-5606 exhibited crack growths of .26" after 7 and 28 days in environment. The titanium/R-5000 crack growth ranged from 0" at RT after 7 days to .11" after 28 days. Crack growth was not present on steel/PKXA and titanium/PKXA specimens after exposure in MIL-H-5606 for 7 and 28 days.

MIL-L-7808

The R-5000 bonded titanium and steel lap shear specimens that were environmentally exposed in MIL-L-7808 for 90 days exhibited decreases in shear strengths of 22% and 31%, respectively, when tested at RT and 30% and 37% when tested at 300°F.

Steel lap shear specimens that were bonded with PKXA adhesive and environmentally exposed in MIL-L-7808 for 90 days showed that the environmental aging had very little affect on the adhesive bonds. Specimens that were exposed and tested at RT and 300°F after 90 days showed 2% and 14% loss in shear strength. PKXA/titanium lap shear specimens that were exposed for 90 days and tested at RT and 300 showed losses in shear strengths of 1% when tested at 300° and a 17% increase when testing at room temperature. A summary of these data are found in Tables A-1 through A-5 in the Appendix. Figures 9 through 13 are plots of the data from the fluid exposure.

Composite lap shear specimens that were placed into the environmental aging fluid for 90 days and tested at RT and 300°F showed decreased in shear strength after aging. However, the failures were within the composite adherends and did not necessarily reflect the adhesive bond strength. These data are summarized in Figure 13.

Crack extension specimens that were bonded with R-5000 adhesives show crack grow ths of .44" on steel and .27" on titanium after exposures to MIL-L-7808. The steel and titanium crack extension specimens that were bonded with PKXA adhesive showed no crack growth after 90 days in environment.

2.1.2.3 Deicing Fluid, Fuel and Cleaning Solvent Exposure

Deicing Fluid

The deicing fluid appeared to have no significant effect on the shear strength of the R-5000 and PKXA bonded specimens. The test results did not fit any particular trend. Most of the test results showed an increase in shear strength after aging when compared to the control data. This trend is probably due to data scatter and/or variations in primer or adhesives. The percentage change in lap shear strength ranged from +44% for steel/R-5000 specimens to -14% for steel/PKXA bonded specimens. These data are reported in Figures 14 through 18.

Crack extension specimens that were bonded and environmentally exposed in the deicing fluid showed no signs of crack growth. From these test results, it is apparent that the deicing fluid had no detrimental effect on the R-5000 and PKXA adhesive.

JP-4

Specimens from each of the adherend-adhesive combinations were environmentally exposed in JP-4 fuel for 28 and 90 days and subsequently tested at RT and 300°F for residual shear strength. The R-5000 and PKXA bonded specimens in most cases, failed at shear stresses which were lower than the control specimens.

The R-5000 adhesively bonded specimens exhibited lap shear strength with a range from -8% to -24% of the control values. PKXA specimens showed an increase in shear strength after 28 days and 90 days aging when tested at RT, but a decrease in shear strength when tested at 300°F.

Crack extension specimens from both adhesive systems displayed no crack growth after the 28 or 90 days in

After carefully analyzing the failed specimens and test data, it was apparent that the JP-4 had no real effect on the R-5000 or PKXA adhesives systems. The JP-4 environmental aging data represented in Figures 14 through 18 and Tables A1-A5 in the appendix.

Organic Cleaning Solvent (Stoddard)

Specimens bonded with R-5000 adhesive exhibited decreases in shear strength when immersed in the cleaning solvent for 28 and 90 days and then tested for residual shear strength at RT and 300°F. The steel and titanium lap shear specimens decrease in shear strength with a range of 11% to 24%. See Figures 14-18 for complete summary of the shear strength loss after aging.

The titanium PKXA lap shear specimens that were tested at RT after immersion in the cleaning solvent for 28 to 90 days exhibited a slight increase in shear strength. However, the steel PKXA specimens showed a decrease in shear strength after aging.

The composite lap shear specimens showed a loss of 8-25% from their control values. These data are reported in Figures 14-18 and in Tables 1-5 in the appendix.

Steel and titanium crack extension specimens that were immersed in the organic cleaning solvent exhibited crack growth of 0 to .38 inches. The 0 crack growth was recorded for 90 day aged titanium R-5000 specimens and 28 day steel PKXA specimens. The spread in the test data led us to believe that the aging time in solution had very little to do with crack propagation. The crack extension data are found in Table A1-A4 in the appendix.

2.1.2.4 Salt Water, Caustic Solution, Corrosion Inhibitor, and Naptha Exposure

Salt Water

Unstressed lap shear specimens from all five adherend-adhesive combinations were exposed in 5% sodium chloride solution for 90 days and tested at -65°F, RT, 180°F and 300^{o}F. The shear strength after exposure of all specimens was decreased at all test temperatures. In some cases the decrease was substantial. The reductions are apparently due to the fluids attacking the primer as indicated by the steel and titanium specimens failing in the polyimide primer or primer metal interface. The percentage reduction in shear strength after exposure is plotted in Figure 19 through 23.

The crack extension specimens after 90 day exposure exhibited crack growth which ranged from .44" for steel bonded with R-5000 adhesive to no crack growth for steel bonded with PKXA adhesive. The data are summarized in Tables A-1 through A-4 in the Appendix.

Caustic Solution

Lap shear specimens that were exposed to caustic solution for 28 and 90 days exhibited reductions in shear strength for all adhesive-adherend combinations studied. This reduction ranged from 11% for titanium bonded with R-5000 adhesive to 47% for steel bonded with PKXA.

The failure modes of these specimens were in the primer-adhesive interface with the larger portion of the failure in the polyimide primer. Figure 19 through 23 are plots of the caustic data.

Crack extension specimens exhibited crack growths of ranging from .05" for PKXA titanium specimens to .45 for steel R-5000 specimens.

MIL-C-85054 Corrosion Inhibitor

The R-5000 bonded steel and titanium lap shear specimens exhibited reduction in shear strength after exposing the bonds to MIL-C-85054 corrosion inhibitor. The steel bonded specimens failed with a 18-19% loss in RT +300^{o}F shear strength after 7 days and a 21-22% loss after 28 days in the same environment. Titanium R-5000 specimens failed with a 21% decrease in shear strength at RT and the loss in 300^{o}F strength was reduced to 33-34% of ultimate values. Steel and Titanium PKXA specimens RT shear strength results were higher than the control values when exposed for 7 days and 28 days but showed a loss of shear strength when tested at 300^{o}F.

Composite lap shear specimens bonded with PKXA adhesive showed a decrease in shear strength after exposure to MIL-C-85054 corrosion inhibitor but the decrease did not follow any particular trend in days aging or test temperatures. The test results are presented in Figures 19 through 23.

MIL-C-85054

Titanium and steel crack extension specimens bonded with R-5000 adhesive exhibited crack growth of .38" - .41". Crack growth was not observed among the titanium and steel PKXA bonded crack extension specimens. The crack extension data is found in Table A1-A4 in the appendix.

Naphtha

Neither the R-5000 or PKXA were appreciably affected by naphtha exposure. The greatest lap shear strength reduction (-24%) occured in the R-5000/Titanium bonds tested at room temperature after 28 days exposure. See Figures 19 through 23 and Tables A1 through A5 for test results.

The crack extension specimens displayed very little crack growth during the environmental aging in naphtha for seven and 28 days. The crack growth ranged from 0 to .03". The failures in the crack extension specimens were in the adhesive scrim. The crack extension data is presented in Table A-1 through A-4.

2.2 Task II Mechanical Properties

Under Task II, three basic mechanical properties of PKXA and R-5000 adhesive systems were evaluated on steel and titanium adherends. The properties evaluated included climbing drum peel strength, static creep, and fatigue life. The test parameters are given in Table VII and test results are discussed in 2.2.1 through 2.2.3.

2.2.1 Peel Strength

Peel strength of R-5000 and PKXA adhesives on steel and titanium adherends were determined using the climbing drum method. The specimens were fabricated with an .012" peeling skin and .032" back skin. The cleaning and bonding was the same as used in section 2.1. Specimens were tested at RT and 250^{o}F. There was no significant difference in the peel strength between specimens tested at RT and 250°F. For all conditions tested, peel strengths ranged from 31 to 39 in #/in width The failure mode was mostly cohesive within the adhesive scrim with some failures within the polyimide primer. Figures 24 and 25 are typical failures of the peel specimens after testing.

The peel strength of the R-5000 and PKXA adhesives on titanium and steel are summarized in Table VIII.

2.2.2 Creep Resistance

The creep resistance of PKXA and R-5000 on steel and titanium adherends was determined at RT and 250°F. Both adhesive systems exhibited good creep resistance. A comparison of the data with the applicable Military specification (MMM-A-132) requirement is presented in Table IX. Complete test results are presented in the Appendix (Tables A-6 through A-9).

2.2.3 Fatigue Life

Fatigue characteristics of PKXA and R5000 adhesive bonds on steel and titanium adherends were determined at room temperature. The standard lap shear specimen was used (Figure 27) to evaluate fatigue performance at 30, 50, and 80 percent of ultimate tensile shear strength. The stress ratio was .1 and cycling rate was 1800 cpm. Test results are presented in Figures 28 through 31.

At eighty percent of ultimate, specimens consistently endured 1×10^3 cycles regardless of adherend or adhesive evaluated except for PKXA/steel bond joints where two out of three specimens failed after 25 cycles. At the other end of the load spectrum (30%) the PKXA/steel bonds performed better than the other combinations: two specimens had not failed after 1×10^6 cycles and testing was discontinued on another specimen after 1.25×10^7 cycles.

2.3 Data Assessment

In this section, an assessment will be made of the PKXA and R-5000 performance in areas evaluated during this program. The areas are thermal stability, environmental resistance, metal-to-metal peel strength, creep resistance and fatigue life.

In the areas evaluated, performance of systems on the steel and titanium adherends was comparable to commonly used structural adhesives and exceeded the applicable military specification requirements (MMM-A-132, Class II). Additionally, certain exposure and test parameters were intentionally selected to exceed the MMM-A-132 requirements in order to better assess the performance of the thermoplastic adhesives.

In the PKXA/composite bond joint study, the lap shear strength consistently exceeded the strength of the polyimide/fiberglass adherends.

2.3.1 Thermal Stability

Thermal aging of PKXA and R-5000 adhesives at 180 and 300°F for 90 days did not appreciably affect the strength of either adhesive systems on metal adherends.

The PKXA/composite bonds were the only joints which consistently experienced lap shear strength loss after thermal aging. The most severe reduction (32%) occurred in the room temperature strength after aging 90 days at 300°F.

2.3.2 Environmental Exposure

The environmental resistance of both PKXA and R-5000 exceeded MMM-A-132 requirements for fluid exposure to deicing fluids, JP-4 fuel, MIL-H-5606 hydraulic fluid, MIL-L-7808 synthetic lubricant and salt water (5%). This is also true of exposure to fluids (Stoddard cleaning solvent, MIL-C-85054 corrosion inhibitor and Naphtha) not required by the MMM-A-132.

The lap shear specimens that were immersed in caustic solution for 28 and 90 days were affected significantly from the exposure. The attack on the bonded joints seem to be more on the primer and polyimide composite than on the adhesives. The aluminum and polyimide materials were points of attack for the basic solution. The composite lap shear specimens failed in the composite materials. The greatest reduction (48%) occurred in steel/PKXA bonds tested at RT and 300°F after 90 days exposure. There is no MMM-A-132 requirement for exposure in this solution. Also the 90 day exposure duration is longer than likely to be encountered in aerospace applications.

The MIL-H-83306 hydraulic fluid exposure of 90 days, completely deteriorated the R-5000 bonds with strength loss exceeding 90% for the R-5000/Steel bonds tested at 300°F. However, the PKXA adhesive was not affected by this exposure: the worst strength loss (22%) occurred in the PKXA/titanium joints tested at 300°F.

2.3.3 Peel Strength

The peel strengths of the R-5000 and PKXA adhesives were adequate for structural bonding when measured by the climbing drum peel test method. The values ranged from 30-40 in./lbs. on both adhesive systems when tested at RT and 250°F. The failure modes of these adhesive systems were basically cohesive within the adhesive with some primer failure.

2.3.4 Creep Resistance

The work performed under this contract indicates that the R-5000 and PKXA adhesive systems have acceptable creep resistance when bonded to stainless steel and titanium adherends. However, as the testing progressed, it became apparent that the adhesive primer system was influencing test results appreciably. Specifically, some specimens were failing within the adhesive primer or primer/metal interface at shorter time-to-failure periods than expected for the loads imposed. Because of the poor reliability of the adhesive primer systems, there is some scatter in the data. Even so, the adhesives exhibited acceptable creep, as specified by the MM-A-132 specification. It is apparent that the adhesive systems are suitable in creep resistance for structural bonding applications.

2.3.5 Fatigue Life

The PKXA and R-5000 fatigue data which was obtained on bonded steel and titanium lap shear specimens were considered adequate for most structural bonding applications. No direct comparison is possible with MM-A-132 requirements since a higher load was used in this evaluation than the 750 psi required in the military specification. For comparative purposes, the PKXA/steel joints tested at 1068 psi had not failed after 10^6 cycles and in one case 10^7 cycles when testing was discontinued.

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 Conclusions

Of the two adhesives systems evaluated in this program, the general conclusion reached is that PKXA is suitable for structural aerospace applications while RADEL 5000 should not be used in applications where exposure to phosphate ester type fluids is a possibility. This conclusion is applicable to titanium, stainless steel, and polyimide/fiberglass composite adherends over the service temperature range of -65 to 300^{o}F.

The best surface preparations of those evaluated were chromic acid anodize for titanium, sulfuric acid anodize for stainless steel and grit blast followed by solvent wipe for polyimide/fiberglass composites.

The adhesive primer system used in this program was not optimum. It consisted of a commercially available polyimide adhesive primer (BR34 by American Cyanamid) being applied to the faying surfaces, dried, and then overcoated with a PKXA or RADEL 5000 primer to match the adhesive film used.

3.2 Recommendations

This program has established the basic structural adequacy of thermoplastic adhesives on titanium, stainless steel, and polyimide/fiberglass composite adherends. However, additional efforts appears warranted to both optimize and further characterize the adhesive systems. Candidate areas for these efforts include:

- adhesive primer optimization
- postforming studies to establish bend radius/residual stress relationship
- develop bonding techniques applicable to aluminum adherends.
- develop and/or evaluate methods for rapid assembly bonding.

REFERENCES

1. NAVAIR Contract N00019-74-C-0226, "Evaluation of Thermoplastic Composites and Adhesives".

2. NAVAIR Contract N00019-76-C-0170, "Evaluation of Thermoplastic Adhesives".

TABLE I - THE EFFECT OF SURFACE PREPARATION ON PKXA LAP SHEAR STRENGTH

Adherend	Surface Preparation	Primer(s)	Shear Strength (psi)	
			RT	300°F
Titanium	Chromic Acid Anodize	BR-34	3160	2140
		BR-34/PKXA	4380	2680
		PKXA	3230	2390
	Paśa Gel	BR-34	1920	1705
		BR-34/PKXA	3230	2390
		PKXA	5175	3080
	Phosphate Fluoride	BR-34	2780	2105
		BR-34/PKXA	3960	2745
		PKXA	2985	1900
Steel	Sulfuric Acid Anodize	BR-34	3510	2820
		BR-34/PKXA	4345	2375
		PKXA	2575	2035
Polyimide Composite	Scotchbrite + Solvent	BR-34/PKXA	3360	2140
	Vacuum Blast + Solvent	BR-34/PKXA	3820	2530
	Peel Ply	BR-34/PKXA	3040	1875

TABLE II - THE EFFECT OF SURFACE PREPARATION ON R-5000 LAP SHEAR STRENGTH

Adherend	Surface Preparation	Primer(s)	Shear Strength (psi)	
			RT	300°F
Titanium	Chromic Acid Anodize	BR-34	3325	2460
		BR-34/R-5000	3685	2830
		R-5000	4380	2680
	Pasa Gel	BR-34	3860	2300
		BR-34/R-5000	4080	2900
		R-5000	3190	2760
	Phosphate Fluoride	BR-34	3780	2680
		BR-34/R-5000	4120	2785
		R-5000	3365	2635
Steel	Sulfuric Acid Anodize	BR-34	3460	2162
		BR-34/R-5000	4080	3205
		R-5000	2965	2205
Polyimide Composite	Scotchbrite + Solvent	BR-34/R-5000	2965	1840
	Vacuum Blast + Solvent	BR-34/R-5000	3435	2155
	Peel Ply	BR-34/R-5000	3140	1955

