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Development of Thermoplastic Structural Adhesives.

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By

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S. G./Hill
J. T./Hoggatt

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FOREWORD

This report covers the work conducted by the Boeing Aerospace Company during the period of 5 November 1975 to 1 March 1977 for the Naval Air Systems Command, United States Department of the Navy, under Contract N00019-76-C-0170, entitled "Development of Thermoplastic Structural Adhesives." Mr. J. J. Gurtowski (AIR 52032C) was Program Monitor. *new*

This program was conducted by Boeing Military Airplane Development Organization of the Boeing Aerospace Company, Seattle, Washington. Mr. J. T. Hoggatt was Program Manager and Mr. S. G. Hill was Technical Leader. Mr. R. Hodges and Ms V. Monroe were major contributors to the program.

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1.0 INTRODUCTION AND SUMMARY

Structural adhesive bonding is one of the most highly desirable methods of joining similar and dissimilar materials for aerospace structures in terms of manufacturing cost and structural efficiency. Under previous contracts (References 1 and 2) it was demonstrated that thermoplastic materials were suitable for adhesives and were highly desirable in areas where post-forming of parts was required. Thermoplastic materials are cheaper than epoxies, have shorter processing times, and can be post-formed. The intent of this program was to select and evaluate some thermoplastic materials as potential adhesives; to explore different surface treatments for bonding stainless steel, aluminum, and titanium metals; to establish processing parameters for bonding with thermoplastics; to modify thermoplastic materials for adhesives; and to evaluate thermoplastic adhesives in environmental conditions under stress.

Phase I of this program was devoted to materials selection. Under this phase of the program, an industry survey was made of existing thermoplastic resins and composites having elevated temperature capabilities (+300°F service). Six different classes of thermoplastic polymers were selected for investigation. They were: (1) the polysulfones, (2) polyether sulfones, (3) polyarylsulfones, (4) polyimides, (5) polyphenylene sulfides, and (6) amides-imides. After studying the available data, five polymers from three classes were selected for material screening. The initial screening was conducted on two polysulfones (PKXA and P-1700, manufactured by Union Carbide Corporation), one polyphenylene sulfide (Ryton V-1, manufactured by Phillips Petroleum Corp.), and two polyimides (NR150A2 and 2080D, manufactured by E. I. DuPont and the Upjohn Corp., respectively). In the initial screening, lap shear specimens were fabricated using aluminum or titanium adherends and tested at -65°F, RT, and elevated temperatures. The polyphenylene sulfide material was eliminated because it was brittle. Subsequently, polyether sulfone (100P) replaced the polyphenylene sulfide.

The objectives of the second phase of this program were as follows: (1) to evaluate three different surface preparation's techniques for each metal substrate and select the best surface preparation technique for each substrate,

(2) to select the most economical bonding process for each material, one that did not require a sacrifice in bond strength, and (3) to modify an adhesive system to improve the adhesive quality. Under this phase of the program, PKXA adhesive system was modified with aluminum powder, amorphous boron, scrim cloths, and polyphenylene sulfide. Lap shear specimens were fabricated and tested. Test results indicated that the modifications evaluated did not improve the adhesive quality. The scrim cloth helped to control the flow of material in the bond area but the other modifications resulted in a trade-off of either ambient or elevated temperature properties. The best all-around properties were obtained from specimens bonded with unmodified adhesives on scrim cloth.

The last phase of the program was devoted to evaluating PKXA and NR150A2 adhesives under stress in different environments. The test method selected for this phase of the program was the double cantilever beam specimen. Specimens were fabricated from the three different adherend materials and the selected adhesives. The specimens were stressed and exposed to salt spray, humidity, fluid, and thermal environments. Test results show that the surfaces and/or adhesives need more work to improve the reliability of the thermoplastic bonds; however, good structural bonds can be obtained with aluminum, titanium and stainless steel adherends.

2.0 STUDY PROGRAM

The objectives of this program were: (1) to assess the potential of using high temperature thermoplastic resins as structural adhesives in metal-to-metal bonded joints, (2) to explore the effects of surface treatments of metallic substrates and processing parameters on bond strengths and failure modes of selected adhesives, and (3) to assess the effects of environmental exposures on the bond stability of adhesive/surface treatment combinations. The program was divided into three principal areas: Phase I - Materials Selection; Phase II - Processing Study; and Phase III - Environmental Exposure.

2.1 PHASE I - MATERIALS SELECTION

In Phase I, six thermoplastic resins were evaluated as metal-to-metal adhesives with high temperature capabilities. Based on lap shear strength, two resins (PKXA and NR150A2) were selected for further study.

2.1.1 Candidate Selection

An industry survey was made of existing data on thermoplastic resins and composites having elevated temperature capabilities of +300°F. A total of six different classes of thermoplastic polymers were surveyed. They were: (1) polysulfones, (2) polyether sulfones, (3) polyarylsulfones, (4) polyimides, (5) polyphenylene sulfides, and (6) amide-imides. The material class, manufacturer, and the manufacturer's designations of the surveyed materials are shown in Table I. After studying the available data, five polymers from the three classes of materials were selected for further evaluation. Those classes were polysulfone, polyphenylene sulfide, and polyimides (see Table I).

2.1.2 Adherend Surface Preparation

The surface preparations for aluminum, steel, and titanium were phosphoric acid anodize, sulfuric acid anodize, and chromic acid anodize, respectively. The anodized panels were primed with either a dilute solution of the resin systems (polyimides) or BR-127 (epoxy) corrosion-inhibiting primer from American Cyanamid Company. The epoxy primer was used for all adhesives except

the polyimides. Work under previous Navy contracts (References 1 and 2) showed that BR-127 epoxy primer could withstand short periods of time at 500 - 600°F without significant degradation of the epoxy primer. The epoxy primer was sprayed to a thickness of .2 to .4 mils, air dried for 30 minutes, and cured one hour at $260 \pm 5^\circ\text{F}$ in an air-circulating oven. The NR150A2 and 2080D polyimide resins were diluted to 10% resin solids with ethyl alcohol and DMF, respectively. The NR150A2 solution was brushed on the adherend faying surface, air dried for two hours, and then forced air dried in an oven at 500°F for one hour. The 2080D polyimide was brushed on the adherends in an oven at 110°F to 115°F to prevent the pick-up of moisture on the adhesive. After the primed panels remained at 110 - 115°F for two hours, the temperature was raised to 550°F for one hour.

It should be noted that thermoplastic adhesives were used on aluminum adherends to evaluate their structural potential. It is realized that the processing temperatures used are above acceptable limits for aluminum but alternate methods of heating or fusing the thermoplastic adhesive exists if acceptable strengths are obtained.

2.1.3 Specimen Preparation

2.1.3.1 Polyphenylene Sulfide

Steel lap shear assemblies were fabricated from Ryton V-1 (polyphenylene sulfide) film adhesive and epoxy primed steel five-finger lap shear panels. A 10-mil film of the Ryton material was fabricated by heating the resin in a cavity mold under vacuum in a laboratory press. The material was heated from room temperature to 650°F at 11 - 13°F/minute, held 20 minutes and cooled. Full vacuum and 50 psi were held on the material throughout the molding cycle.

2.1.3.2 Polyimides

NR150A2 polyimide film adhesive was prepared by coating glass fabric (112 E-glass/A-1100) with resin. The glass was coated and dried until the total film thickness was between 8 and 12 mils. The solvent content of the film ranged from 8 - 10%. The same process was used to make the 2080D film.

Polyimide lap shear specimens were fabricated from primed metal adherends and the prepared polyimide adhesive films. The titanium and steel adherends were five-finger panels, while the aluminum adherends were standard 4" x 6" panels. The panels were laid up on steel bonding tools with the polyimide films between the lap areas of the panels. The adhesive films were cut to allow approximately 1/8 inch of excess material around the lap joint. The bonding tool assembly was envelope bagged and full vacuum was applied. The bagged tool assembly was carefully placed into the press and 75 psi platen pressure was applied. The temperature was increased from room temperature to 550°F at 3° - 4°F/minute, held for one hour, increased to 600°F at 3° - 4°F/minute, held for 20 minutes and cooled. The bonded assemblies were cut into individual lap shear specimens.

2.1.3.3 Polysulfones

Metal-to-metal lap shear specimens were fabricated with neat P-1700 and PKXA adhesive films with a glass carrier (112 E-glass/A-1100). The PKXA neat adhesive film was prepared by placing solid PKXA polymer into a cavity mold which was purged with argon and heated to 650°F for 10 minutes with 200 psi platen pressure on the mold. The mold was cooled under pressure prior to removing the film. The P-1700 neat adhesive was purchased from the manufacturer. The PKXA and P-1700 polysulfone adhesive films with glass scrim were fabricated by coating glass fabric (112 E-glass/A-1100) with 25% solutions of the polymers in methylene chloride and drying until the films were 8 - 12 mils thick. After the film thickness had reached 8 - 12 mils, the film was dried until constant weight was obtained. The drying cycle was used to remove all traces of methylene chloride.

The polysulfone lap shear specimens were laid up for bonding by placing the epoxy primed adherends on the bonding tools with the polysulfone adhesive films between the adherends in the overlap areas of the panels. The adhesive films were cut to allow approximately 1/8 inch of excess adhesive around the lap joint. The bonding tool assembly was placed into the press and a pressure of 50 psi was placed on the part. The bondline temperature was raised from room temperature to 500°F at a rate of 11° - 13°F/minute, held for 20 minutes and cooled under pressure. The panel assemblies were cut into individual lap shear specimens.

2.1.4 Test Results

The screening of the materials was carried out in two phases as follows. In the initial screening phase, lap shear specimens were fabricated with one or two adherends and tested at -65°F , RT, and elevated temperatures (300°F or 500°F). The purpose of the initial screening was to insure that there were no gross deficiencies in the selected systems and to assess the degree and type of modifications that may be required before proceeding further into the program. In the second screening phase, specimens were fabricated using each adhesive system to bond assemblies from each of the three adherends (steel, titanium, and aluminum) and tested from -65°F to their maximum estimated service temperatures. The lap shear specimens were tested on a 20K Instron machine using a loading rate of .05 inch/minute.

2.1.4.1 Polyphenylene Sulfide (PPS)

The polyphenylene sulfide bonded assemblies were very brittle and easily broken by hand or a sharp impact. Because of the excellent solvent resistance of the PPS material, several other unsuccessful attempts were made to improve the strength of the PPS bonds. Figure 1 is a photograph of a PPS bonded lap shear assembly that was failed by hand. The PPS adhesive was replaced in the test plan with polymer 100P (polyether sulfone) when it was determined that the PPS would not satisfy the adhesive requirements of this program.

2.1.4.2 Polyimides

In the initial screening, NR150A2 and 2080D polyimide titanium lap shear specimens were tested at -65°F , RT and 500°F . The NR150A2 adhesive exhibited lap shear strengths that were significantly better than the lap shear strengths of the 2080D when tested at -65°F and RT. The shear strengths deviated by 150 psi when tested at 500°F . These data are reported in Table II. In the final screening phase, titanium, steel and aluminum 2080D and NR150A2 polyimide lap shear specimens were tested at all three of the previous test temperatures. The lap shear specimens bonded with NR150A2 adhesive were significantly stronger than the specimens bonded with 2080D. The specimens bonded with NR150A2 failed 85 - 93% cohesively while the 2080D specimens failed 70 - 85% cohesively. Lap shear test results are presented in Table III. Figures 2 and 3 are photographs of the failed NR150A2 and 2080D lap shear specimens, respectively. The NR150A2 polyimide was selected for further testing.

2.1.4.3 Polysulfones

The PKXA and P-1700 bonded lap shear specimens were tested at -65°F, RT, and 300°F. In the initial screening phase, specimens were tested with neat resin adhesives and adhesives with glass carrier. The specimens with glass carrier exhibited the better shear test results with the P-1700 adhesive, while specimens tested with the neat resin adhesive gave the better test data for the PKXA adhesive. The results from the initial screening are presented in Table II. In examining the failed shear specimens, it was noted that specimens bonded with adhesives having glass carrier were more uniform in bond thickness and failure modes. See Figures 4 and 5.

In the final screening phase, all specimens tested had scrim cloth in the adhesives. The specimens were tested at all three temperatures (-65°F, RT, and 300°F). The PKXA specimens were significantly stronger than the specimens tested in the initial screening phase, while the P-1700 specimens compared favorably with the initial specimens (see Tables II and III). As expected, the average shear strength was greatest for the steel specimens with one exception. Test results for steel PKXA shear specimens tested at 300°F were lower than titanium or aluminum. Figure 6 is a photograph of tested specimens.

The polysulfone lap shear specimens from both of the screening phases failed 85 - 93% cohesively. Table III is a summary of the test results from the final screening. The PKXA adhesive was selected for further testing.

2.2 PHASE II - PROCESSING STUDY

Tests were performed to determine the optimum processing parameters for the selected resin systems, PKXA and NR150A2. The tests consisted of three separate studies - one to optimize surface preparation techniques for each of the three metallic substrate materials; a second to optimize processing parameters of temperature and pressure; and a third for adhesive modification and the effects of temperature and pressure on the modified adhesives.