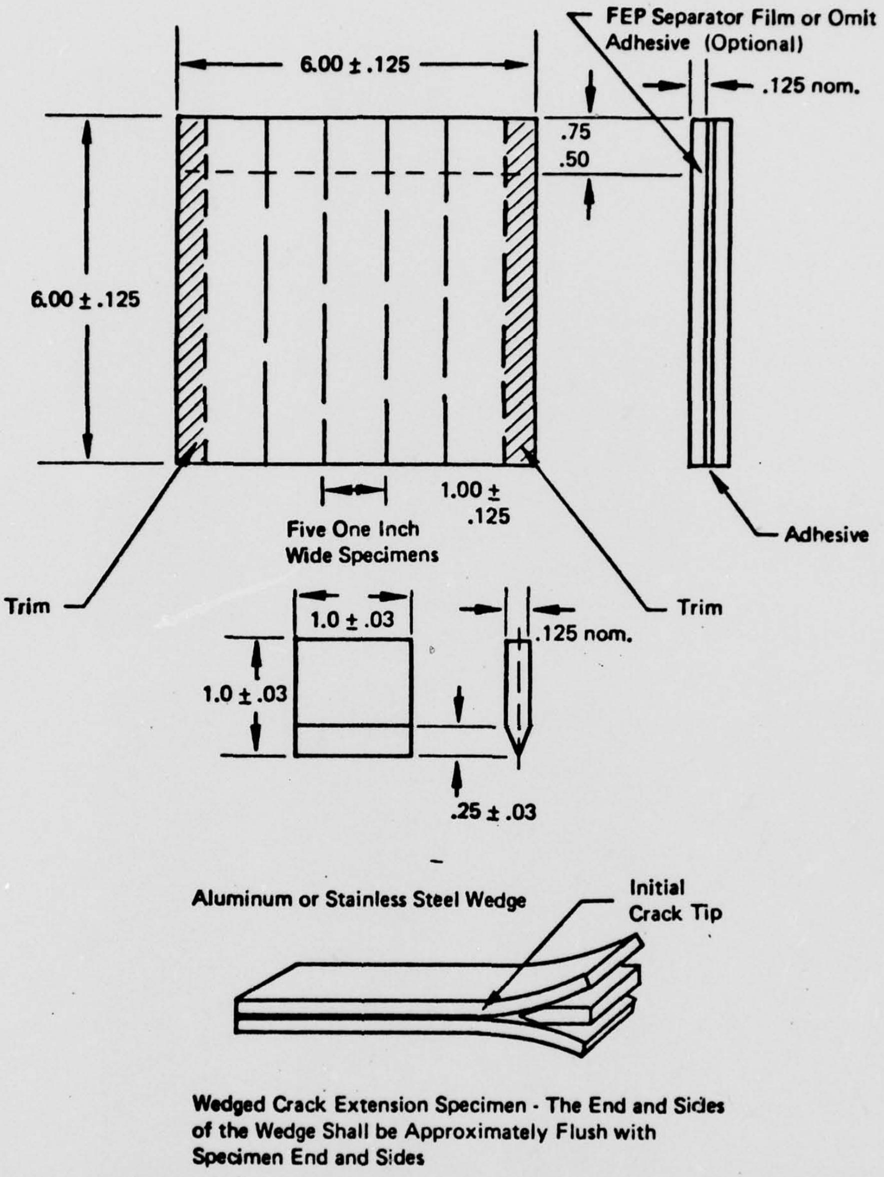


Fig. 1 CRACK EXTENSION SPECIMEN

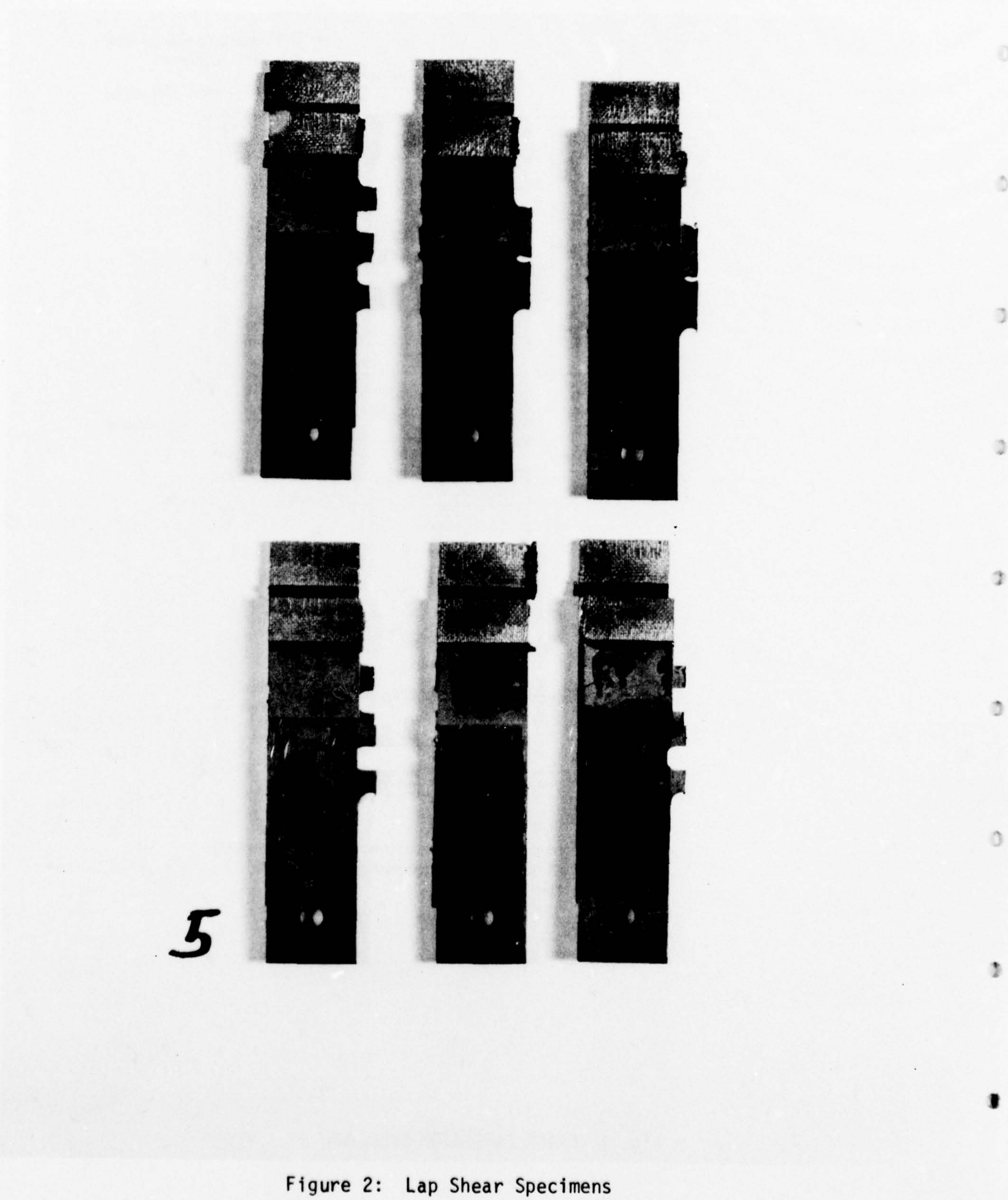


Figure 2: Lap Shear Specimens

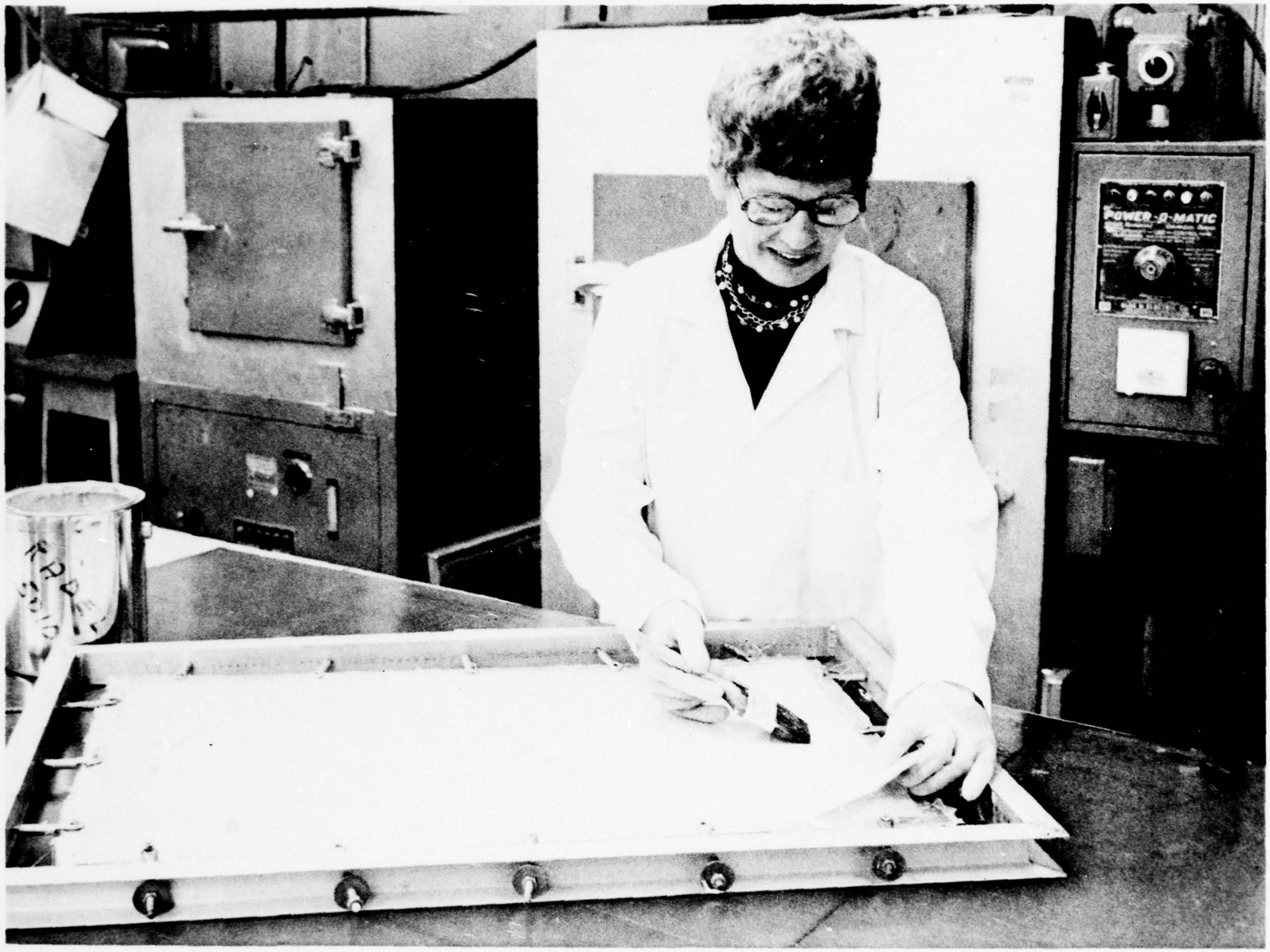


Figure 3: Adhesive Film Preparation

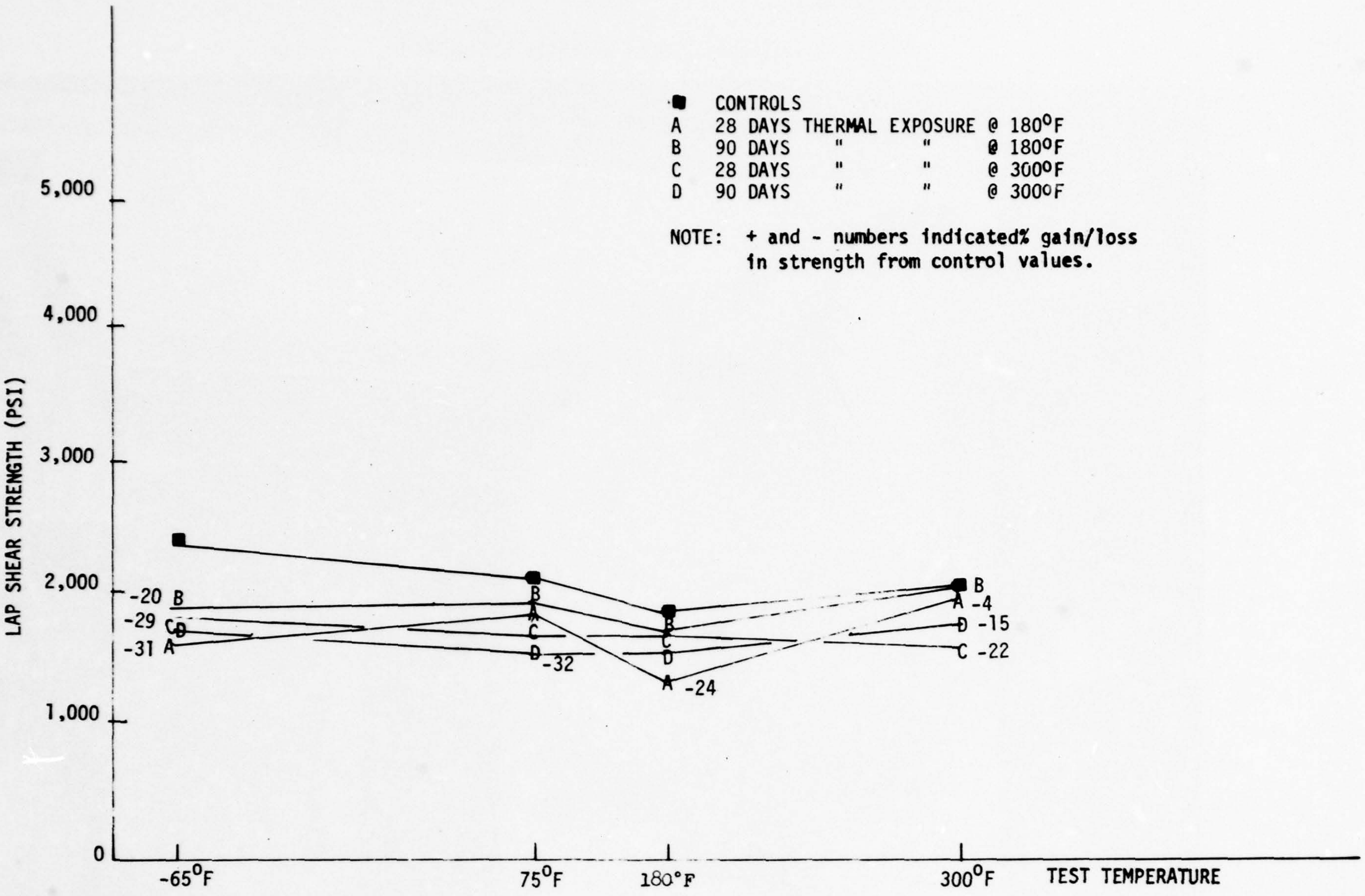
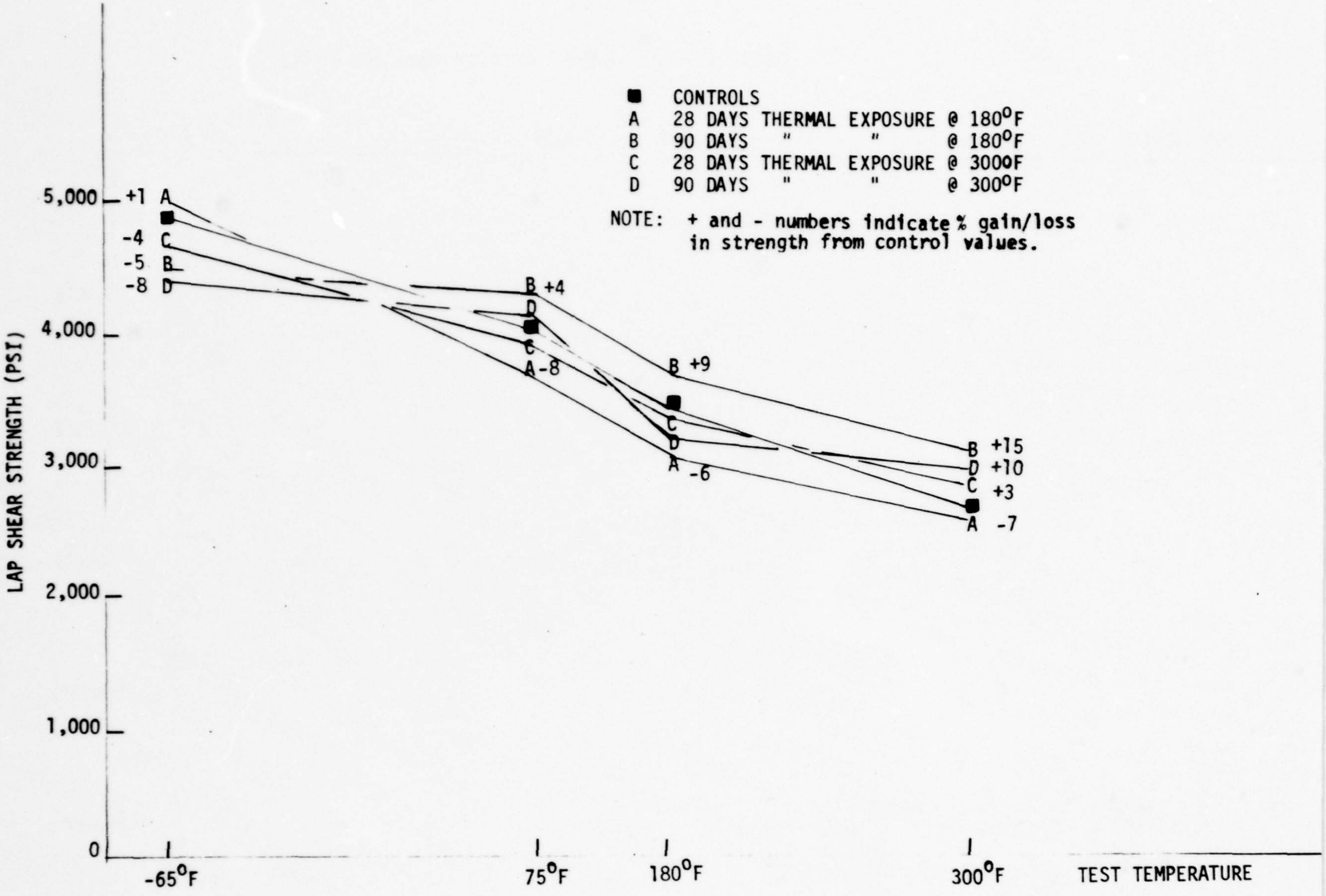


Fig. 4 THERMAL AGING OF PKXA/COMPOSITE BOND JOINTS



NOTE: + and - numbers indicate % gain/loss in strength from control values.