2.2.1 Surface Preparation Study

After completing the screening work in Phase I, PKXA and NR150A2 were selected for further evaluation. The adhesives were evaluated with each substrate material (2024-T3 bare aluminum, 17-7PH stainless steel, and 6Al-4V

titanium) using three different surface preparation techniques for each adherend. These surface preparations were:

Aluminum (2024-T3 bare)

Sodium Dichromate - Sulfuric Acid Anodize
Chromic Acid Anodize
Phosphoric Acid Anodize

Titanium (6Al-4V)

Chromic Acid Anodize
Pasa-Gel
Nitric-Hydrofluoric Acid Etch

Stainless Steel (17-7PH)

Sulfuric Acid Anodize
Nitric-Hydrofluoric Acid Etch
Alkaline Clean (Only)

The comparative test method used to evaluate the bondability of the surfaces produced from each of the surface preparation techniques was the crack extension shown in Figure 7. The method was selected because it is sensitive to surface preparation and very cost-effective. Qualitative comparisons between specimens are made by measuring the degree of crack extension following the initial wedge insertion and then comparing the progressive failure (crack extension) with time. The initial and progressive modes of failure are also recorded in laboratory notebooks for future reference. Figure 8 is a typical titanium crack extension specimen under stress with the stress wedge in place.

Specimen Fabrication and Testing - The titanium and stainless steel crack extension panels were fabricated from .050 inch sheet material, while the aluminum panels were fabricated from .125 inch plate aluminum. All the panels were sheared to size ($6.0" \pm .125" \times 6.0" \pm .125"$). Panels from each adherend material were processed with each of the three surface treatment techniques previously noted for that particular material and primed for bonding. Panels bonded with NR150A2 polyimide were primed with a dilute solution (5% solids) of the NR150A2 adhesive thinned with ethyl alcohol. The primer was applied with a spray gun, air dried for two hours, then forced air dried in

an oven at 550°F for one hour. The temperature was increased from room temperature to 550°F at a rate of 5° - 7°F/minute. BR127 (epoxy) primer was applied to all panels bonded with PKXA adhesive. The BR127 primer was sprayed to a thickness of .2 to .4 mil, air dried for 30 minutes, and cured in an air-circulating oven at 260°F ± 5°F for one hour. The primed adherends were bonded with 10 - 12 mil films of PKXA and NR150A2 adhesives using the same cure cycles that were previously used in Phase I of this program. After bonding, each 6-inch x 6-inch bonded assembly was cut into five (1" x 6") crack extension specimens. A 0.125 inch thick wedge was then driven into the bondline of each specimen. The resulting crack length was marked and each specimen exposed 200 hours at room temperature and 200 hours at their respective maximum use temperature. The maximum use temperatures for PKXA and NR150A2 were 300°F and 500°F, respectively. The crack lengths, initial and after aging, were measured and recorded for each specimen. Table IV is a data summary of the crack extension specimens.

Test Results - All three surface preparations for aluminum bonded with PKXA and NR150A2 adhesives were approximately equal in bond strengths. Phosphoric acid anodize appeared to be the most consistent of the processes and was selected on this basis for Phase III. See Figures 9 - 14 for photographs of the aluminum specimens.

The sulfuric acid anodize treatment for stainless steel appeared to be the best cleaning method for bonding steel with both PKXA and NR150A2 adhesives. The alkaline process was the least effective method of the three processes used to prepare the surfaces of stainless steel. The epoxy primer did not bond well to the alkaline or nitric acid surface treated panels and resulted in the PKXA adhesive system failing 65% and 40% cohesively for the alkaline and nitric acid processes. Figures 15 - 20 are photographs of these steel specimens. The sulfuric acid anodize treatment for stainless steel was selected for Phase III.

Initially, the pasa-gel surface treatment for titanium appeared to be the best surface for bonding NR150A2 adhesive. The specimens had an average crack growth of .38 inch, while the crack growths for the nitric hydrofluoric acid etch and chromic acid anodize specimens were .38 inch for the HF complete delamination for the CrO₃. Figures 21 - 23 are photographs of these specimens.

The failure mode of specimens from all three surface treatments was high in adhesive failure (75 - 95%) when tested at 500°F (see Table IV). After further analysis of the failed specimens, it was noticed that the adhesive failure of the chromic acid anodize specimens was between the metal oxide and the titanium metal. It was concluded that solvent from the NR150A2 adhesive system or some unknown mechanism caused embrittlement of the oxide layer which was developed in the chromic acid process. The embrittled oxide failed with low tensile strength under stress.

It was decided to re-evaluate the pasa-gel and chromic acid anodize processes using NR150A2 adhesive with solvent content of 5% or less. After this re-evaluation of the pasa-gel and chromic acid processes, the average initial crack and crack growth were 1.82" and 0.32" for the pasa-gel specimens, while the average initial crack and crack growth for the chromic acid anodize specimens were 1.62" and 0.28". The failures were 80 - 85% cohesive for the pasa-gel and 85 - 95% cohesive for the chromic acid anodized specimens. The latter method was chosen for the NR150A2 adhesive with a restriction imposed on the solvent content level of the adhesive (<5%).

The chromic acid anodize surface treatment was the best surface for bonding with PKXA adhesive. These specimens failed 95 - 100% cohesively, while specimens bonded with pasa-gel completely delaminated during cutting with 5 - 25% cohesive failure. The nitric hydrofluoric acid etch specimens failed 65 and 75% cohesively after room temperature and elevated temperature (300°F) tests, respectively. Photographs of these specimens are shown in Figures 24 - 26. Consequently, the chromic and anodize surface treatment for titanium was selected also for the PKXA adhesive in Phase III.

2.2.2 Processing Parameters

Processing studies were conducted with PKXA and NR150A2 adhesives using a single adherend, aluminum, to minimize variables. The selected surface treatment for aluminum (phosphoric acid anodized) was used for processing all panels and the selection was based on tests conducted previously in the program (Section 2.2.1). The panels were primed as discussed in Section 2.2.1.

PKXA Processing Studies - Lap shear specimens were fabricated from aluminum adherends and PKXA adhesive using three different bond temperatures and pressures. Specimens were bonded using 475°F, 35 psi; 475°F, 50 psi; 475°F, 75 psi; 500°F, 35 psi; 500°F, 50 psi; 500°F, 75 psi; 525°F, 35 psi; 525°F, 50 psi; and 525°F, 75 psi as the cure temperatures and pressures. The assemblies were heated to the required temperatures in a vacuum bag under pressure, held for 20 minutes, and cooled. Specimens were tested at room temperature and 300°F. Test results are tabulated in Figure 27. The specimens failed 95 - 100% cohesively in all cases. The specimens bonded at 525°F and 35 psi displayed the best shear strength at ambient temperature and at 300°F and these parameters were selected for bonding the PKXA adhesive for the environmental exposure study, Phase III (Section 2.3). No flow or processing problems were encountered with the PKXA adhesive. However, the amount of flow varied with the temperature and pressure cycle used.