Fig. 5 THERMAL AGING OF R-5000/STEEL BOND JOINTS

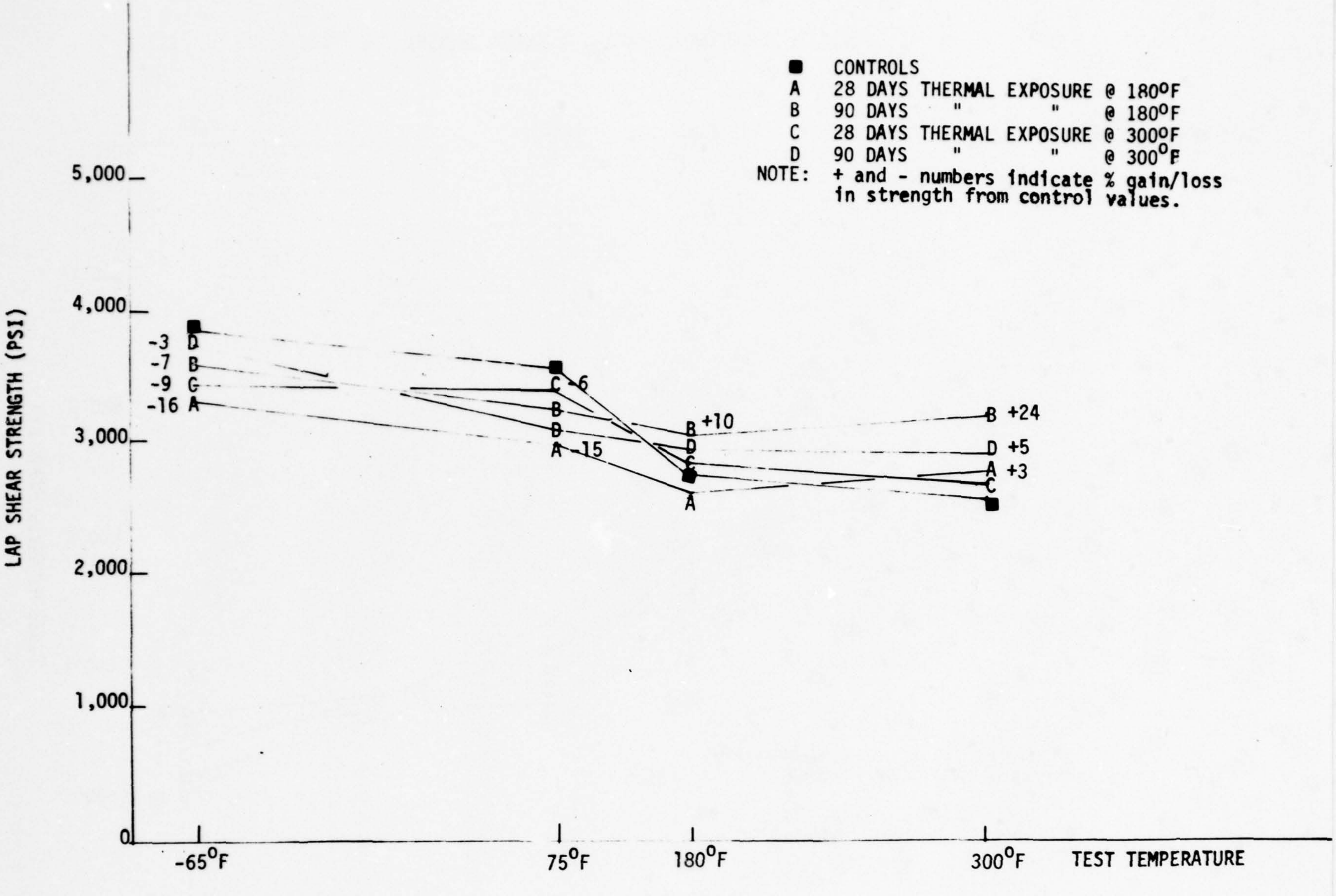


Fig. 6 THERMAL AGING OF PKXA/STEEL BOND JOINTS

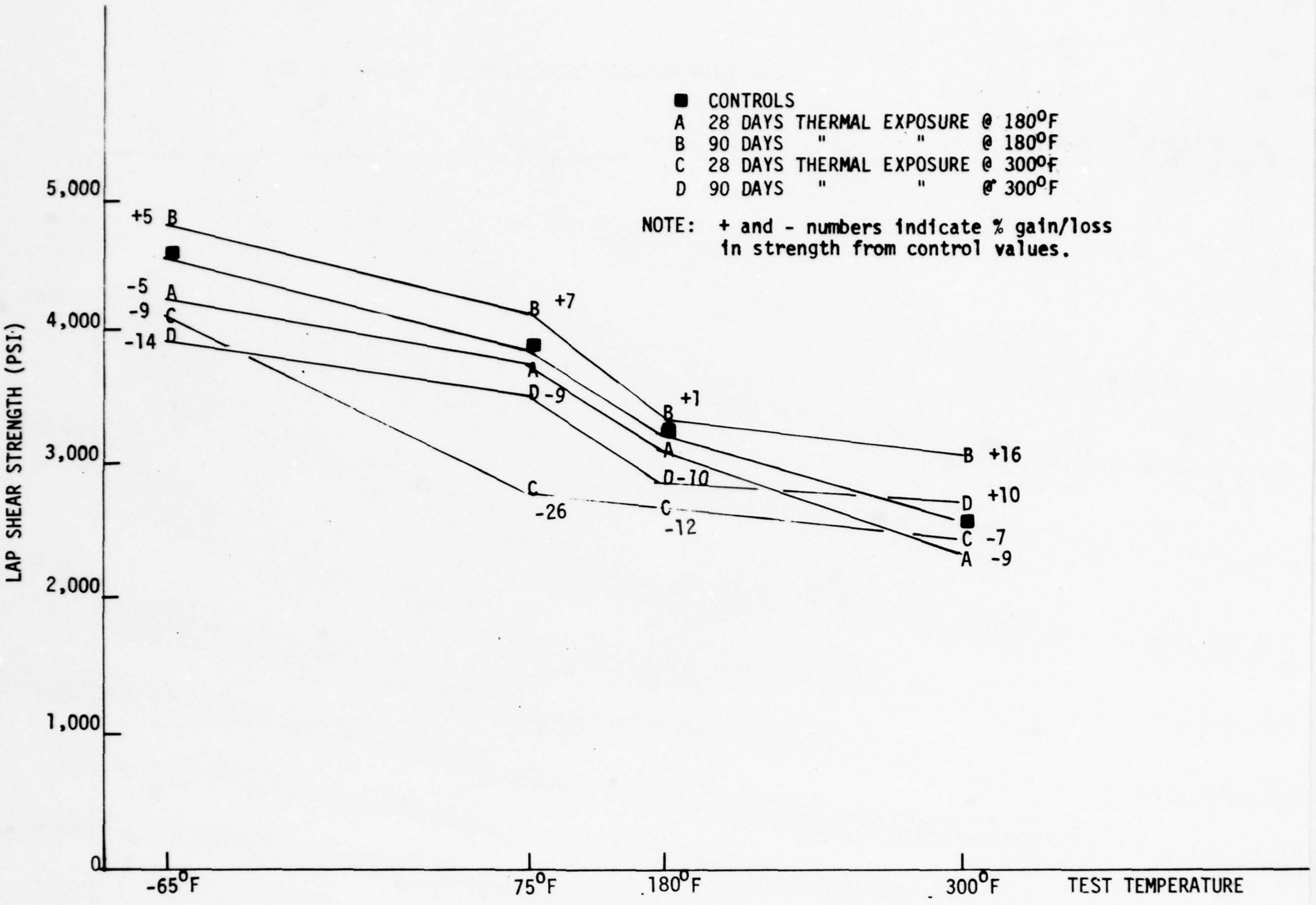


Fig. 7 THERMAL AGING OF R-5000/TITANIUM BOND JOINTS

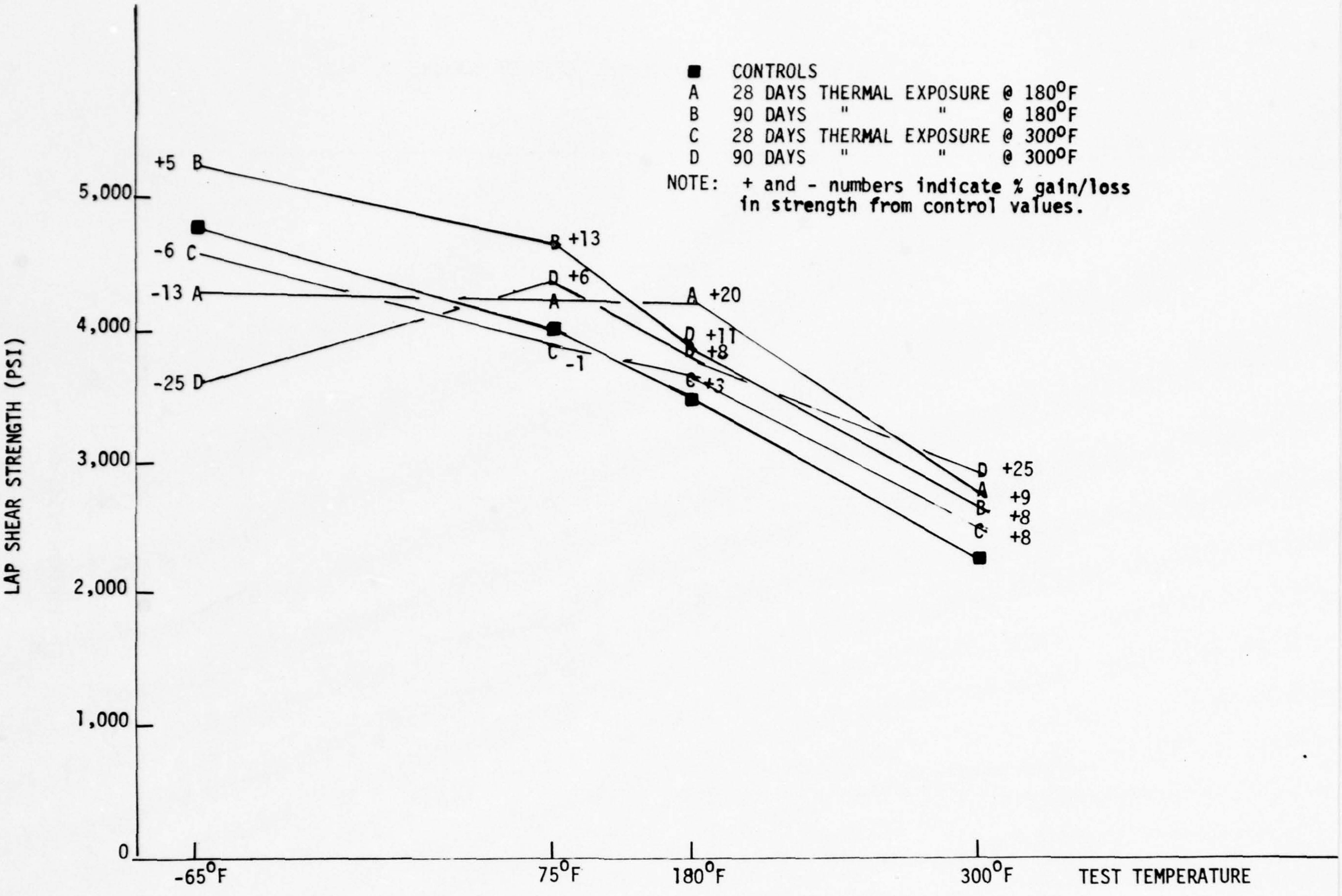


Fig. 8 THERMAL AGING OF PKXA/TITANIUM BOND JOINTS

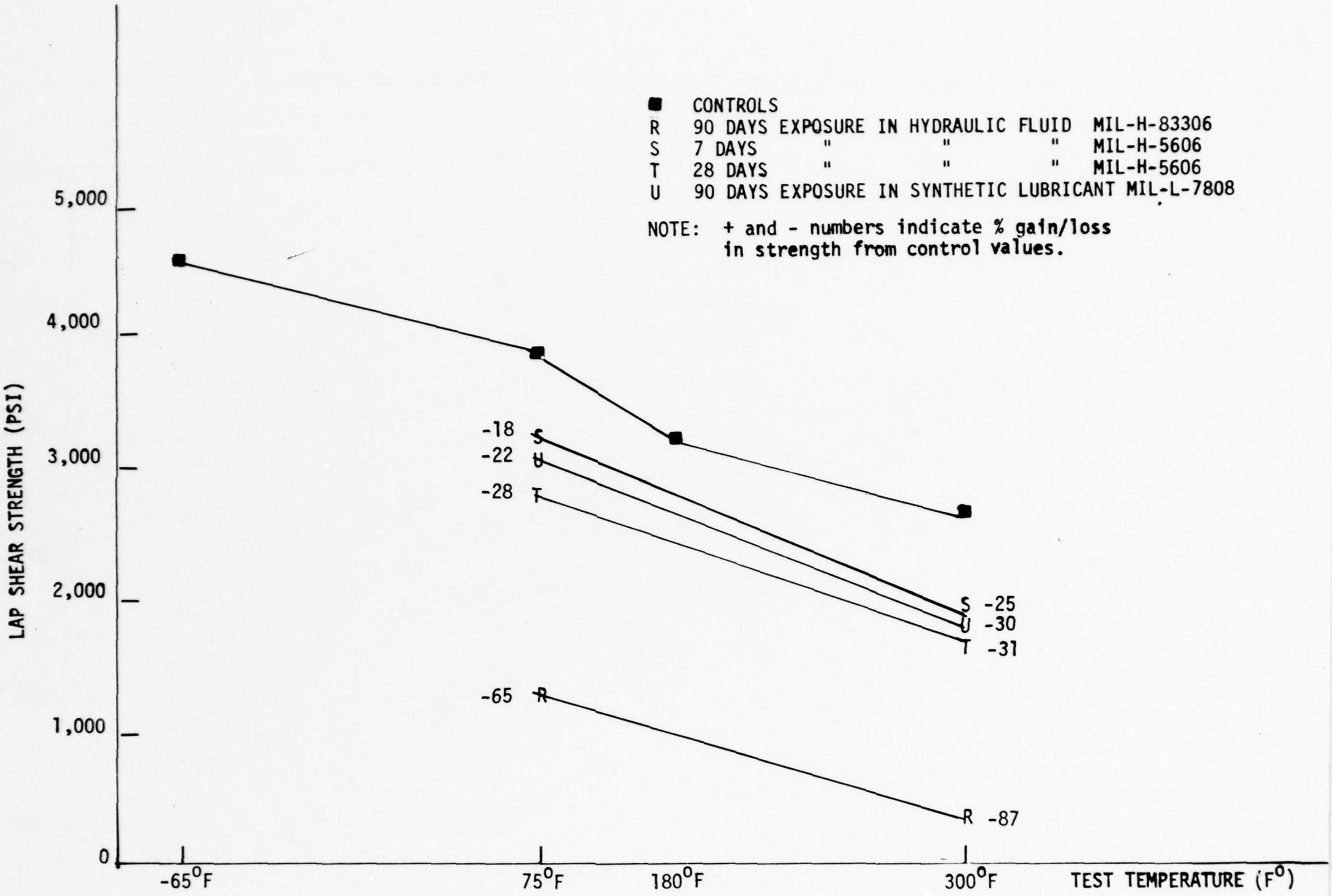


Fig. 9 EFFECT OF HYDRAULIC FLUIDS AND SYNTHETIC LUBRICANTS ON R-5000/TITANIUM BONDS

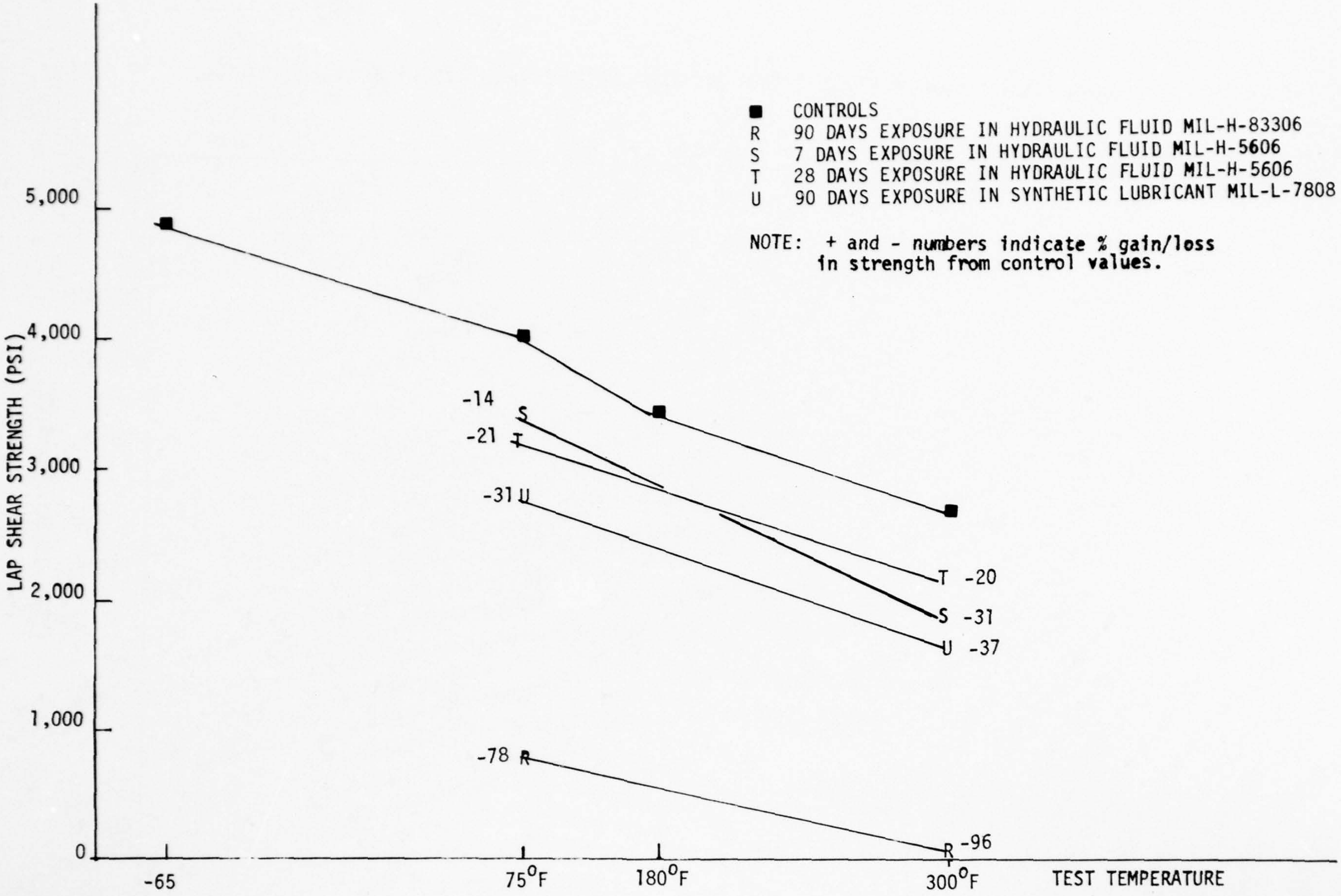


Fig. 10 EFFECT OF HYDRAULIC FLUIDS AND SYNTHETIC LUBRICANT ON R-5000/STEEL BONDS

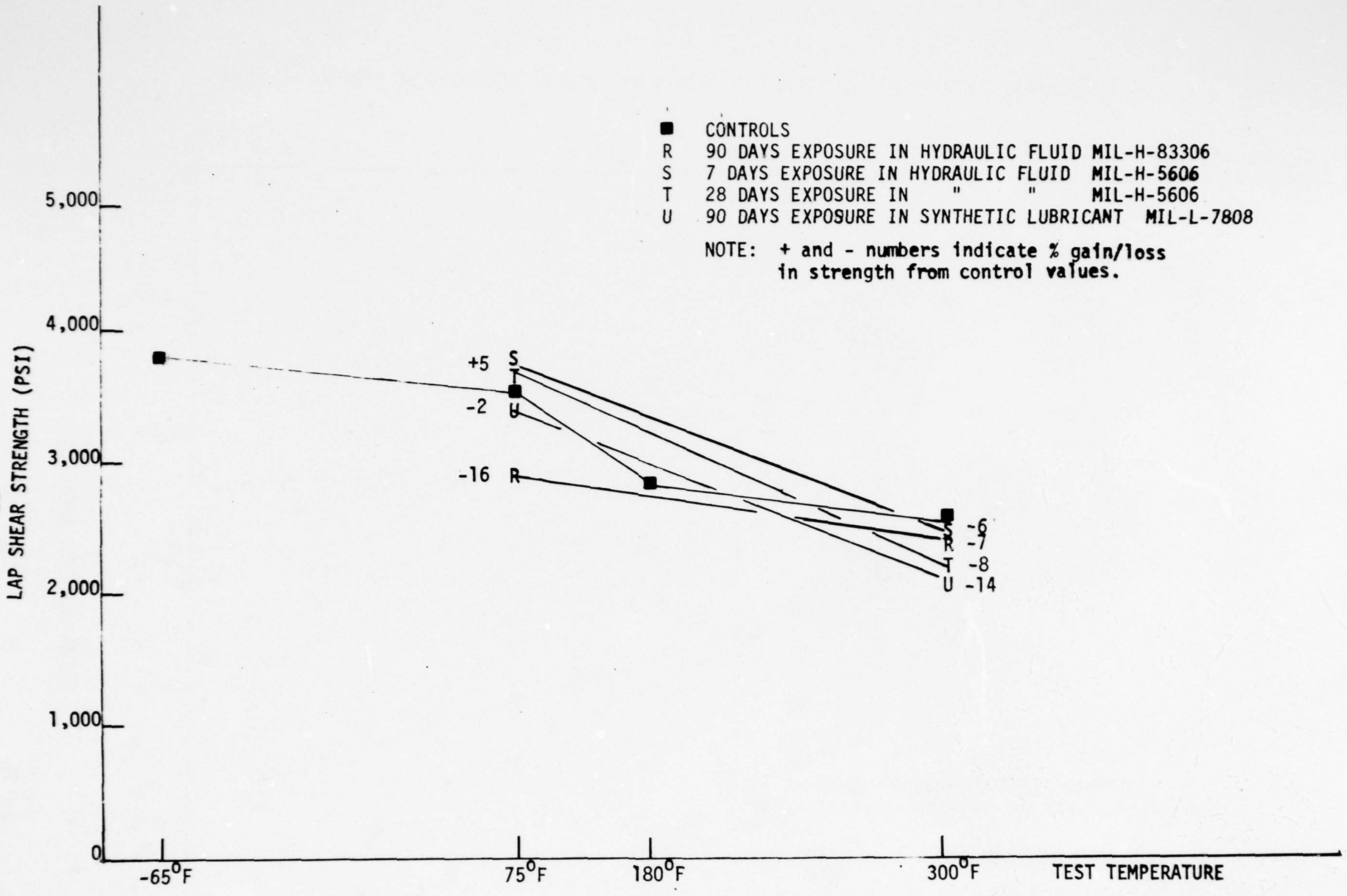


Fig. 11 EFFECT OF HYDRAULIC FLUIDS AND SYNTHETIC LUBRICANTS ON PKXA/STEEL BONDS

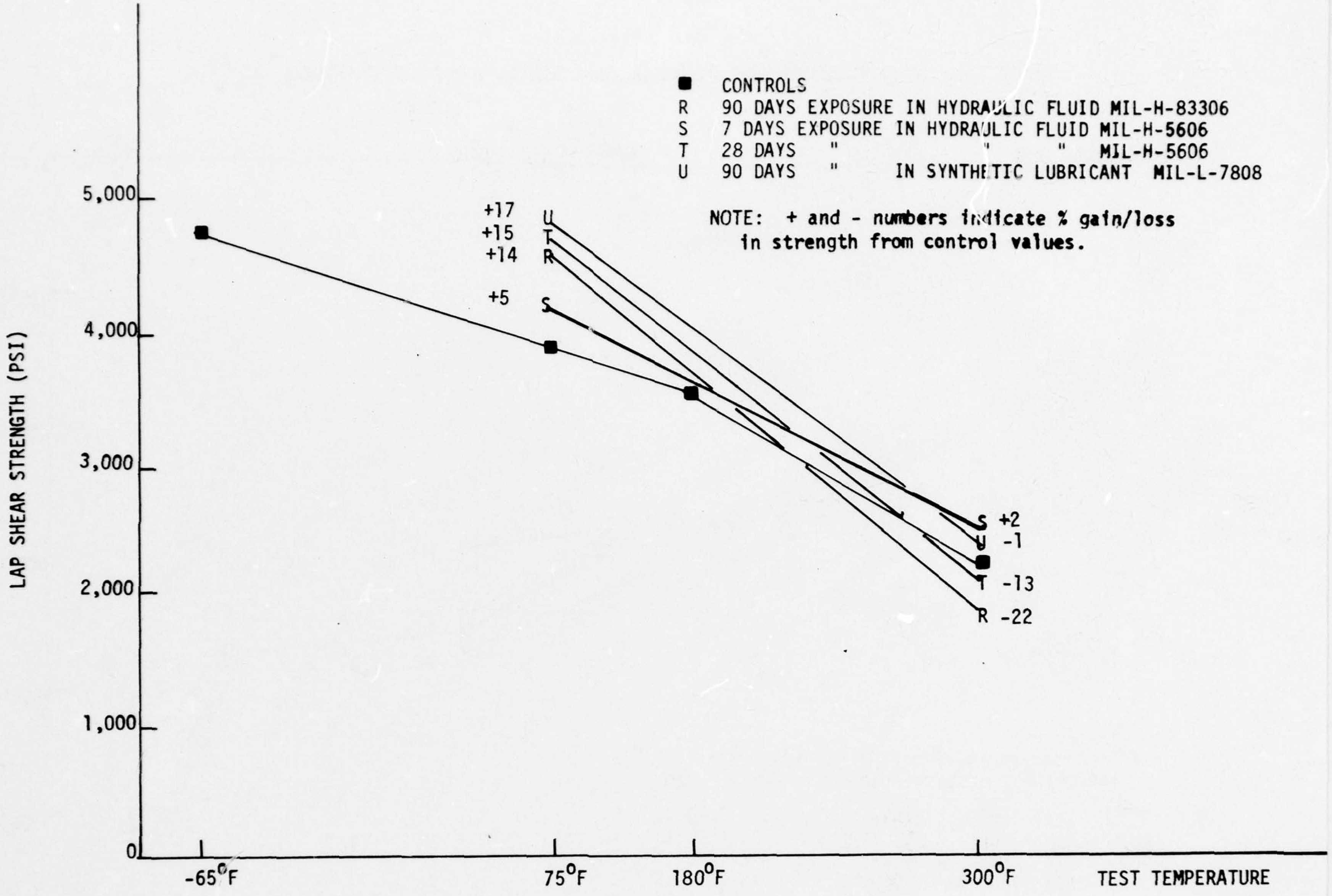


Fig. 12 EFFECT OF HYDRAULIC FLUIDS AND SYNTHETIC LUBRICANTS ON PKXA/TITANIUM BONDS

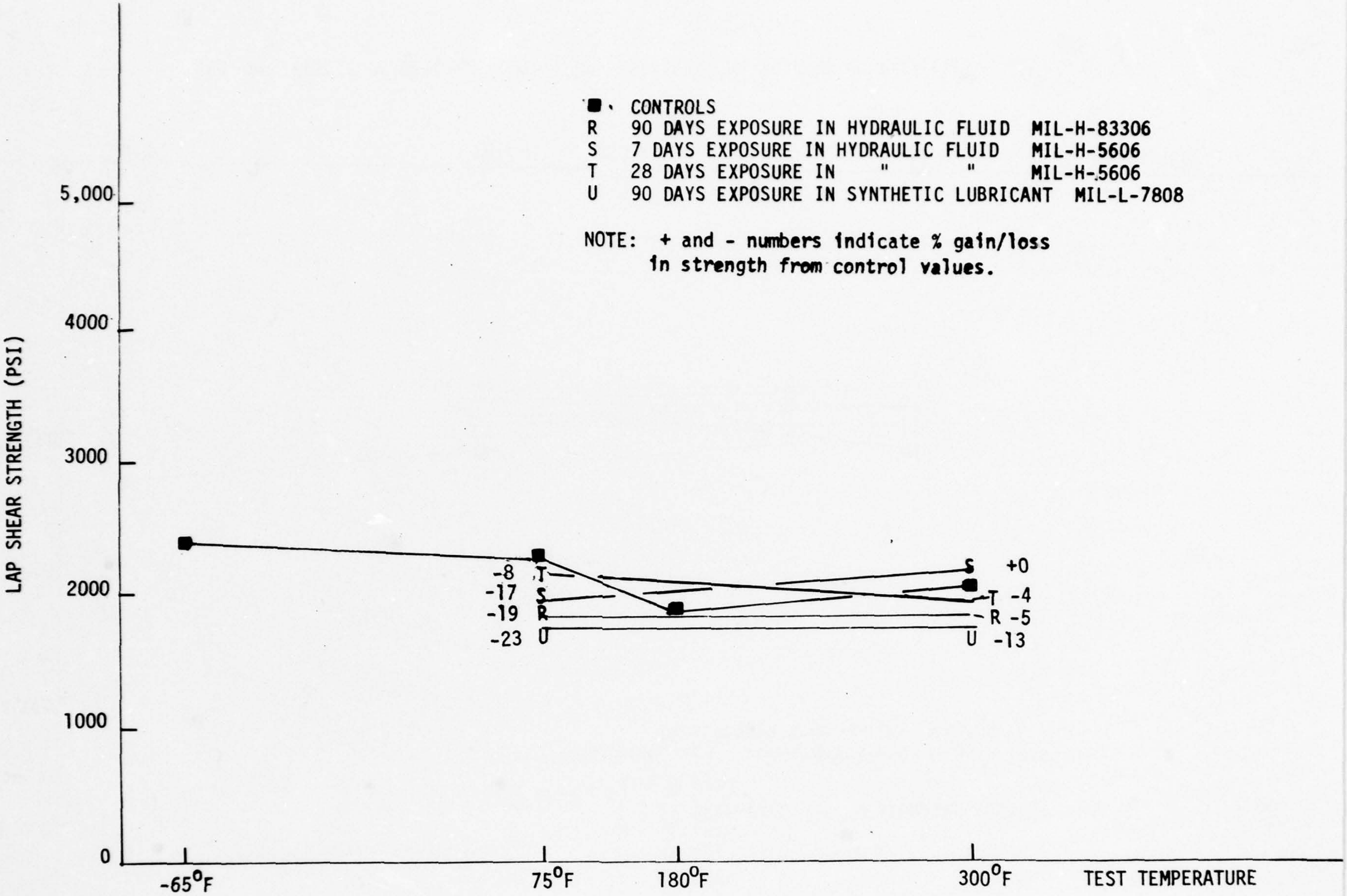


Fig.13 EFFECT OF HYDRAULIC FLUIDS AND SYNTHETIC LUBRICANT ON PKXA/COMPOSITE BONDS