NR150A2 Processing Study - Aluminum lap shear specimens were fabricated using NR150A2 adhesive with three different cure temperatures and pressures. The cure temperatures and pressures were 550°F; 600°F; 550°F and 600°F; and 50 psi, 75 psi and 100 psi. A total of nine different cures, which are noted in Figure 28, were evaluated.

For the 550°F and 600°F cures, the temperature was increased from room temperature to 550 or 600°F under pressure at 3 - 4°F/minute, held for one hour and cooled. In the cure using 550 and 600°F, the temperature was increased from room temperature to 550°F under pressure, held for one hour, increased to 600°F at 3 - 4°F/minute, held for 20 minutes and cooled. The bonds were under full vacuum throughout the cure schedule.

The specimens bonded with the 550°F cure temperature under 50, 75, and 100 psi failed in RT shear at 3120, 3400, and 3180 psi, respectively. Specimens bonded at 600°F failed at shear strengths lower than obtained with the other two cure cycles with one exception (600°F, 75 psi). The specimens bonded using the one hour at 550°F plus 20 minutes at 600°F under 75 psi gave the best shear data when tested at RT and 500°F and this bonding cycle was selected for Phase III. These specimens failed in shear at 4700 and 3340 for the RT and 500°F, respectively.

Specimens bonded under 50 psi displayed very little flow of the adhesive, while specimens bonded under 100 psi showed excessive flow and some distortion in the glass scrim. The amount of flow was directly proportional to the pressure and solvent content of the adhesive. Test results from these specimens are tabulated in Figure 28.

2.2.3 Adhesive Modification

The PKXA adhesive system was selected for modification studies toward improvement in the adhesive strength of the resin system. The modification studies were conducted using BR127 (epoxy) primed, phosphoric acid anodized aluminum lap shear panels as the adherends. The three basic modifications evaluated were: scrim cloth, metallic and nonmetallic fillers. It was determined early in Phase I that adhesive with scrim cloth was an improvement over the neat resin film. In this study, seven different adhesive films were evaluated. Six of the films were modified and one film consisted of the neat resin only. The modifications were: (A) PKXA, (B) PKXA with 15% amorphous boron, (C) PKXA with 15% amorphous boron and 5% polyphenylene sulfide, (D) PKXA with 15% powdered aluminum, (E) PKXA with 15% powdered aluminum, 5% polyphenylene sulfide, (F) PKXA with 15% amorphous boron, 3% powdered aluminum, (G) PKXA with 15% amorphous boron, 3% polyphenylene sulfide. The above films contained 112 glass scrim in the adhesive films for thickness control.

Lap shear specimens were fabricated using three different bonding temperatures and two different bonding pressures. These bonding temperatures and pressures were 475°F, 500°F, 525°F, and 35 and 50 psi, respectively. These specimens were laid up, vacuum bagged, and bonded in a press under pressure using 11 - 13°F/minute rate rise with a 20-minute hold at temperature prior to cooling.

Lap shear specimens fabricated from amorphous boron and aluminum modified adhesives (Modifications B, C, D, E, and F) failed at low shear strengths. After examining the failed specimens, it was noted that the PKXA resin had a tendency to migrate to the outer surface of the adhesive tapes, leaving the boron and aluminum fillers resin starved. One possible way of eliminating this problem is to reduce the amount of boron or aluminum used in the modifications. Lap shear specimens fabricated from Modification "D" (PKXA

with 15% powdered aluminum on glass scrim) and cured at 500°F, 50 psi, exhibited the best lap shear test results of the filler modified adhesives specimens. The unfilled PKXA on glass scrim (Modification "A") exhibited the best shear test data. Test results from the modified PKXA adhesive are reported in Tables V and VI. Figures 29 - 33 are photographs of some of the failed specimens from the modification study.

These limited modifications of the PKXA adhesive did not improve the room temperature or elevated temperature shear strengths of the adhesive over the properties obtained from bonding with neat PKXA adhesive on glass scrim. More work is required to fully explore other possibilities of modifying the PKXA resin to improve its adhesive strength.

2.3 PHASE III - ENVIRONMENTAL EXPOSURE

Adhesive bonds have the tendency to deteriorate under various environments, causing bond failure in the adhesive and/or at the adherend-adhesive interface. This phase of the program was undertaken to assess the problem(s) and evaluate the structural integrity of thermoplastic bonds under stressed conditions in chosen environments. The device selected to evaluate the thermoplastic adhesives under stress conditions in chosen environments was the double cantilever beam (DCB) shown in Figure 34. The mechanics of this test specimen are discussed in detail in Reference 10.

Double cantilever beam and lap shear specimens were fabricated from stainless steel, titanium, and aluminum adherends bonded with PKXA and NR150A2 adhesives. The surface preparation for steel, titanium and aluminum adherends were sulfuric acid anodize, chromic acid anodize and phosphoric acid anodize, respectively. The procedure for priming the adherends in Section 2.2.1 was used for the adherends and lap shear panels. The aluminum DCB specimens were cut from bonded 12" x 12" x 0.5" assemblies (bonded per Section 2.2.2). The steel and titanium DCB specimens were bonded as individual 1" x 12" x 0.3" specimens. The steel and titanium adherends were cut and bonded as individual specimens to eliminate heating the bond area during cutting.

The steel and aluminum DCB specimens were drilled and tapped so the ends could be separated by tightening bolts to make the initial crack and place stress on

the bonds. Because of the work and expense involved in drilling and tapping titanium, steel wedge spacers were used to make the initial crack on the titanium specimens. Qualitative comparisons between specimens were made by measuring the degree of crack extension following the initial opening of the specimens (by wedge or torque on the bolts) and then examining the progressive failure (crack growth) with time in the selected environments. The initial crack length and failure mode were recorded for all stressed specimens for comparison with the unstressed specimens after environmental aging.

Stressed and unstressed DCB specimens were exposed in thermal, salt water, humidity and fluid environments for different periods. Upon completion of the aging cycle for a group of specimens, stress and unstressed specimens were removed from the environments. The difference between the initial crack arrest point (before aging) and the final crack arrest point (after aging) was measured for the stressed specimens and reported as crack growth. The unstressed specimens after exposure were placed in the stressed mode and the initial crack length and type of failure was compared to the data of the environmentally stressed specimens prior to exposure. All of the specimens that were environmentally aged in the unstressed condition failed 95 - 100% cohesively with initial cracks comparable to the stressed specimens prior to environment. The test results of the stressed specimens are covered in the following sections. The control lap shear data which are shown on Figures 35 - 39 are the average of PKXA and NR150A2 specimens which were bonded in the same autoclave cure as the respective DCB specimens. The control values for PKXA and NR150A2 lap shear specimens were 3470 and 3460 psi, respectively.