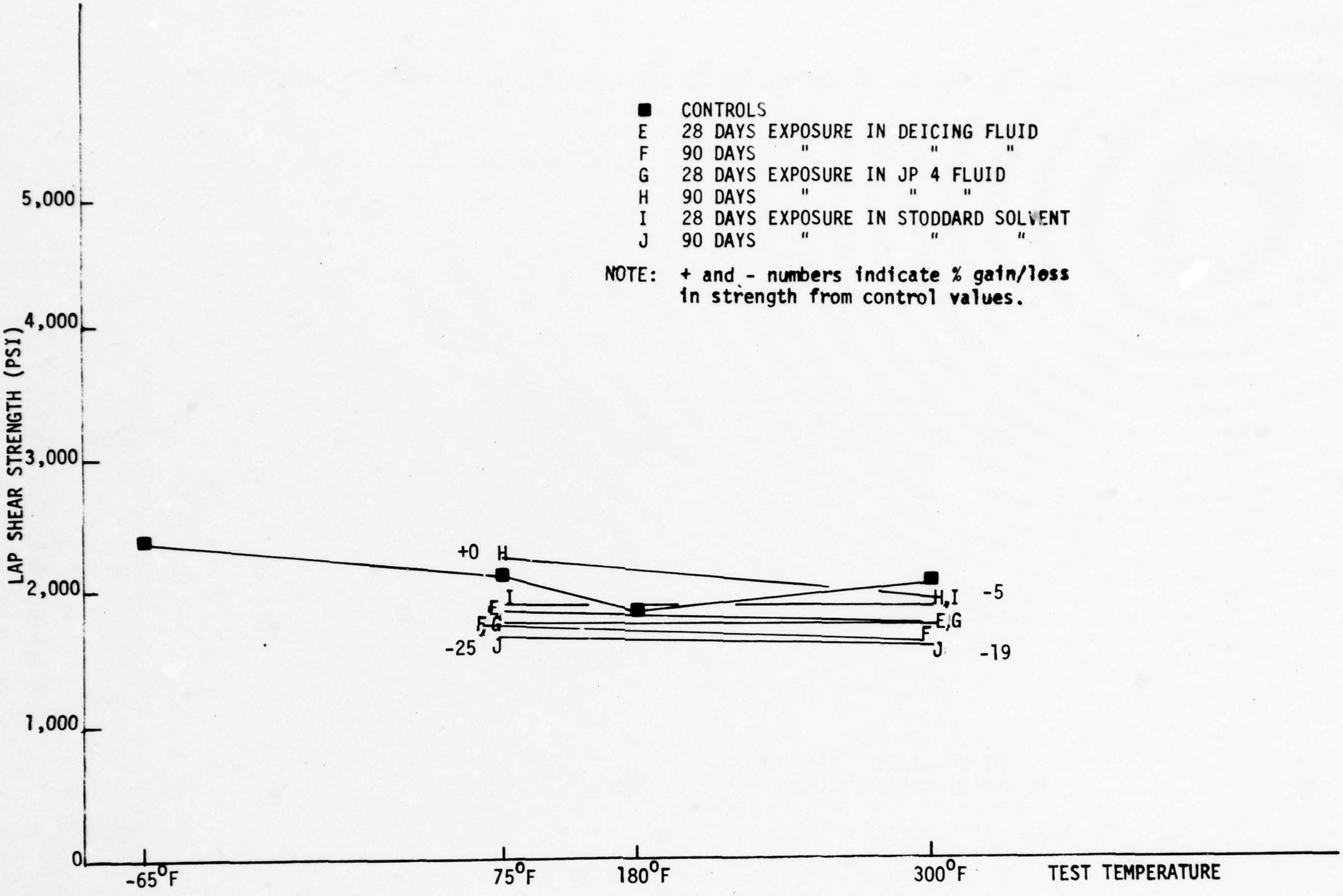


Fig. 14 EFFECT OF DEICING FLUID, FUEL AND CLEANING SOLVENT ON PKXA/COMPOSITE BONDS

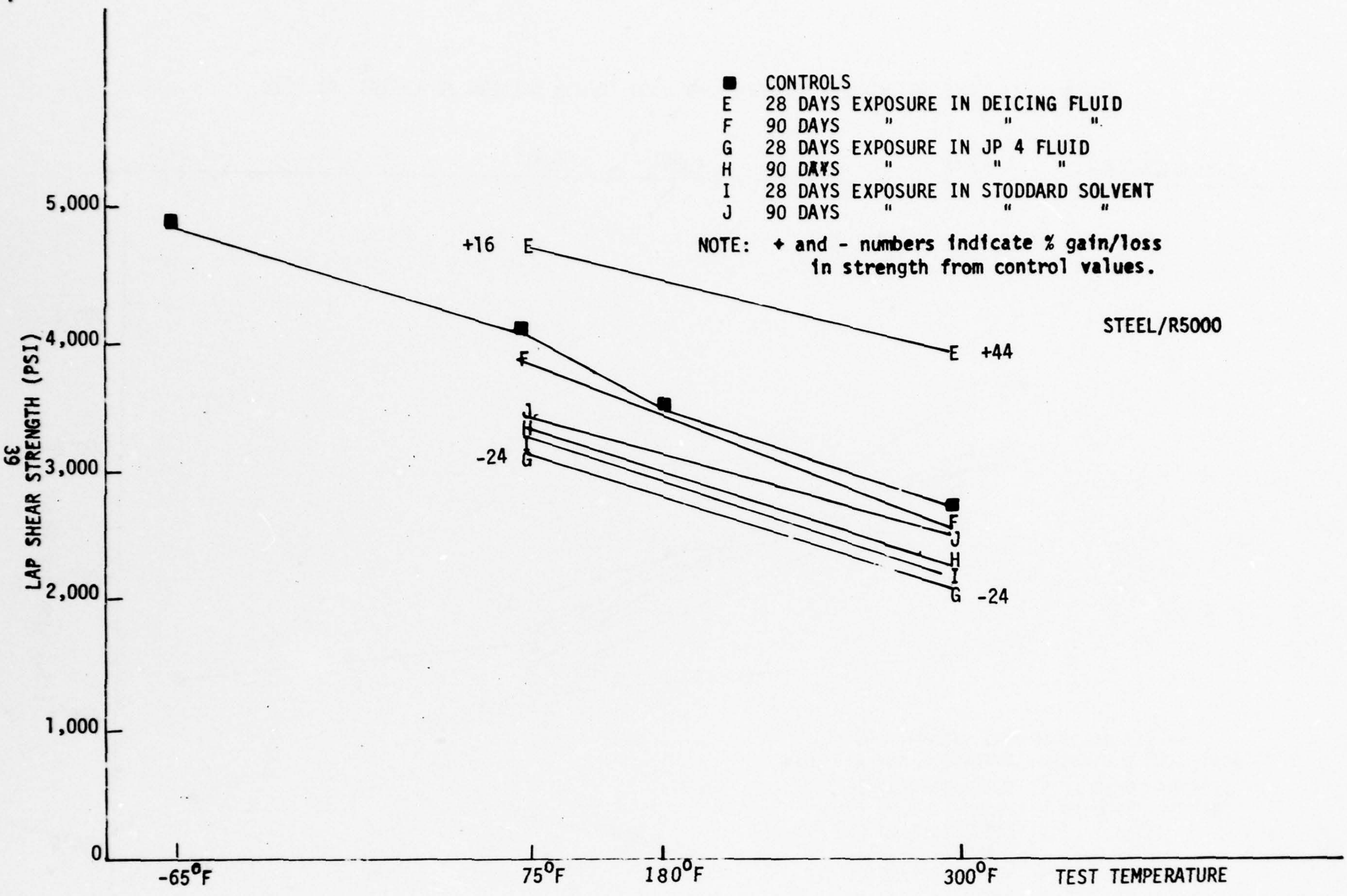


Fig. 15 EFFECT OF DEICING FLUID, FUEL AND CLEANING SOLVENT ON R-5000/STEEL BONDS

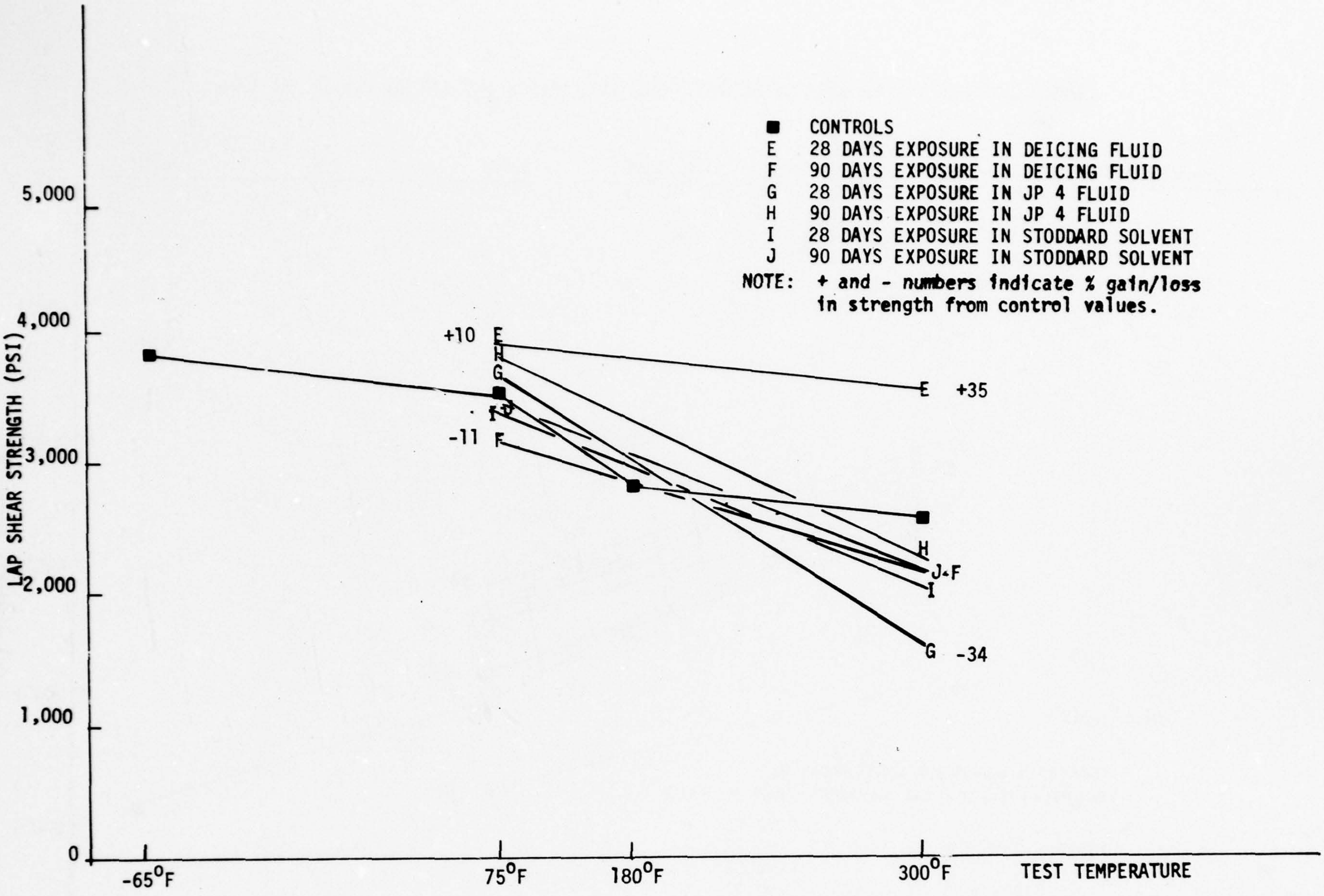


Fig. 16 EFFECT OF DEICING FLUID, FUEL AND CLEANING SOLVENT ON PKXA/STEEL BONDS

TABLE III- PKXA CRACK EXTENSION TEST RESULTS AFTER 200 HOURS IN CONDENSING HUMIDITY

Adherend	Surface Treatment	Primer(s)	Type of Failure (% Cohesive)	Initial Crack Length (inches)	Crack Growth (in.)
Titanium	Chromic Acid Anodize	BR-34	85 - 90	1.56	.05
		BR-34/PKXA	91 - 94	1.52	None
		PKXA	87 - 92	1.47	.4
	Pasa Gel	BR-34	75 - 80	1.72	.21
		BR-34/PKXA	78 - 85	1.63	.18
		PKXA	72 - 77	1.77	.32
	Phosphate Fluoride	BR-34	85 - 92	1.63	.17
		BR-34/PKXA	83 - 90	1.57	.23
		PKXA	80 - 86	1.66	.28
Steel	Sulfuric Acid Anodize	BR-34	85 - 89	1.46	.07
		BR-34/PKXA	90 - 92	1.42	.03
		PKXA	89 - 90	1.48	.05

TABLE IV - R-5000 CRACK EXTENSION TEST RESULTS AFTER 200 HOURS IN CONDENSING HUMIDITY

Adherend	Surface Treatment	Primer(s)	Type of Failure (% Cohesive)	Initial Crack Length (inches)	Crack Growth (inches)
Titanium	Chromium Acid Anodize	BR-34	80 - 85	1.51	.03
		BR-34/R-5000	90 - 92	1.43	None
		R-5000	85 - 90	1.54	.07
	Pasa Gel	BR-34	85 - 87	1.47	.04
		BR-34/R-5000	83 - 85	1.43	.01
		R-5000	80 - 82	1.49	.05
	Phosphate Fluoride	BR-34	75 - 80	1.48	None
		BR-34/R-5000	91 - 93	1.45	.03
		R-5000	80 - 85	1.48	.05
Steel	Sulfuric Acid Anodize	BR-34	87 - 91	1.47	.07
		BR-34/R-5000	90 - 95	1.43	.02
		R-5000	85 - 90	1.49	.07

TABLE V : ENVIRONMENTAL TEST PROGRAM

ENVIRONMENT	EXPOSURE TIME	LAP SHEAR STRENGTH DETERMINATION [4]				CRACK EXTENSION, [1] +70^{0}F STRESSED
		-65^{0}F	+70^{0}F	+180^{0}F	+300^{0}F	
Control	28 Days	10	10	10	10	5
Thermal Exposure, +180^{0}F	28 Days	3	3	3	3	2
	90 Days	3	3	3	3	
Thermal Exposure, +300^{0}F	28 Days	3	3	3	3	2
	90 Days	3	3	3	3	2
Deicing Fluid	28 Days	-	3	-	3	2
	90 Days	-	3	-	3	-
JP-4 Jet Fuel	28 Days	-	3	-	3	2
	90 Days	-	3	-	3	-
Hydraulic Fluid (Phosphate ester type)	90 Days	-	3	-	3	2
Hydraulic Fluid (Standard petroleum type)	7 Days	-	3	-	3	2
	28 Days	-	3	-	3	2
Synthetic Lubricant	90 Days	-	3	-	3	2
Salt Water	90 Days	3	3	3	3	2
Caustic	28 Days	-	3	-	3	2
	90 Days	-	3	-	3	-
Cleaning Solvent (Stoddard)	28 Days	-	3	-	3	2
	90 Days	-	3	-	3	-
Naphtha	7 Days	-	6	-	6	6
Corrosion Inhibitor	28 Days	-	6	-	6	-
TOTAL		25	73	25	73	37
TOTAL SPECIMENS (5 systems) [3]		125	365	125	365	148 [2]

[1] Crack Extension Specimen - 4 Systems with Metallic Adherends Only (See [3])

[2] Four systems only

[3] 5 systems: 1. Steel + PKXA 2. Titanium + PKXA 3. Steel + R-5000 4. Titanium + R-5000 5. Composite + PKXA

[4] No. of test specimens per system.

TABLE VI: Effects of Thermal Exposure on Lap Shear Strength

	TEST TEMPERATURE AFTER EXPOSURE																			
	-65^{O}F					RT					180^{O}F					300^{O}F				
Exposure	PKXA/COMPOSITE	R5000/STEEL	PKXA/STEEL	R5000/Ti	PKXA/Ti	PKXA/COMPOSITE	R5000/STEEL	PKXA/STEEL	R5000/Ti	PKXA/Ti	PKXA/COMPOSITE	R5000/STEEL	PKXA/STEEL	R5000/Ti	PKXA/Ti	PKXA/COMPOSITE	R5000/STEEL	PKXA/STEEL	R5000/Ti	PKXA/Ti
180^{O}F for 28 days	30	+1	15	5	13	22	8	15	1	1	23	6	+3	2	+20	4	7	+3	9	+9
180^{O}F for 90 days	20	5	7	+5	+3	22	0	2	+7	+13	10	+9	+9	+1	+ 8	0	+15	+23	+16	+9
300^{O}F for 28 days	29	5	9	9	6	26	4	6	27	1	12	+2	4	10	+ 3	22	+3	+1	7	+9
300^{O}F for 90 days	29	8	2	14	25	32	+4	14	9	+5	19	6	+1	12	+10	15	+10	+5	+9	+25

NOTES:
1. Average of three specimens per condition.
2. All + values indicate % strength increase above control value.
3. See Tables 1 through 5 in the Appendix for strength values.