2.3.1 Thermal Aging

NR150A2

Steel stressed DCB specimens, bonded with NR150A2 adhesive, exhibited crack growth of 1.16 inches after 1000 hours of aging at 500°F, while aluminum and titanium specimens exhibited crack growths of 1.67 and 2.05 inches, respectively. Since the failure modes of these DCB specimens were 75 - 85% cohesive, it was concluded that the difference in crack growth was probably due to variability in the adhesive or its processing, rather than thermal aging under stress. The crack growths were larger on some specimens after 100 hours of aging than other specimens aged for 1000 hours. This is a result of aging separate groups of specimens for each aging period rather than aging the same specimens for

10, 100, and 1000 hours. The mode of failure is the significant factor and it remained cohesive throughout the exposures. Table VII is a summary of the DCB data.

Lap shear specimens bonded with NR150A2 exhibited test results for the steel specimens that were much lower (>15%) than aluminum or titanium specimens (see Figure 35). This is the opposite of what was expected since the least amount of crack growth was measured for the steel DCB specimens. Since the lap shear specimens failed 75 - 85% cohesively, we cannot explain the low shear strength values obtained.

PKXA

Double cantilever beam specimens bonded with PKXA adhesive showed very little crack growth in thermal aging. Only one aluminum specimen and two titanium specimens exhibited crack growth after 100 hours and 1000 hours, respectively. Crack growth was not detected in any of the other specimens. Crack growth data are reported in Table VII.

Steel lap shear specimens, bonded with PKXA adhesive, displayed the highest shear strength results after 10 and 100 hours of aging. The test results for all the shear specimens are greater than 3100 psi. These shear data are reported in Figure 35.

The failure modes of the PKXA shear and double cantilever beam specimens were 80 - 95% cohesive.

It was concluded that the PKXA adhesive has excellent resistance to crack growth under stress and good lap shear strength after aging up to 1000 hours at 300°F.

2.3.2 Salt Water Environment

To assess the effects of salt water upon PKXA and NR150A2 bond strengths, double cantilever beams and lap shear specimens were evaluated after 200 hours of immersion in a salt spray chamber in accordance with ASTM B-117-64. The chamber was maintained at 100% RH, $100 \pm 5^\circ\text{F}$ temperature with a $5 \pm .5\%$ sodium chloride solution.

NR150A2

Steel, titanium, and aluminum stressed double cantilever beam specimens bonded with NR150A2 adhesive exhibited an average crack growth of .25 inch on steel specimens. No crack growth was noted for the titanium and aluminum specimens. Test results are reported in Table VIII.

The average shear strength for titanium specimens was 5130 psi or 148% of the original strength. These specimens failed 100% cohesively. Steel and aluminum lap shear specimens failed at 2385 and 2455 psi, respectively, which is 31% and 29% of their respective controls. These specimens failed 40 - 60% adhesively. Shear data are reported in Figure 36.

Some salt corrosion was noted on the aluminum specimens which was a contributing factor for the strength reduction.

PKXA

Steel and titanium stressed double cantilever beam specimens showed no crack growth, while one aluminum specimen exhibited a crack growth of 1.75 inches. This specimen switched from the cohesive failure mode to adhesive mode for the length of the crack. Salt corrosion of the aluminum under the BR127 primer was the cause of the switch in the failure mode from cohesive to adhesive. The data from these DCB specimens were previously reported in Table VIII.

Steel, titanium and aluminum lap shear specimens failed 80 - 85% cohesively. Some of the aluminum specimens switched from the cohesive mode to adhesive mode during testing. Specimens bonded with steel adherends yielded the best shear strength. These data are presented in Figure 36.

It was concluded that the lap shear strength of the PKXA bonded specimens was reduced slightly (<17%) from exposure to salt water environment.

2.3.3 Humidity Environment

NR150A2

NR150A2 lap shear specimens and double cantilever beam specimens were exposed in condensing humidity environment. The humidity chamber was maintained at

120 ± 5°F with 100% relative humidity. Specimens were aged for 7 days and 24 days.

Double cantilever beam specimens exhibited some crack growth with all three adherends when bonded with NR150A2 adhesive. The largest crack growths (1.28") were among the aluminum specimens after 24 days in environment under stress. The stressed specimens failed 95 - 100% cohesively.

Lap shear specimens bonded with steel and titanium exhibited the best shear strength. The aluminum and steel specimens failed 60 - 85% cohesively. Titanium specimens failed 95 - 100% cohesively.

It was concluded that humidity environment had no adverse effect on the stressed DCB specimens but the lap shear strength of the steel and aluminum were affected by the humidity environment. The shear specimens failed 15 - 40% adhesively with the primer lifting from the metal surfaces.

PKXA

Humidity environment had no adverse effect upon the steel stressed double cantilever beam specimens bonded with PKXA adhesive, while the titanium and aluminum specimens exhibited minor crack growth. Test results from the humidity environment are reported in Table IX and Figure 37.

Lap shear specimens exposed to humidity for 7 and 24 days exhibited a decrease in shear strength. These decreases for specimens aged 7 and 24 days were 34 and 37% for steel, 19 and 7% for titanium, and 33 and 28% for aluminum, respectively. The failure modes of the steel and aluminum specimens were 80 - 85% cohesive, while the titanium specimens failed 95 - 100% cohesive.

2.3.4 Fluid Environments

Adhesives are widely used in the manufacturing of aircraft, missiles, and other aerospace-related hardware. Adhesives used in fabrication of some of these items require resistance to a large number of solvents, lubricants, fuels, and hydraulic fluids. These tests were to assess the effects of JP-4 jet fuel, MIL-L-7808 aircraft lubricant, and MIL-H-5606 hydraulic fluid on NR150A2 and PKXA adhesives.

2.3.4.1 JP-4 Environment

NR150A2

Double cantilever beam specimens bonded with NR150A2 adhesive showed crack growth on all specimens of each substrate. The greatest crack growth was on aluminum specimens in JP-4. One specimen exposed at room temperature delaminated completely in the environment. The JP-4 at 160°F had the most effect on the aluminum specimens, while the steel specimens exhibited more crack growth with JP-4 at room temperature. Table X is a summary of the test results.

The shear strength of titanium and steel lap shear specimens bonded with NR150A2 was reduced from 50 - 80% when specimens were soaked in JP-4 at 160°F. The titanium specimens had the greatest loss. The specimens failed adhesively. The failures were between the metal and primer. Test results are reported graphically in Figures 38 and 39.

It was concluded that JP-4 at RT and 160°F had a detrimental effect on both the stressed DCB specimens and lap shear specimens, with the failure mode in both cases being between the primer and metal interface.