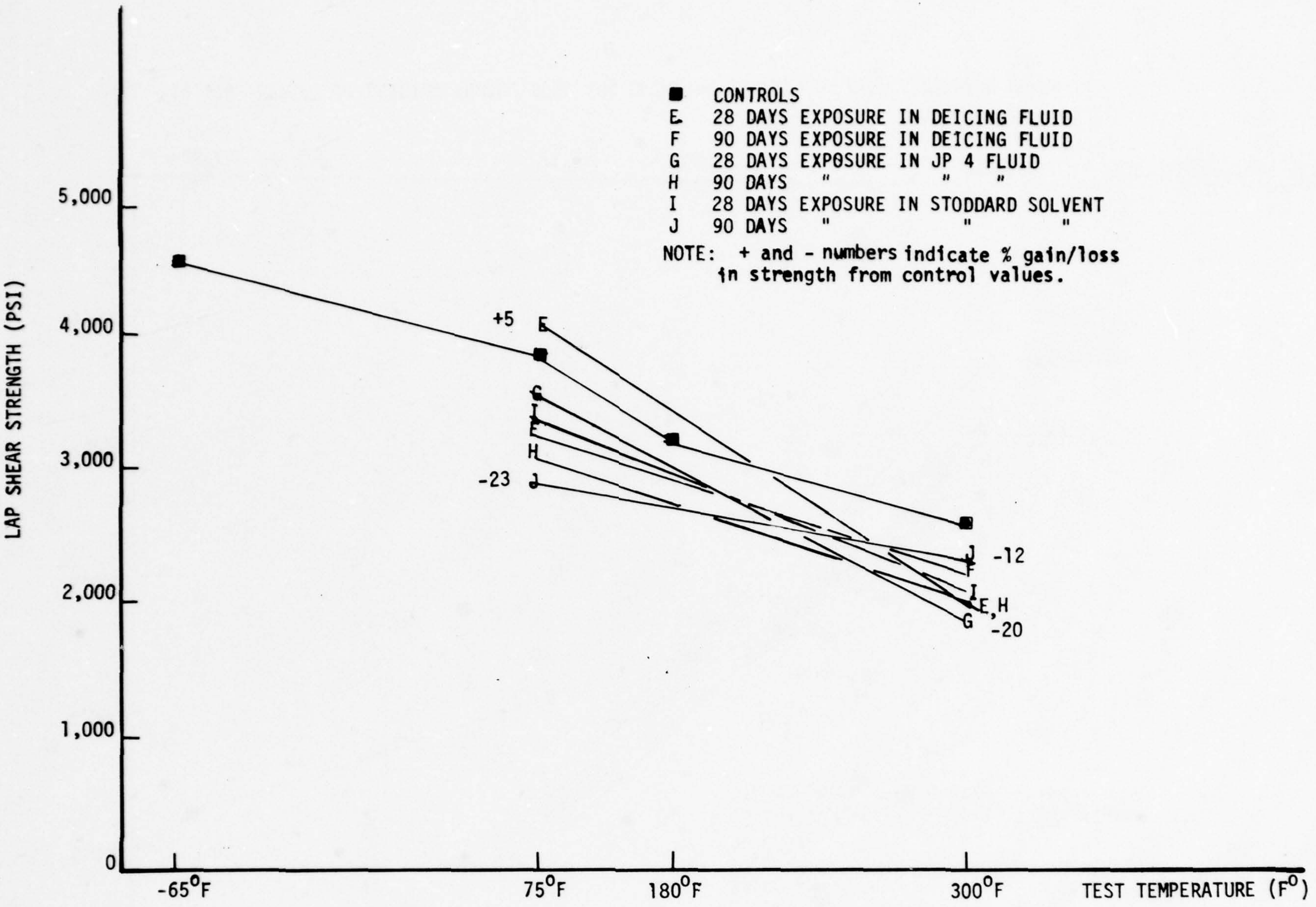


Fig. 17 EFFECT OF DEICING FLUID, FUEL AND CLEANING SOLVENT ON R-5000/TITANIUM BONDS

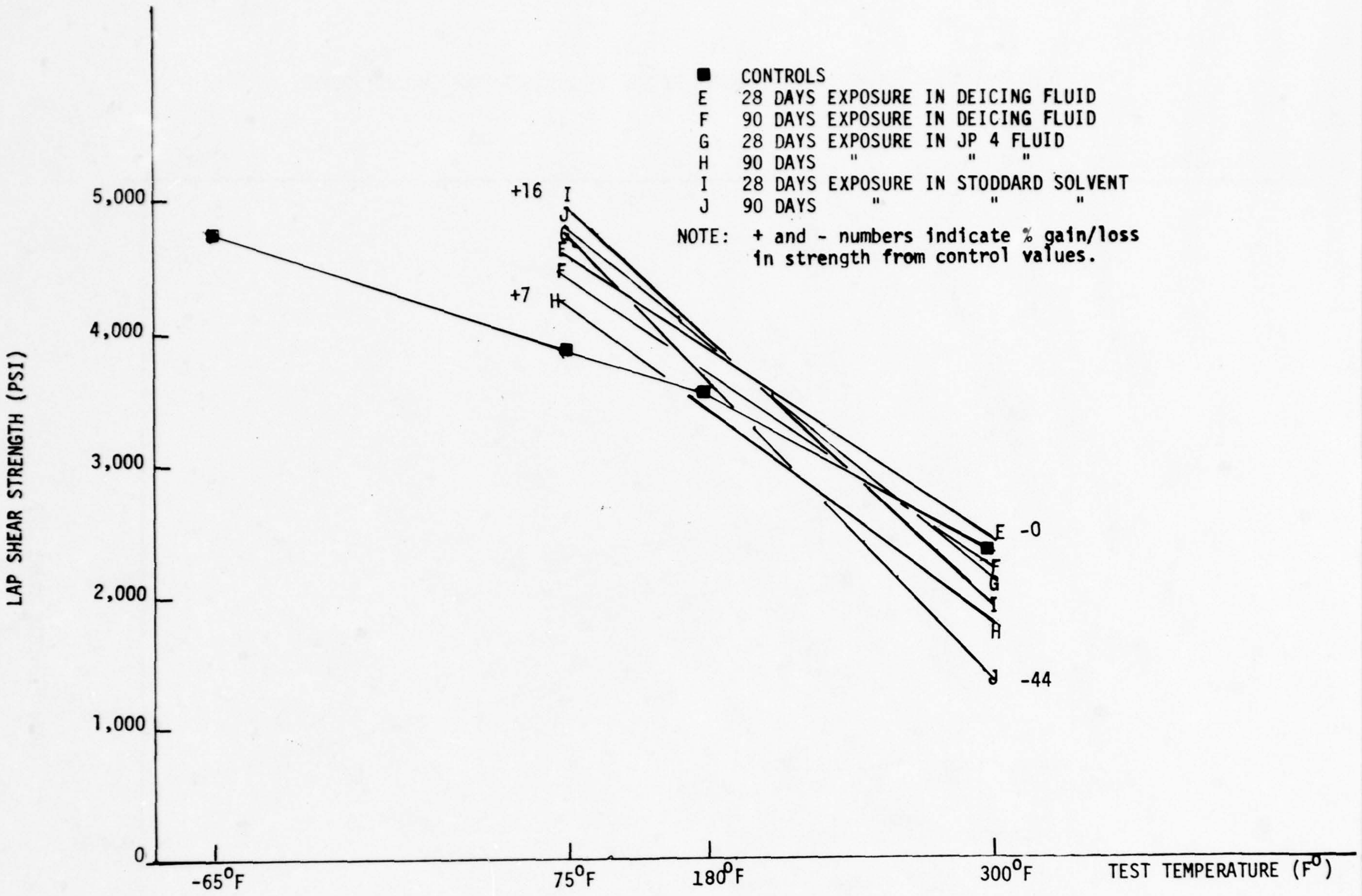


Fig. 18 EFFECT OF DEICING FLUID, FUEL AND CLEANING SOLVENT ON PKXA /TITANIUM BONDS

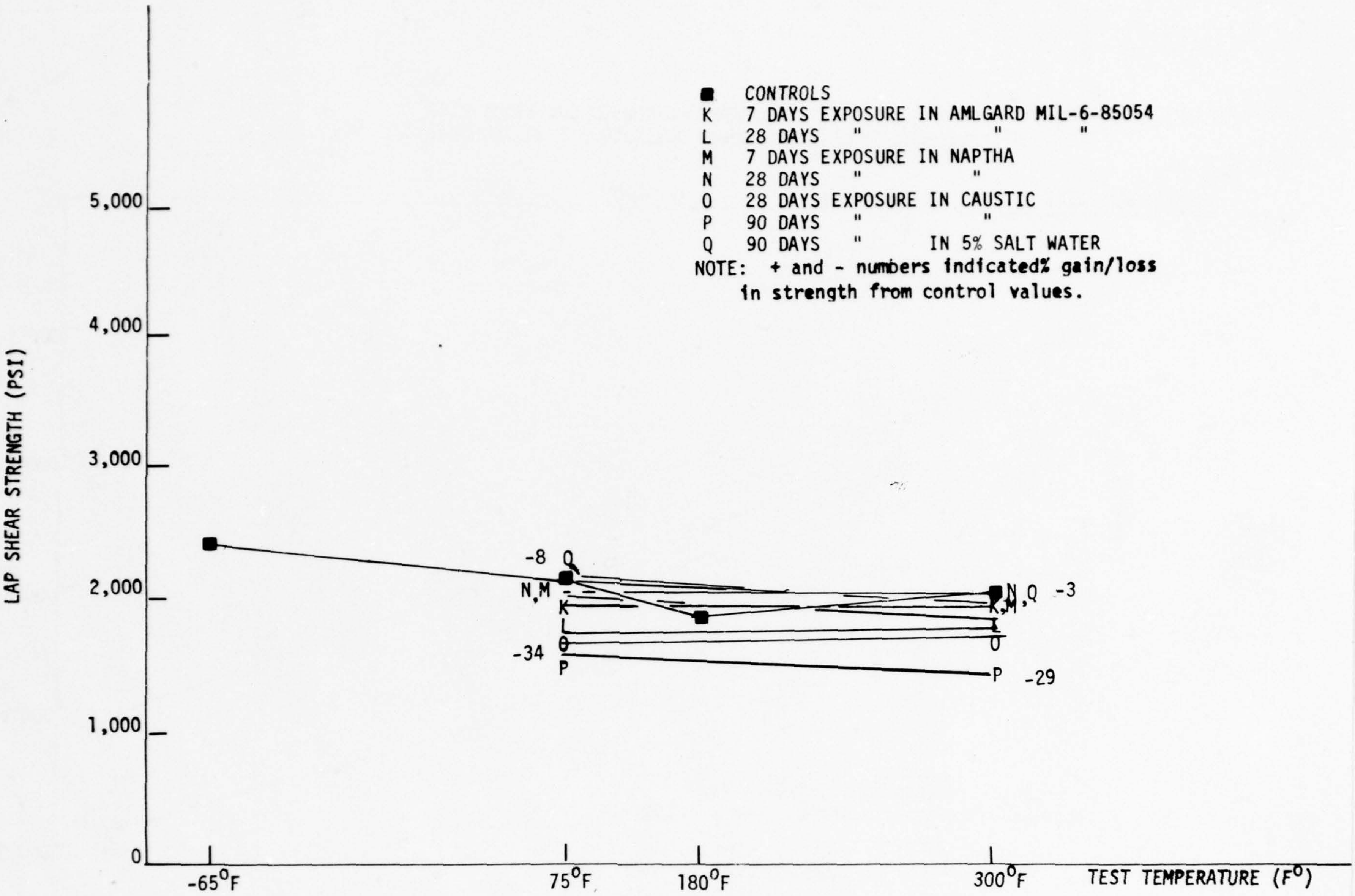


Fig. 19 EXPOSURE OF PKXA/COMPOSITE BONDS TO NAPHTHA, CAUSTIC, SALT WATER, AND CORROSION INHIBITING SOLUTION

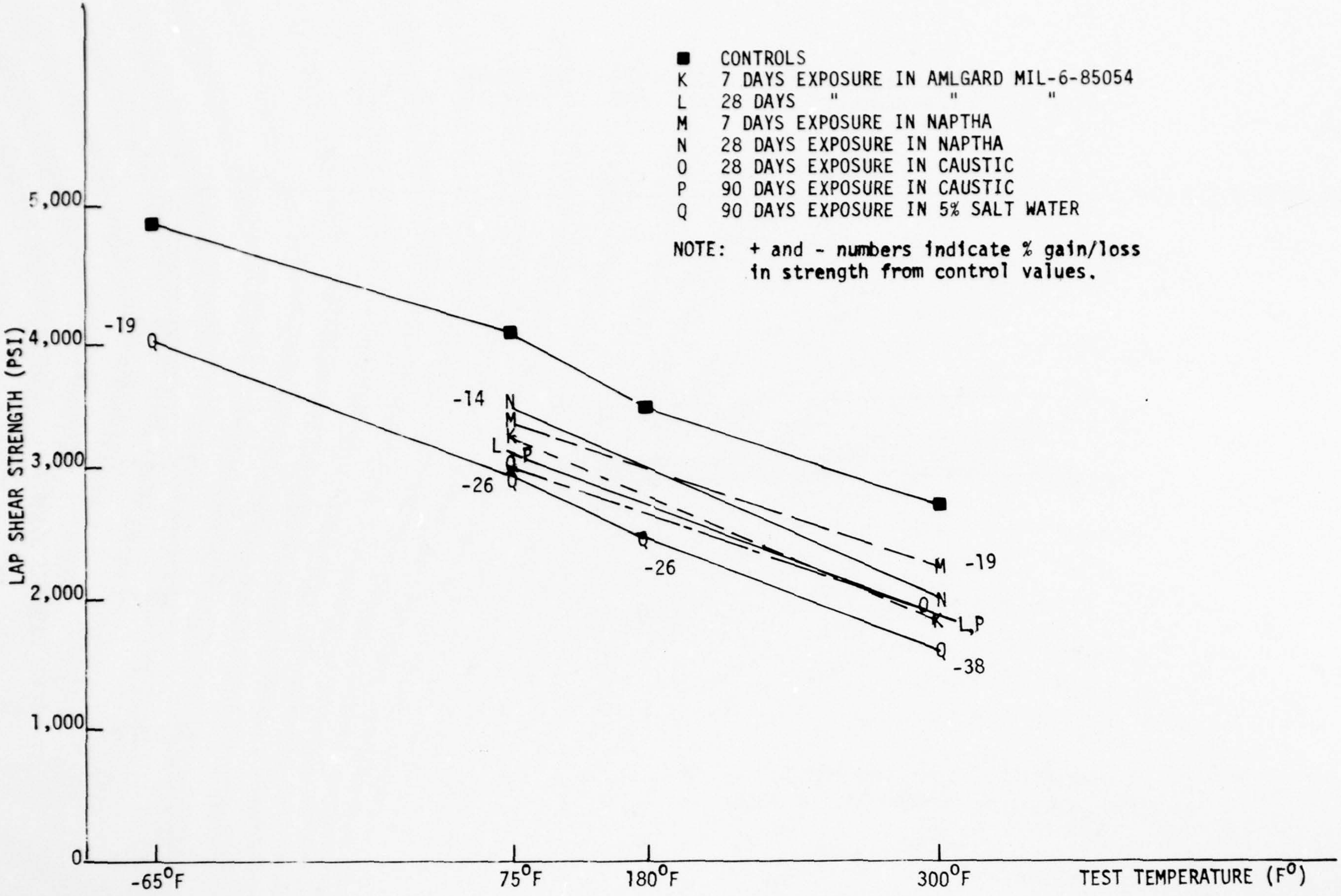


Fig. 20 EXPOSURE OF R-5000/STEEL BONDS TO NAPHTHA, CAUSTIC SALT WATER AND CORROSION INHIBITING SOLUTION

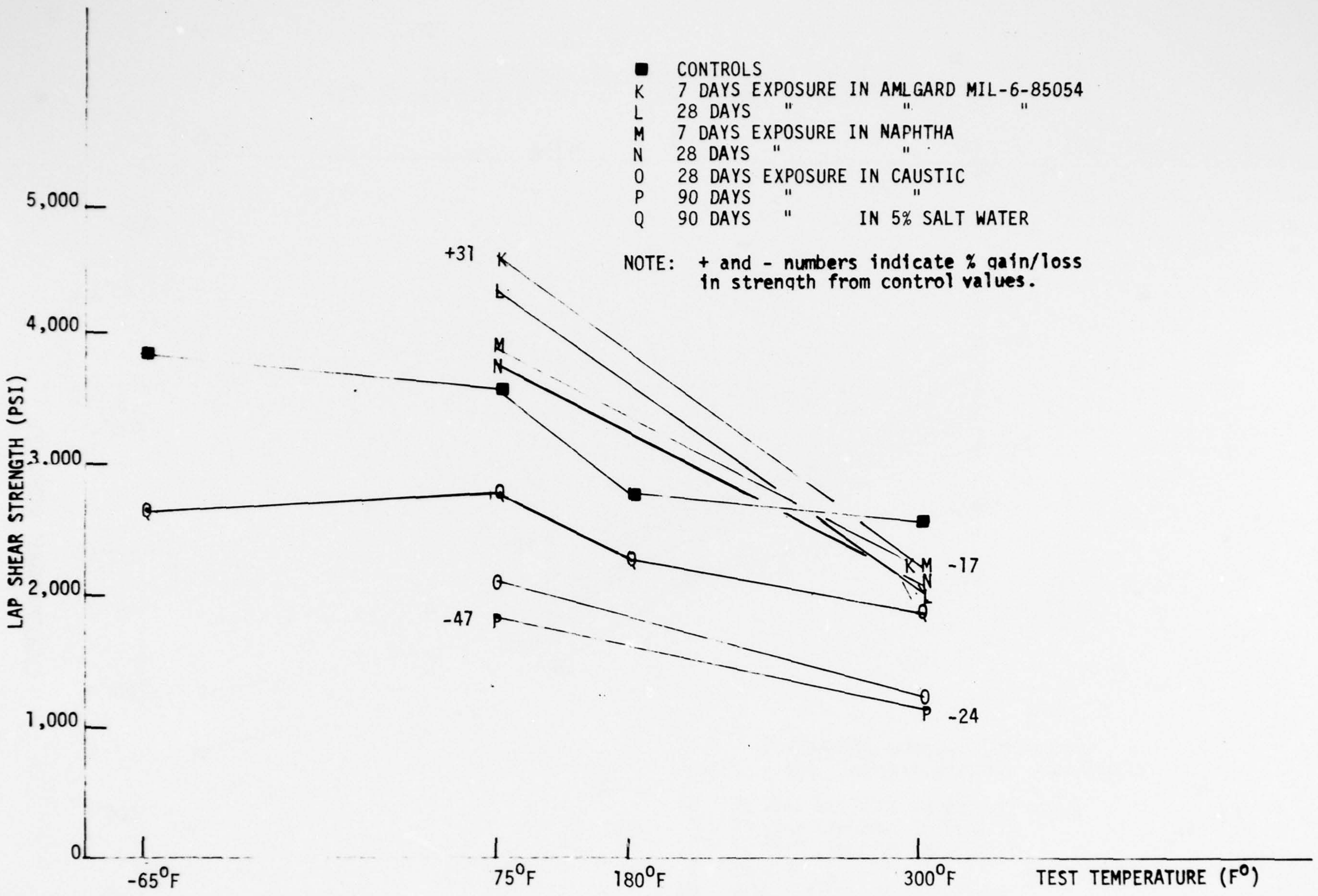


Fig. 21 EXPOSURE OF PKXA/STEEL BONDS TO NAPHTHA, CAUSTIC, SALT WATER, AND CORROSION INHIBITING SOLUTION

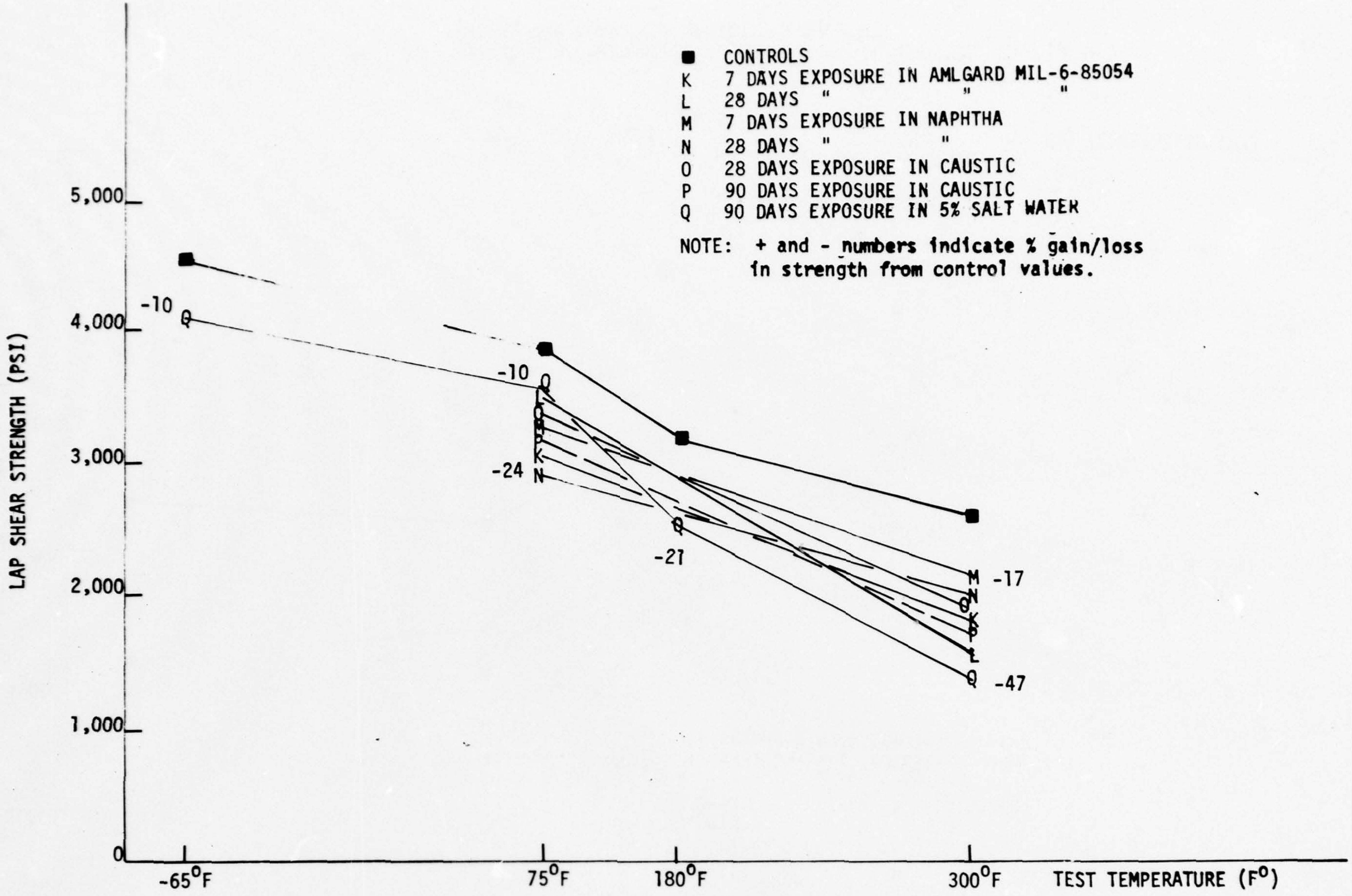


Fig. 22 EXPOSURE OF R-5000/TITANIUM BONDS TO NAPHTHA, CAUSTIC, SALT WATER AND CORROSION INHIBITING SOLUTION

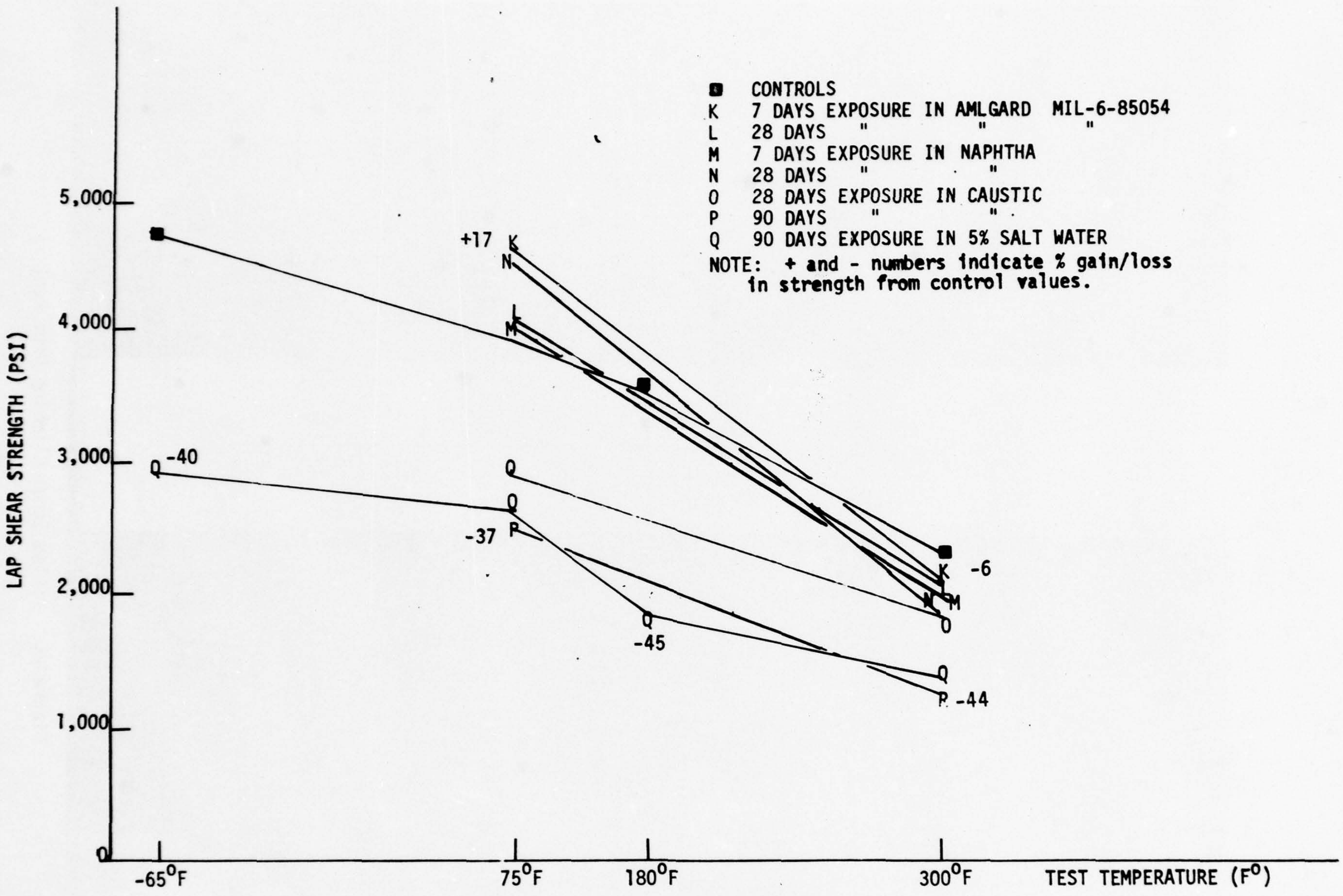


Fig. 23 EXPOSURE OF PKXA TITANIUM BONDS TO NAPHTHA, CAUSTIC, SALT WATER AND CORROSION INHIBITING SOLUTION

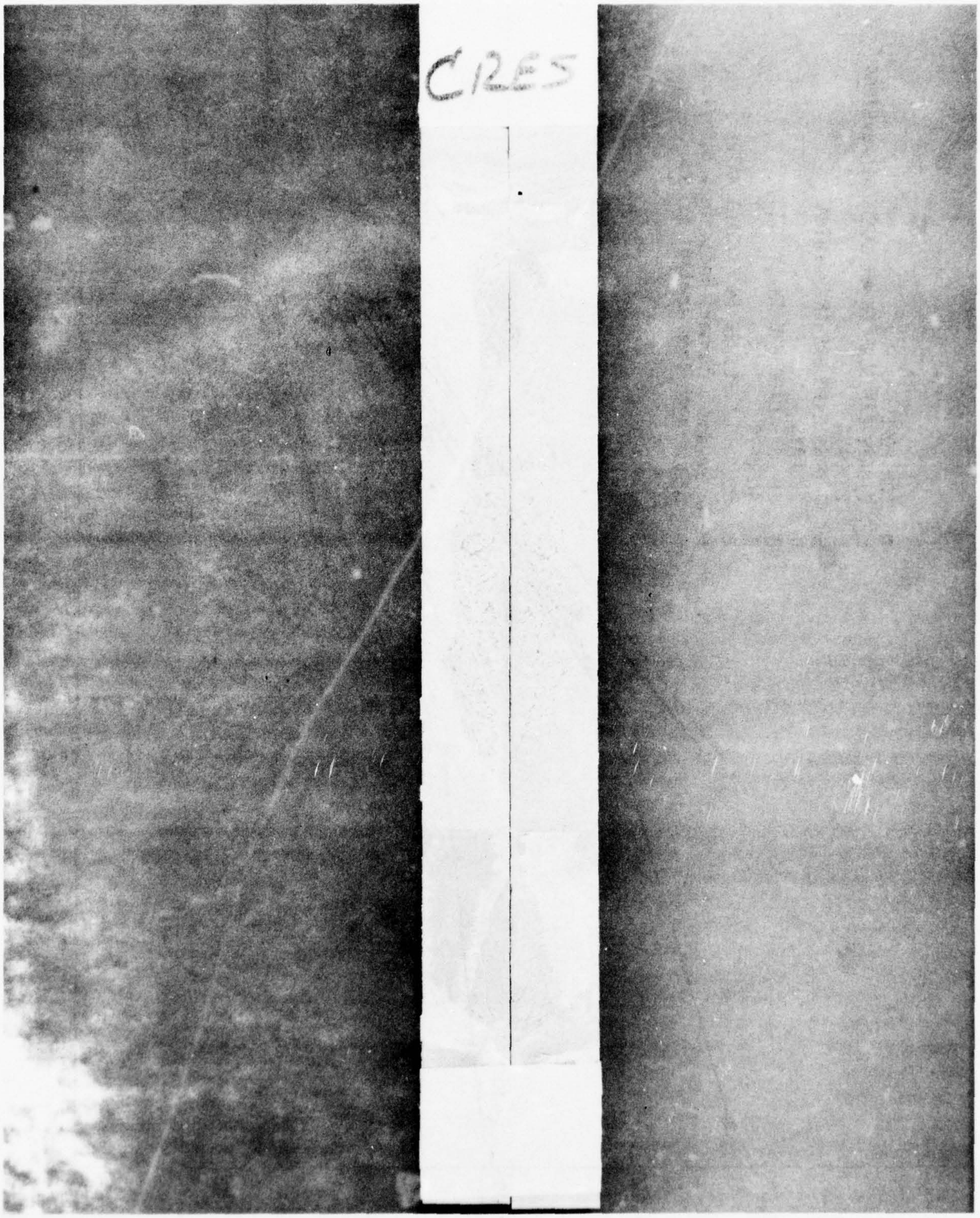


Figure 24: Failed Steel Climbing Drum Peel Specimen

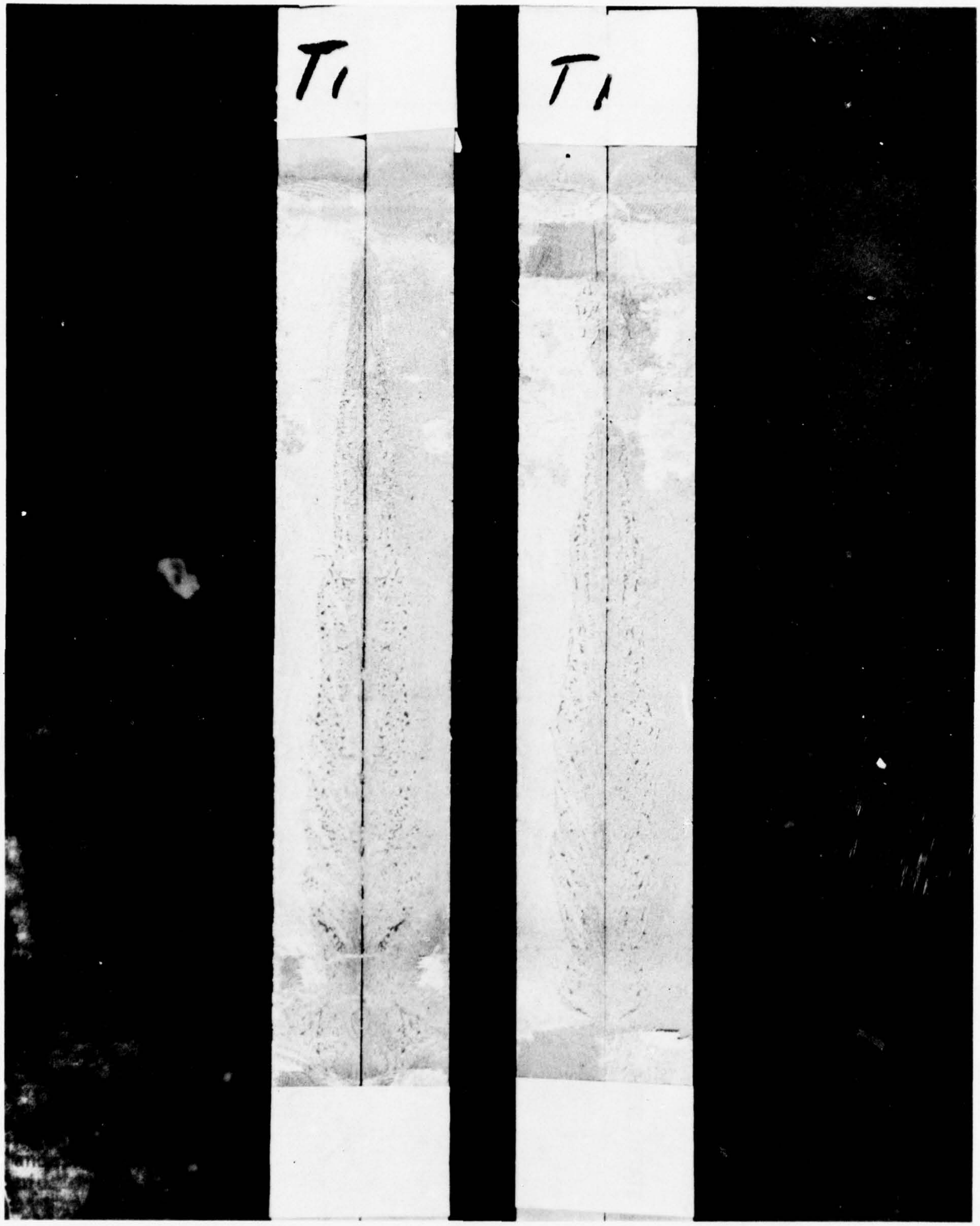


Figure 25: Failed Titanium Climbing Drum Peel Specimen

TABLE VII, TASK II: MECHANICAL PROPERTY TESTS

PROPERTY	TEST TEMP. (^oF)	LOAD (% Ult)	NO. OF SPECIMENS
PEEL STRENGTH	+70	-	2 per system
	+250	-	2 per system
STATIC CREEP	+70	[1]	53 Total
	+250		
	+70		
	+250		
	+70		
	+250		
FATIGUE LIFE	+70	30	3 per system
	+70	50	3 per system
	+70	80	3 per system

4 Systems: Steel + PKXA
Titanium + PKXA
Steel + R-5000
Titanium + R-5000

[1] Range 40 to 80%, See 2.2.2

TABLE VIII CLIMBING DRUM PEEL DATA

ADHEREND	ADHESIVE	PEEL STRENGTH (in-lbs/in width)	
		RT	250°
Steel	R-5000	39.4	39.0
Titanium	R-5000	37.3	33.1
Steel	PKXA	39.8	33.0
Titanium	PKXA	34.6	31.2

SYSTEM	TEST TEMP $^{\circ}$F	LOAD psi	CREEP in.	TIME hrs.	MMM-A-132 REQUIREMENT
					.015" max after 196 hrs. @ 1600 psi
PKXA/STEEL	RT	2390	.0113	485	↓
R-5000/STEEL	↓	2645	.0054	490	↓
PKXA/Ti	↓	3040	.0127	335	↓
R-5000/Ti	↓	2640	.0051	240	↓
PKXA/STEEL	250°F	1510	.0277	197	.015" max after 196 hrs. @ 800 psi and 180°F or 300°F
R-5000/STEEL	↓	1615	.0002	194	
PKXA/Ti	↓	1860	.0191	245	
R-5000/Ti	↓	2240	.0243	196	

TABLE IX: CREEP RESISTANCE

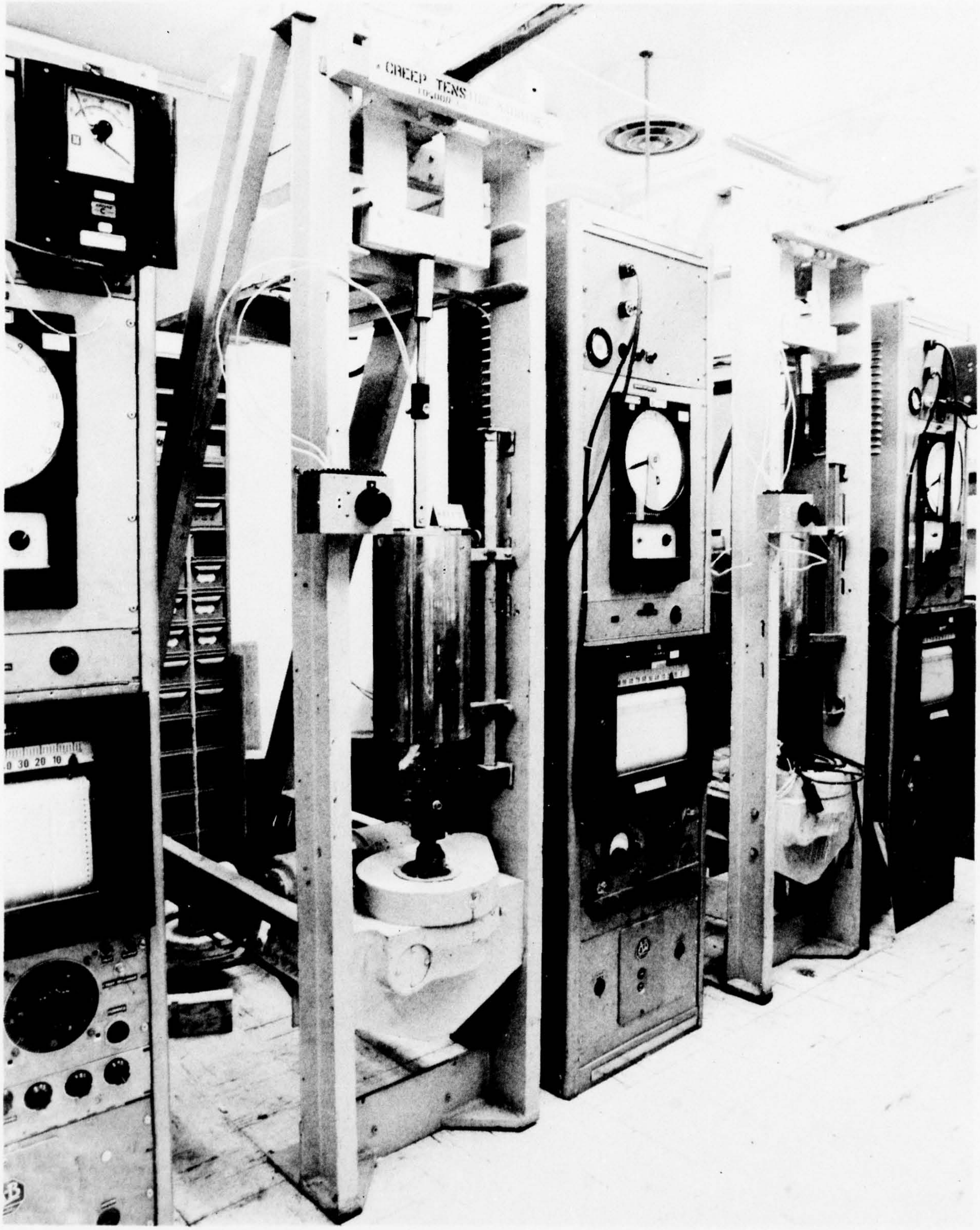


Figure 26: Creep Test Apparatus

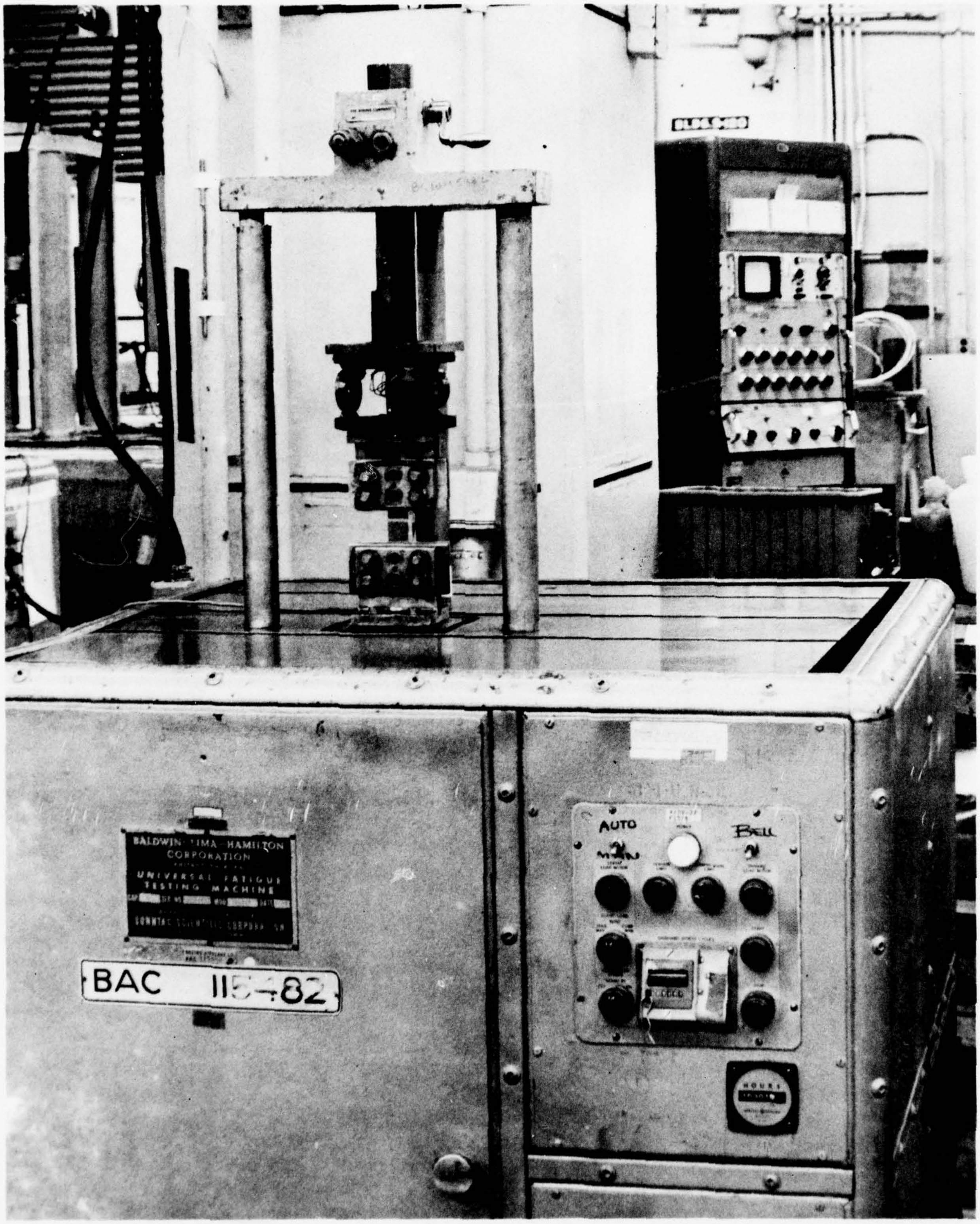


Figure 27: Fatigue Specimen in Test Apparatus

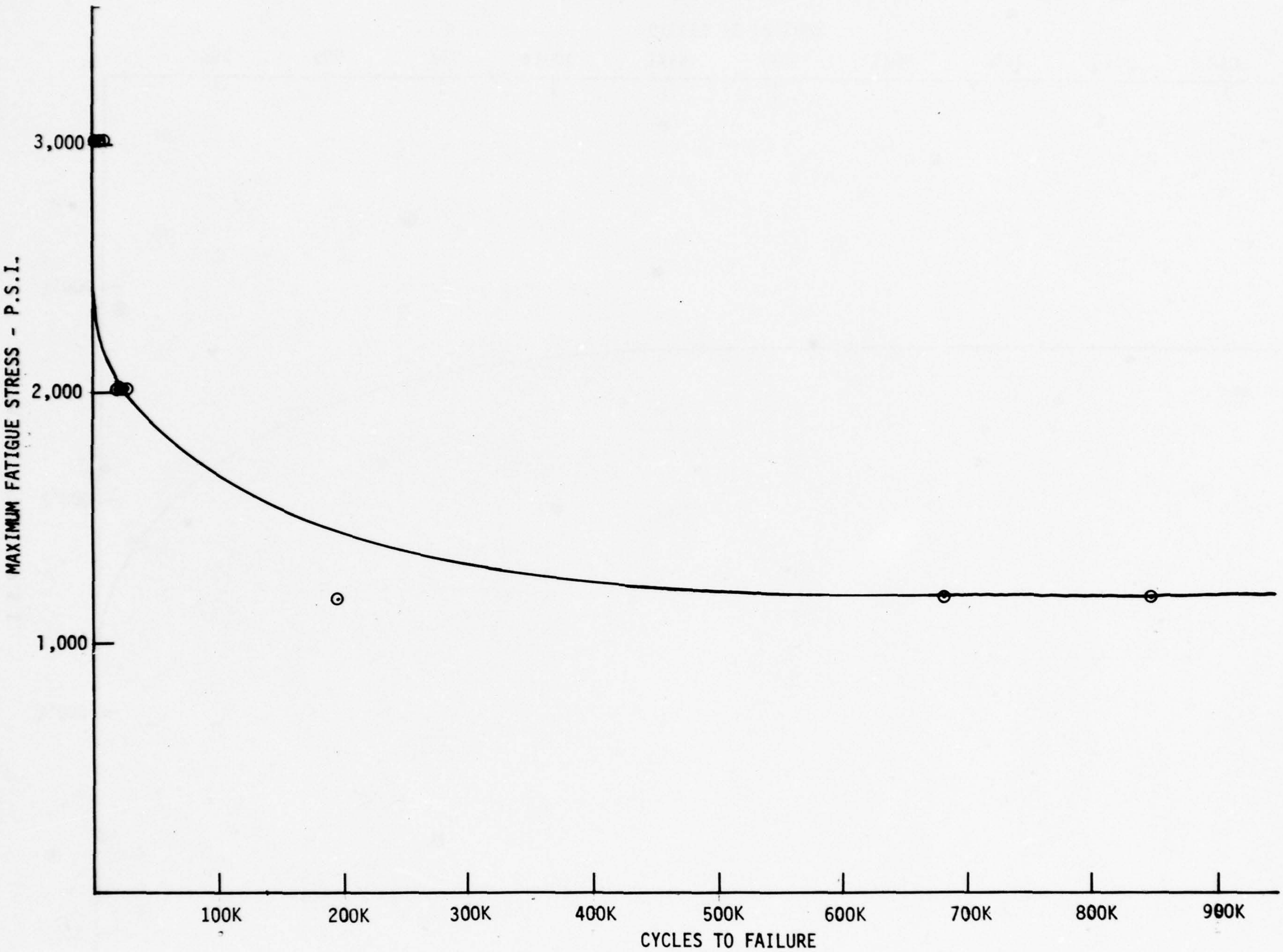


Fig. 28 STEEL/R-5000 FATIGUE DATA

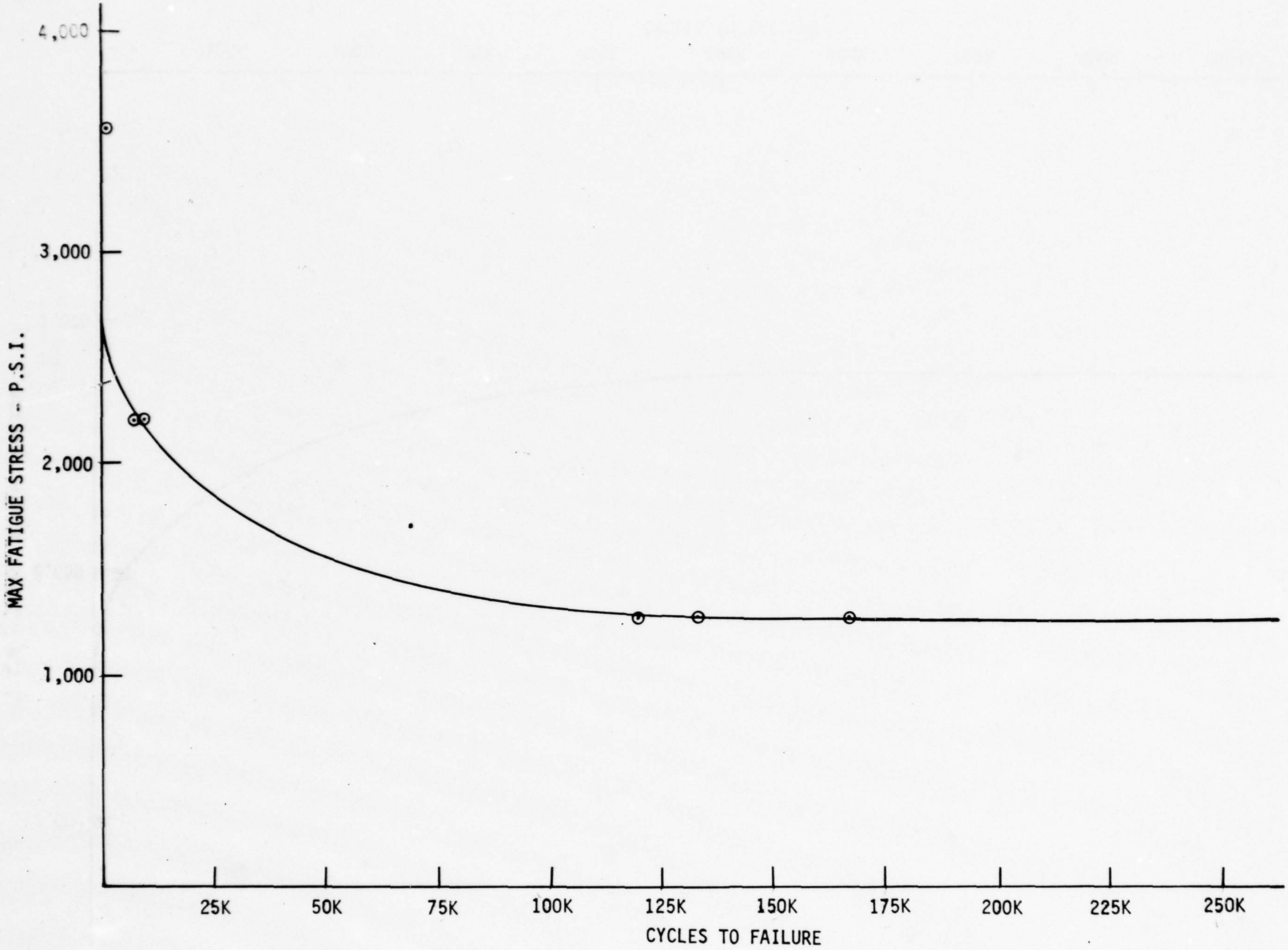


Fig. 29 TITANIUM/R-5000 FATIGUE DATA

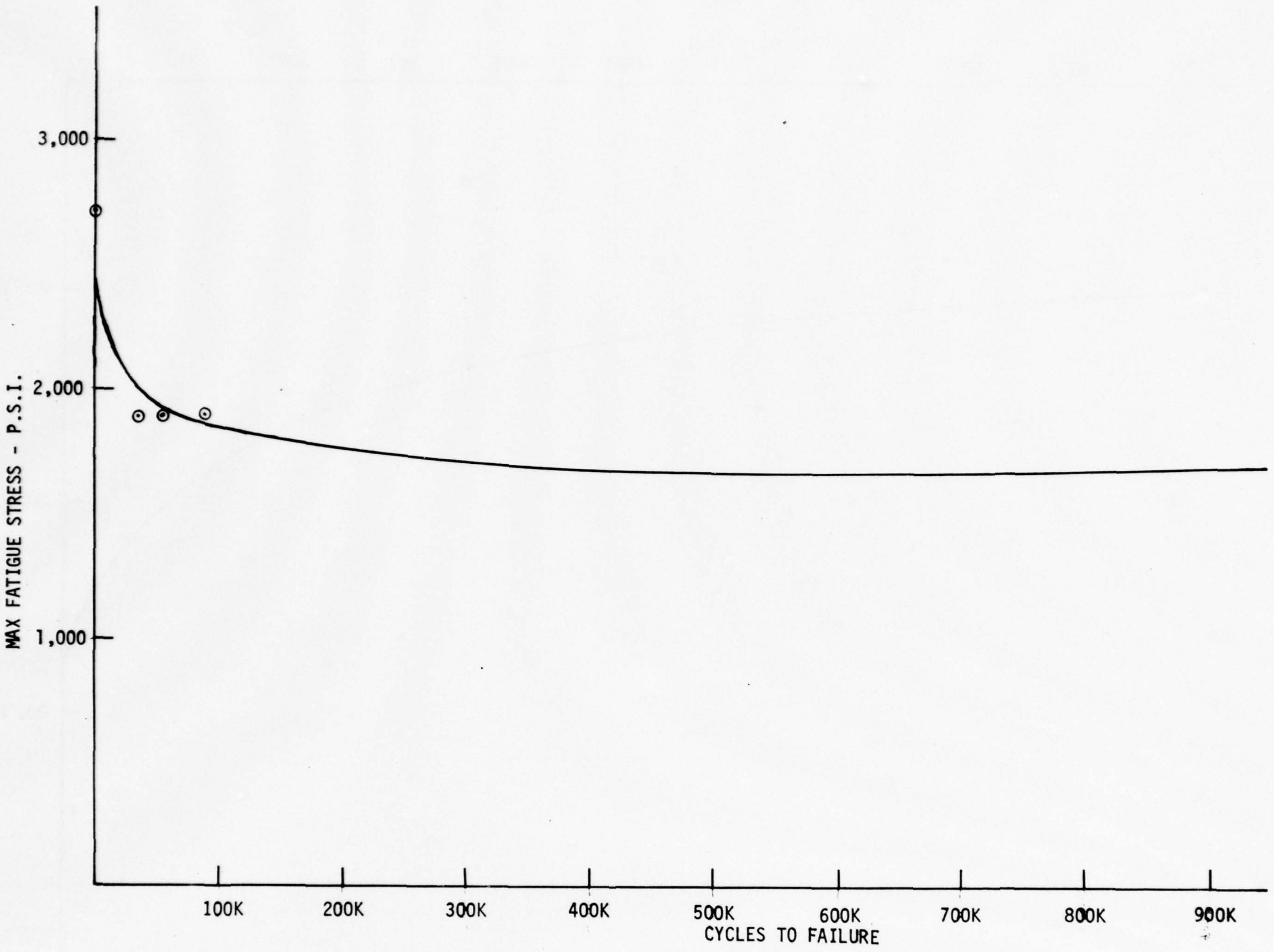


Fig. 30 STEEL/PKXA FATIGUE DATA

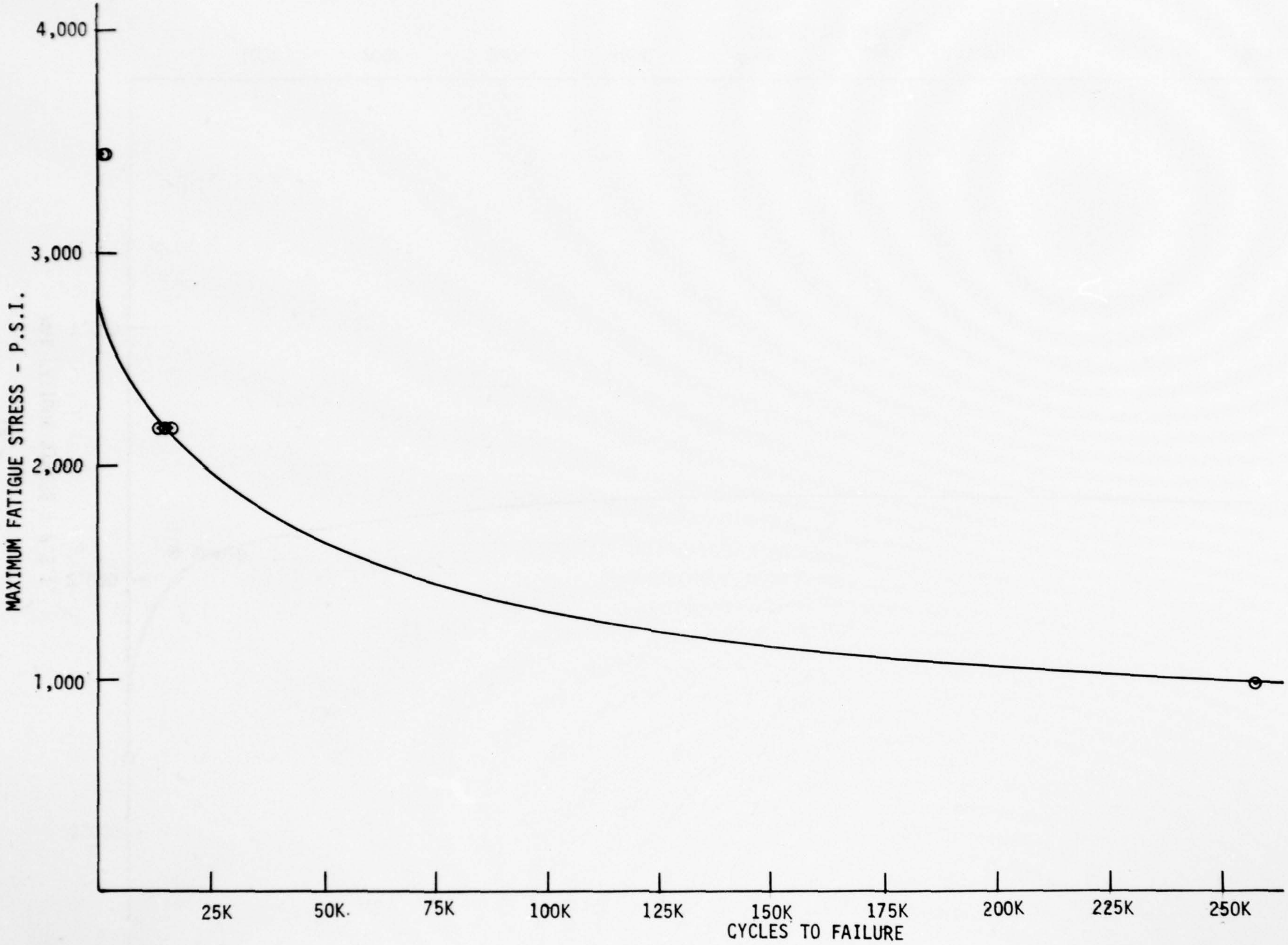


Fig. 31 TITANIUM/PKXA FATIGUE DATA

APPENDIX

TABLE AI: THE EFFECT OF ENVIRONMENTAL EXPOSURE ON STAINLESS STEEL/ R-5000 BONDED SPECIMENS

Environment	Exposure Time	Lap Shear Strength (psi)				Crack Extension Stressed @ RT	
		-65	RT	180^{0}F	300^{0}F	Initial Crack Length(in.)	Crack Growth (in.)
Control		4905	4010	3305	2685	1.44	None
Thermal Exposure, +180^{0}F	28 days 90 days	4960 4645	3700 4015	3105 3605	2510 3100	1.43 1.43	None None
Thermal Exposure, +300^{0}F	28 days 90 days	4685 4525	3845 4180	3365 3121	2775 2965	1.38 1.38	None None
Deicing Fluid (#146 ethylene glycol base)	28 days 90 days	-- --	4660 3395	-- --	3855 2665	1.44 1.44	None None
JP-4 Fuel	28 days 90 days	-- --	3050 3395	-- --	2035 2200	1.43 1.43	None None
Hydraulic Fluids (MIL-H-83306, Phosphate ester type) (MIL-H-5606, Standard Petroleum base)	90 days 7 days 28 days	-- -- --	890 3450 3165	-- -- --	120 1840 2155	1.47 1.43 1.43	Delaminated .26 .26
Synthetic Lubricant - Mil-L-7808	90 days	--	2765	--	1680	1.44	.44
Salt Water (NaCl 5%)	90 days	3985	2965	2430	1660	1.41	.47
Cleaning Solvent, Organic (Stoddard)	28 days 90 days	-- --	3255 3385	-- --	2090 2445	1.39	.37 .37
Amlgard Mil-C-85054	7 days 28 days	-- --	3340 3220	-- --	1815 2085	1.48	.38 .38
Naphtha	7 days 28 days	-- --	3300 3445	-- --	2165 1945	1.44 1.41	.18 .23
Caustic ph9 (Na OH)	28 days 90 days	-- --	3010 3025	-- --	1950 1910	1.47	.41 .45

TABLE A2: THE EFFECT OF ENVIRONMENTAL EXPOSURE ON TITANIUM/R-5000 BONDED SPECIMENS

Environment	Exposure Time	Lap Shear Strength (psi) -65	RT	180^{O}F	300^{O}F	Crack Extension Stressed @ RT Initial Crack Length(in.)	Crack Growth (in.)
Control		4605	3880	3395	2630	1.39	None
Thermal Exposure, +180^{O}F	28 Days	4375	3825	3315	2405	1.47	None
	90 Days	4825	4160	3435	3040	1.47	None
Thermal Exposure, +300^{O}F	28 Days	4205	2825	3040	2435	1.34	None
	90 Days	3940	3545	2980	2885	1.34	None
Deicing Fluid (#146 ethylene glycol base)	28 Days	-	4060	-	2060	1.39	None
	90 Days	-	3325	-	2110	1.39	None
JP-4 Fuel	28 Days	-	3565	-	2095	1.43	None
	90 Days	-	3150	-	2095	1.43	None
Hydraulic Fluids (MIL-H-83306 phosphate ester type)	90 Days	-	1340 66	-	350 87%	1.48	Delaminated
MIL-H-5606 Standard Petroleum base)	7 Days	-	3170 18%	-	1975 25%	1.53	None
	28 Days	-	2805 28%	-	1830 30%	1.53	.11
Synthetic Lubricant (MIL-L-7808)	90 Days	-	3020 22	-	1805 31%	1.53	.27
Salt Water (NaCl 5%)	90 Days	4175	3495	2675	1400	1.48	.43
Cleaning Solvent, Organic (Stoddard)	28 Days	-	3465	-	2005	1.46	.21
	90 Days	-	2985	-	2315	1.46	None
Amlgard MIL-C-85054	7 Days	-	3080	-	1775	1.46	.39
	28 Days	-	3530	-	1735	1.46	.41
Naphtha	7 Days	-	3075	-	2190	1.39	.18
	28 Days	-	2945	-	2075	1.39	.19
Caustic, ph9 (Na OH)	28 Days	-	3455	-	1995	1.44	.39
	90 Days	-	3185	-	1770	1.44	.42

TABLE A3: THE EFFECT OF ENVIRONMENTAL EXPOSURE ON TITANIUM/PKXA BONDED SPECIMENS

Environment	Exposure Time	Lap Shear Strength (psi)				Crack Extension Stressed @ RT	
		-65	RT	180°F	300°F	Initial Crack Length (in.)	Crack Growth (in.)
Control		4870	3985	3515	2345	1.47	None
Thermal Exposure, +180°F	28 Days	4215	3935	4235	2565		
	90 Days	5115	4520	3800	2545	1.43	None
Thermal Exposure, +300°F	28 Days	4565	3965	3635	2545		
	90 Days	3675	4220	3885	2935	1.49	None
Deicing Fluid	28 Days	-	4560	-	2365	1.44	None
(#146 ethylene glycol base)	90 Days	-	4495	-	2305	1.51	None
JP-4 Fuel	28 Days	-	4615	-	2075	1.41	None
	90 Days	-	4275	-	1780	1.47	None
Hydraulic Fluids							
(MIL-H-83306, Phosphate ester type)	90 Days	-	*4560	-	*1830	1.49	.24
(MIL-H-5606, Standard Petroleum base)	7 Days	-	4215	-	2425	1.43	None
	28 Days	-	4595	-	2030	1.41	None
Synthetic Lubricant (MIL-L-7808)	90 Days	-	4675	-	2330	1.44	None
Salt Water (NaCl 5%)	90 Days	2900	2635	1950	1430	1.47	.31
Cleaning Solvent, Organic	28 Days	-	4620	-	2015	1.44	.07
(Stoddard)	90 Days	-	4765	-	1330	1.48	.32
Amlgard MIL-C-85054	7 Days	-	4665	-	2215	1.42	None
	28 Days	-	4255	-	2145	1.44	None
Naphtha	7 Days	-	4025	-	2015	1.42	None
	28 Days	-	4525	-	1995	1.48	.06
Caustic, ph9 (Na OH)	28 Days	-	2960	-	1980	1.49	.05
	90 Days	-	2520	-	1315	1.53	.38

* PKXA 517 Adhesive