PKXA

No crack growth was obtained when steel double cantilever beam specimens were soaked in JP-4 fuel for 10 and 24 days at 160°F. Titanium and aluminum specimens under the same environment displayed crack growths from 0.25 inch to 1.5 inches. The PKXA specimens had much more resistance to the fluid at 160°F than the NR150 specimens. Specimens from all three substrates displayed some crack growth in JP-4 at room temperature. Data from these specimens are shown in Table XI.

Lap shear specimens bonded with PKXA adhesives and soaked in JP-4 for 10 and 24 days at 160°F showed reductions in strength up to 31.1%. These specimens failed 80 - 85% cohesively. Test results are reported in Figures 38 and 39. The PKXA specimens aged for 24 days in RT JP-4 displayed up to 8.2% drop in shear strength; 87 - 95% cohesive failures were obtained.

It was concluded that PKXA adhesive had excellent resistance to crack growth in JP-4 at RT and 160°F but JP-4 at 160°F had a detrimental effect on lap shear strength after 7 days in the fluid.

2.3.4.2 MIL-L-7808 Environment

NR150A2

Double cantilever beam specimens were fabricated from steel, titanium, and aluminum with NR150A2 adhesives. These beam specimens were tested after exposure to RT and 160°F MIL-L-7808. Crack growth was noted on all specimens exposed in MIL-L-7808. One aluminum specimen that was immersed in MIL-L-7808 was completely delaminated. The average crack growth was from 1.25 " - 2.47" for steel, 2.79" to complete delamination for the aluminum, and 0.49" - 1.92" for the titanium specimens. Table XII is a summary of these crack data. These stressed DCB specimens failed 80 - 85% cohesively with the exception of the aluminum specimens that were exposed to hot MIL-L-7808. These aluminum specimens failed 90 - 95% adhesively between the primer and metal interface.

PKXA

The PKXA bonded double cantilever beam specimens displayed excellent crack resistance to MIL-L-7808 solutions. With the exception of one titanium specimen aged at 160°F, the average crack growth was less than 0.80". The average RT crack growth ranged from .00" to .42", while the average crack growth at 160°F ranged from .43" to 1.23". These data are reported in Table XIII. The specimens failed 90 - 95% cohesively.

MIL-L-7808 had a detrimental effect on NR150A2 stressed DCB specimens. This effect was most severe on aluminum bonded specimens in fluid at 160°F. The PKXA stressed DCB specimens showed no detrimental effect after soaking in RT or 160°F MIL-L-7808 fluid.

2.3.4.3 MIL-H-5606 Environment

NR150A2

Double cantilever beam specimens fabricated from steel, titanium and aluminum adherends and NR150A2 adhesives displayed crack growth of from 0.62" to 2.64" when immersed in MIL-H-5606 solution for 24 days. The crack growth of the

specimens immersed in hot solution was significantly larger than specimens immersed at room temperature. The largest crack growth (2.64") was exhibited by the titanium specimens, while the smallest (0.62") growth was displayed by the aluminum specimens. These data are presented in Table XIV. The failure mode of the specimens was 85 - 95% cohesive.

PKXA

The PKXA double cantilever beam specimens exhibited crack growth that was significantly smaller than the NR150A2 adhesive system when exposed to MIL-H-5606 hydraulic fluid. The crack growth of the PKXA specimens ranged from no growth to 0.47". Specimens exposed in MIL-H-5606 solution at 160°F showed less resistance to crack growth than the specimens immersed in solution at 70°F. The failure mode of the specimens was 90 - 95% cohesive. Data from the PKXA specimens are presented in Table XV.

MIL-H-5606 had a significant effect on NR150A2 stressed DCB specimens. The aluminum specimens had more resistance to crack growth than steel or titanium specimens when bonded with NR150A2. PKXA stressed DCB specimens displayed no adverse effect from immersion in RT or hot MIL-H-5606 solution.

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 CONCLUSIONS

From the preliminary work conducted under this program, it is concluded that some of the thermoplastic materials are excellent structural adhesives and will bond to steel, aluminum and titanium adherends using the same surface preparation methods that are used for epoxies and other structural adhesives in production use today. Some of the specific conclusions are as follows.

The best adhesive properties were produced from specimens bonded with 8 - 12 mil adhesive film containing 112 E-glass scrim which helps to control the flow of adhesive and provides a more uniform bond thickness.

Polyphenylene sulfide is too brittle for a structural adhesive without some form of modification. The material has very good adhesion qualities but the adhesive can be readily fractured by hand due to its low elongation.

Polyimide 2080D is difficult to use as a structural adhesive because the polymer is very hygroscopic. However, good bond strength can be obtained when extreme care is used to prevent moisture from condensing on the primed bonding surfaces and the adhesive film.

Polyethersulfone (100P) has excellent adhesive shear properties at -65°F, RT, and 300°F. The shear properties of polyethersulfone are comparable to polysulfone (P-1700 and PKXA) adhesives.

NR150A2 Adhesive

The best surface preparation methods for bonding steel, aluminum and titanium adherends with NR150A2 adhesive were sulfuric acid anodize, phosphoric acid anodize, and chromic acid anodize, respectively.

Spray application of NR150A2 (diluted NR150A2 adhesive) primer on the adherends was an improvement over the brush application. The primer coating was more uniform across the bonding surfaces and thinner primer coats were applied which allowed the solvent to evaporate without lifting the primer from the metal surfaces during solvent removal.

NR150A2 adhesively bonded DCB specimens were significantly affected in thermal, JP-4 (RT and 160°F), and MIL-L 7808 environments. DCB specimens were not significantly affected in humidity and MIL-H-5606 environments.

NR150A2 bonded titanium lap shear specimens were not affected from exposure to JP-4 at RT, humidity, and salt spray environments but a detrimental decrease in shear strength was noted for specimens exposed to thermal (500°F) and JP-4 at 160°F environments.

Aluminum lap shear specimens bonded with NR150A2 adhesive were not affected in thermal aging (500°F), while specimens exposed to humidity, JP-4 at RT, JP-4 at 160°F, and salt spray environments decreased in shear strength by 30%, 30%, 44%, and 30%, respectively.

Steel lap shear specimens bonded with NR150A2 adhesive exhibited a significant decrease in shear strength in salt water, humidity and thermal (500°F) environments. A detrimental decrease in shear strength was noted for specimens immersed in JP-4 at 160°F.

PKXA Adhesive

PKXA adhesively bonded DCB specimens had excellent resistance to crack growth in salt water, JP-4, humidity, MIL-L-7808, MIL-H-5606 and thermal (300°F) environments.

Limited modification of the PKXA neat resin with powdered aluminum, amorphous boron, and polyphenylene sulfide powder did not improve the adhesive properties of the PKXA adhesive. The addition of 112 E-glass scrim in the adhesive film did help control the flow of the material, which resulted in a more uniform bond thickness and greater adhesive strength.