TABLE A4: THE EFFECT OF ENVIRONMENTAL EXPOSURE ON STEEL/PKXA BONDED SPECIMENS

Environment	Exposure Time	Lap Shear Strength (psi)				Crack Extension Stressed @ RT	
		-65	RT	180°F	300°F	Initial Crack Length(in.)	Crack Growth (in.)
Control		3865	3542	2825	2585	1.47	None
Thermal Exposure, +180°F	28 Days	3265	3000	2925	2665	1.42	None
		*4645	*4015	*3605	*3100		
	90 Days	3595	3480	3100	3195	1.45	None
Thermal Exposure, +300°D	28 Days	3530	3335	2705	2610	1.48	None
		*4525	*4180	*3121	*2865		
	90 Days	3765	3060	2865	2715	1.46	.03
Deicing Fluid (#146 ethylene glycol base)	28 Days	-	3905	-	3505	1.44	None
	90 Days	-	3155	-	2230	1.41	None
JP-4 Fuel	28 Days	-	3600	-	1715	1.44	.08
	90 Days	-	3720	-	2290	1.48	None
Hydraulic Fluids							
(MIL-H-83306, Phosphate ester type)	90 Days	-	2975	-	2410	1.49	.03
(MIL-H-5606, Standard Petroleum base)	7 Days	-	3785	-	2430	1.42	None
	28 Days	-	3745	-	2390	1.45	None
Synthetic Lubricant (MIL-L-7808)	90 Days	-	3460	-	2235	1.41	None
Salt Water (NaCl 5%)	90 Days	2640	2725	2332	1970	1.47	None
Cleaning Solvent, Organic (Stoddard)	28 Days	-	3365	-	2150	1.42	None
	90 Days	-	3365	-	2080	1.45	.04
Amlgard MIL-C-85054	7 Days	-	*4665	-	2215	1.48	None
	28 Days	-	4440	-	2085	1.42	None
Naptha	7 Days	-	3960	-	2145	1.50	.03
	28 Days	-	3870	-	2055	1.51	.05
Caustic, ph9 (Na OH)	28 Days	-	2165	-	1385	1.47	.32
	90 Days	-	1880	-	1330	1.42	.37

* PKXA .517 Adhesive

TABLE A5: THE EFFECT OF ENVIRONMENTAL EXPOSURE ON COMPOSITE/PKXA BONDED SPECIMENS

Environment	Exposure Time	Lap Shear Strength (psi)				
		-65	RT	180°F	300°F	
Control		2440	2315	1905	2050	
Thermal Exposure, +180°F	28 Days 90 Days	1680 1955	1800 1795	1450 1700	1970 2045	
Thermal Exposure, +300°F	28 Days 90 Days	1725 1735	1705 1570	1665 1535	1605 1740	
Deicing Fluid (#146 ethylene glycol base)	28 Days 90 Days	- -	1920 1825	- -	1790 1765	
JP-4 Fuel	28 Days 90 Days	- -	1850 2330	- -	1850 1940	
Hydraulic Fluids (MIL-H-83306, Phosphate ester type) (MIL-H-5606, Standard Petroleum base)	90 Days 7 Days 28 Days	- - -	1865 1915 2140	- - -	1970 2060 1945	
Synthetic Lubricant (MIL-L-7808)	90 Days	-	1775	-	1785	
Salt Water (NaCl 5%)	90 Days	-	2140	-	1985	
Cleaning Solvent, Organic (Stoddard)	28 Days 90 Days	- -	1815 1730	- -	1895 1655	
Amlgard MIL-C-85054	7 Days 28 Days	- -	1985 1760	- -	1955 1790	
Naphtha	7 Days 28 Days	- -	2115 2005	- -	1940 2010	
Caustic, pH 9 (Na OH)	28 Days 90 Days	-	1650 1540	-	1710 1480	

Table A6: PKXA/Steel Creep Data

TEST TEMP. (^oF)	% Ult. Load	Load (lbs)	Total Hours	Creep (in)
RT	50	1560	84	[1]
	60	1872	443	.0016
	60	1872	133	[1]
	70	2394	484	.0113
	70	2394	484	.0084
	70	2394	28	[1]
	80	2848	(30 sec.)	[1]
↓	80	2848	6	.0356
RT	80	2848	6	.0005
250	40	1512	197	.0277
	54	2042	21	.0185
	54	2042	21	[1]
	60	2268	110	[1]
	60	2268	0	[1]
↓	70	2646	3	[1]
250	70	2646	5	[1]

[1] **Failed after time indicated.**

Table A7: R-5000/Steel Creep Data

TEST TEMP. (^{o}F)	% Ult. Load	Load (lbs)	Total Hours	Creep (in)
RT	40	1400	196	.0132
	55	1750	216	.0083
	60	2268	234	.0071
	60	2268	234	.0027
	60	2268	234	.0026
	70	2646	491	.0054
	70	2646	491	[1]
	70	2646	491	.0001
	74	2798	.6	[1]
RT	80	3024	0	[1]
250	40	1616	194	.0002
	60	2424	44	.0001
250	60	2424	44	[1]

[1] Failed after time indicated.

Table A8: PKXA/Titanium Creep Data

TEST TEMP. (^{o}F)	% Ult. Load	Load (lbs)	Total Hours	Creep (in)
RT	40	1738	192	.0176
	50	2170	217	.0218
	60	1860	11	▷1
	60	1860	499	.020
	60	1860	64	▷1
	70	3038	335	.0127
	70	2940	0	▷1
	80	3360	0	▷1
RT	80	3360	0	▷1
250	60	1860	243	.0191
250	60	1860	243	.0173

▷1 **Failed after time indicated.**

Table A9: R-5000/Titanium Creep Data

TEST TEMP. (^{o}F)	% Ult. Load	Load (lbs)	Total Hours	Creep (in)
RT	40	1760	196	.0084
↓	50	2120	217	.0065
↓	60	2640	239	.0023
↓	60	2640	239	.0008
↓	60	2640	239	.0051
↓	70	3080	17	[1]
RT	70	3080	447	[1]
250	50	2240	196	.0243
↓	50	2240	196	.0021
↓	60	2688	410	[1]
↓	60	2688	41	[1]
↓	66	2956	.05	[1]
250	66	2956	.03	[1]

[1] Failed after time indicated.

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Two thermoplastic polymers (PKXA Polysulfone and R-5000 polyphenylsulfone) having high temperature capabilities (+300°F) were evaluated as adhesives for bonding steel and titanium adherends. The PKXA polysulfone adhesive was also evaluated on polyimide/glass composites. Chromic acid anodize and sulfuric acid anodize were selected as the surface treatments for titanium and steel, respectfully. The primer system selected was BR-34 polyimide primer (American Cyanimid) subsequently coated with a thin solution of the bonding resin. Prior to bonding, the thermoplastic resins were solution coated on 112E glass with A-1100 finished for bondline control.

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20. The environmental resistance of the PKXA and R-5000 adhesives were evaluated after exposure to thermal environments (180°, 300°F), deicing fluid, JP-4 fuel, hydraulic fluids (MIL-H-83306, MIL-H-5606), synthetic lubricant (MIL-L-7808), salt water, cleaning solvent (Stoddard), Naphtha, corrosion inhibitor MIL-C-85054, Naphtha, and caustic (NaOH) solutions. Lap shear and crack extension specimens were used to evaluate the environmental resistance of the adhesive systems. Peel, creep and fatigue properties were determined for the adhesives on steel and titanium adherends.