The shear strength of PKXA adhesive bonds was not significantly decreased in thermal or room temperature JP-4 environments, but humidity environments reduced the shear strength of the steel, titanium, and aluminum specimens by 34.3%, 19.5% and 33.3%, respectively. PKXA lap shear strength was decreased in JP-4 at 160°F by 22%, 8.4% and 22.6% after 7 days and 31%, 25% and 29% after 24 days for steel, titanium, and aluminum, respectively.

3.2 RECOMMENDATIONS

It is recommended that further studies be conducted on PKXA, polyethersulfone 100P, and NR150A2 adhesives. It is also recommended that studies be conducted to improve the surface treatment process(es) for titanium.

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10. Marceau, J. A., Moji, Y. and McMillen, J.C., "A Wedge Test for Evaluating Adhesive Bonded Surface Durability," The Boeing Commercial Airplane Company, Seattle, Washington, April 1976.
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TABLE I - RESINS SURVEYED

<u>TYPE MATERIAL</u>	<u>DESIGNATION</u>	<u>MANUFACTURER</u>
Polysulfones	P-1700* PKXA*	Union Carbide
Polyarylsulfones	HC 5601 Astrel 360	3M Company
Polyether Sulfone	100P** 200P 300P 720P	ICI Plastics Division
Polyimides	NR150* 2080D*	DuPont Upjohn
Polyphenylene Sulfide	Ryton V-1	Phillips Petroleum
Amide-imide	Torlon 3000 Torlon 4000	AMOCO

* Resins selected for screening

** Ryton V-1 was replaced in screening with this resin.

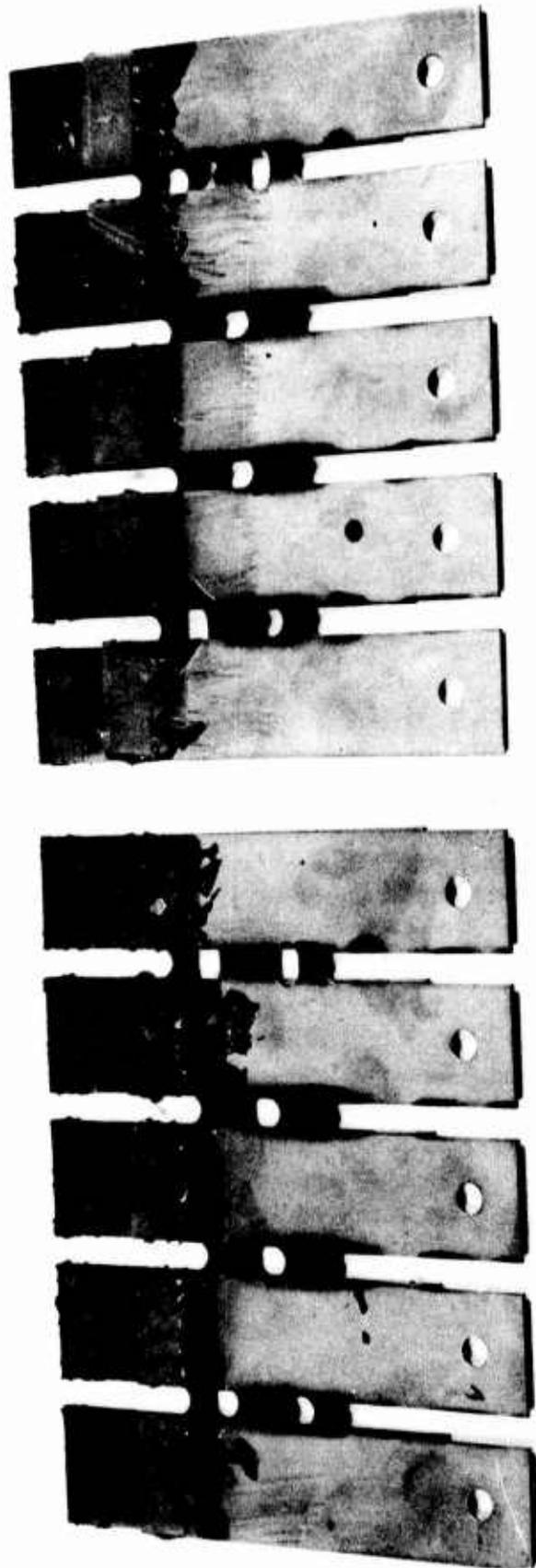


Figure 1: Polyphenylene Sulfide Panels

TABLE II - INITIAL SCREENING TEST RESULTS

Adhesive	Substrate	Average Shear (psi)			
		RT	-65°F	300°F	500°F
P-1700	Al	3360	3640	2360	
P-1700 *	Ti	3810	4100	2410	
PKXA	Ti	3260	3580	2690	
PKXA *	Ti	3000	3410	2500	
100P *	Ti	2940	3160	2870	
NR-150 *	Ti	3550	3800		3200
2080D *	Ti	2720	3000		3050

* Specimens were bonded with glass scrim in bondline

TABLE III - SCREENING TEST RESULTS

Adhesive	Substrate	Average Shear (psi)			
		RT	-65°F	300°F	500°F
P-1700	Al	3840	4100	2120	
	Ti	4000	4200	2345	
	SS	4160	4280	2440	
PKXA	Al	4820	5210	2680	
	Ti	4600	4830	2420	
	SS	5470	5510	2175	
100P	Al	3810	4155	3490	
	Ti	4075	4360	3545	
	SS	4480	5145	3540	
2080D	Al	2900	3128		1900
	Ti	3260	3085		2100
	SS	3420	3860		1875
NR-150A2	Al	3400	3670		2490
	Ti	3645	3920		2760
	SS	3840	4120		2800

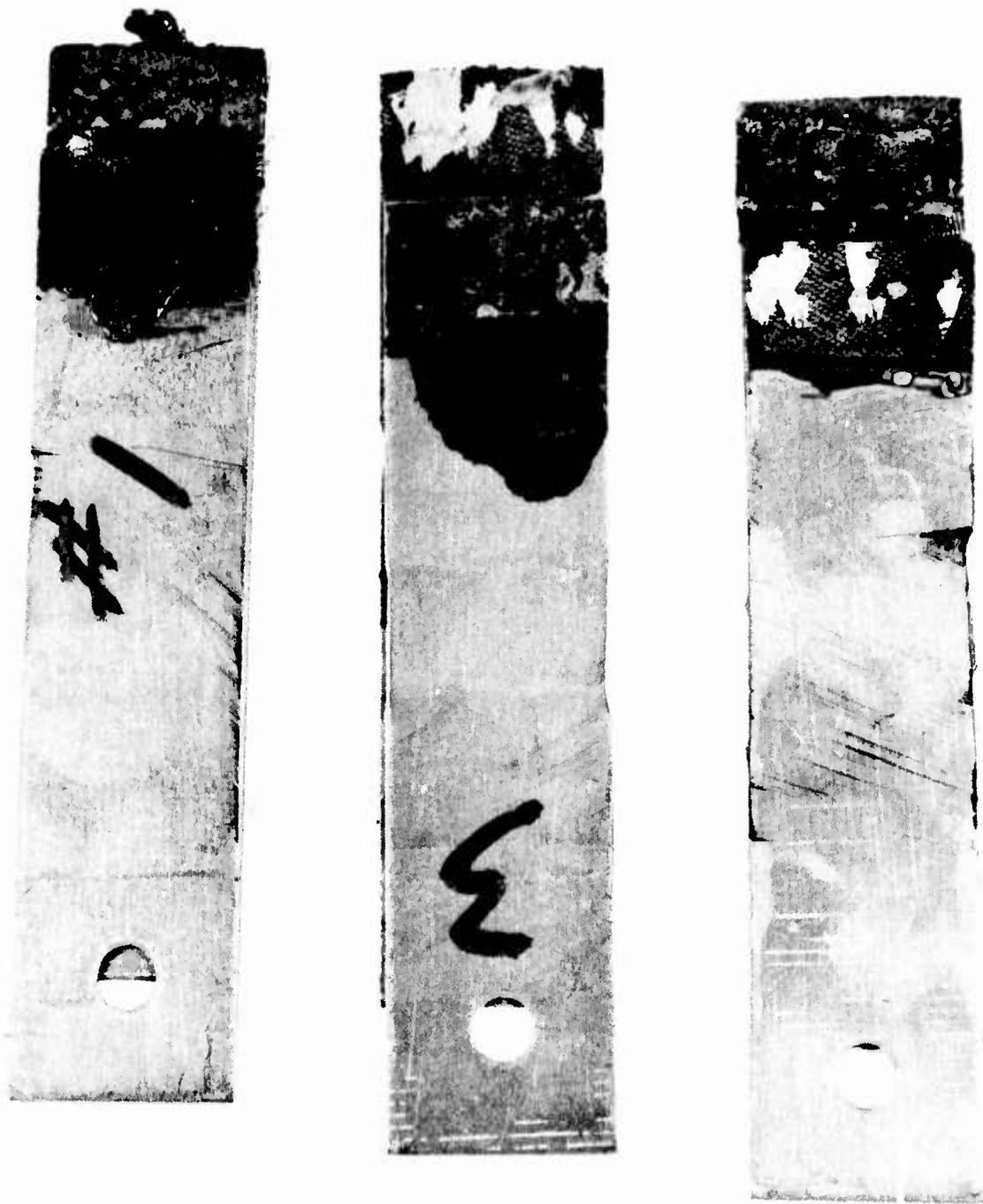


Figure 2: Tested NR150 Lap Shear Specimens

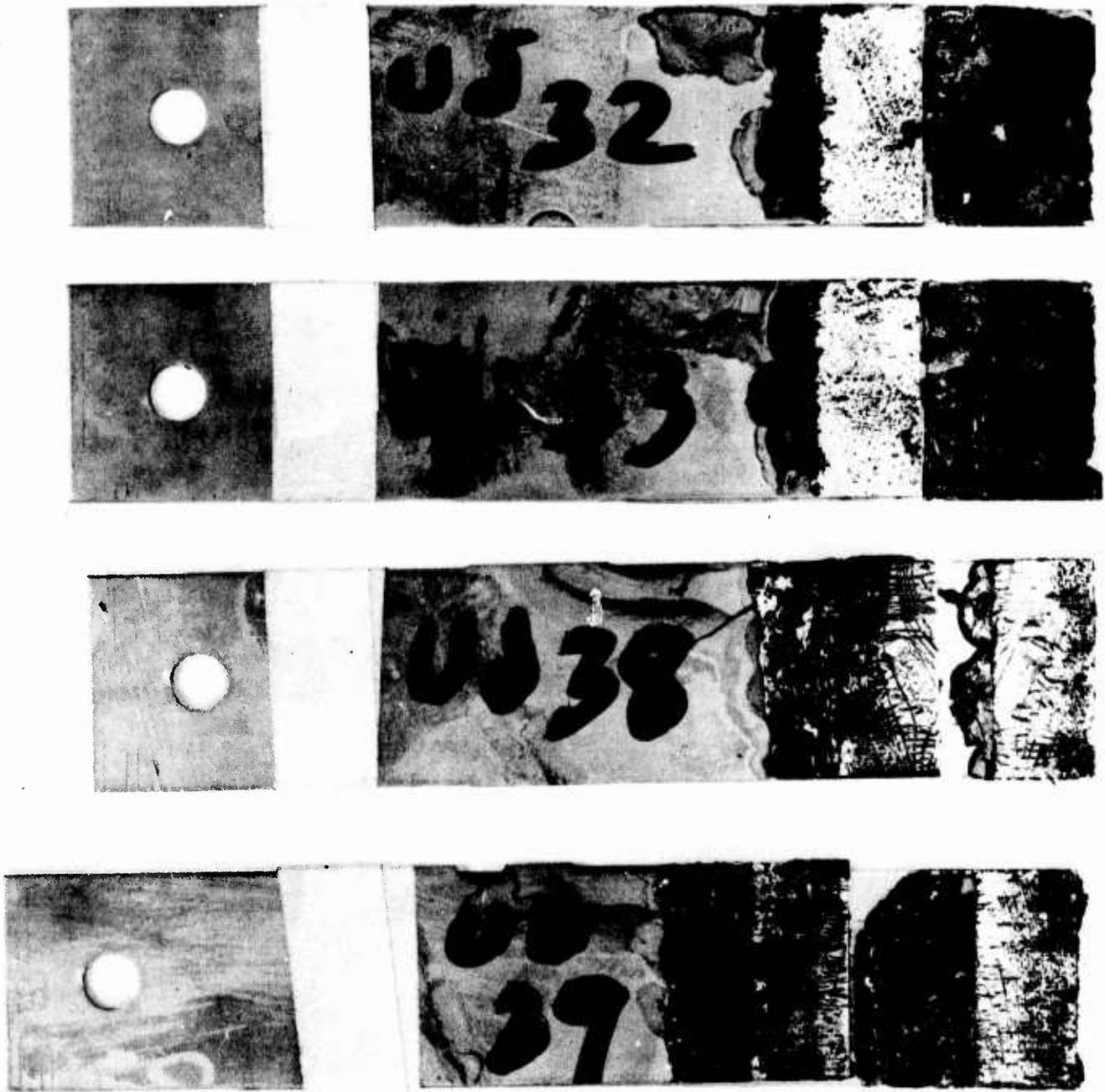


Figure 3: Tested UpJohn 2080 Lap Shear Specimens

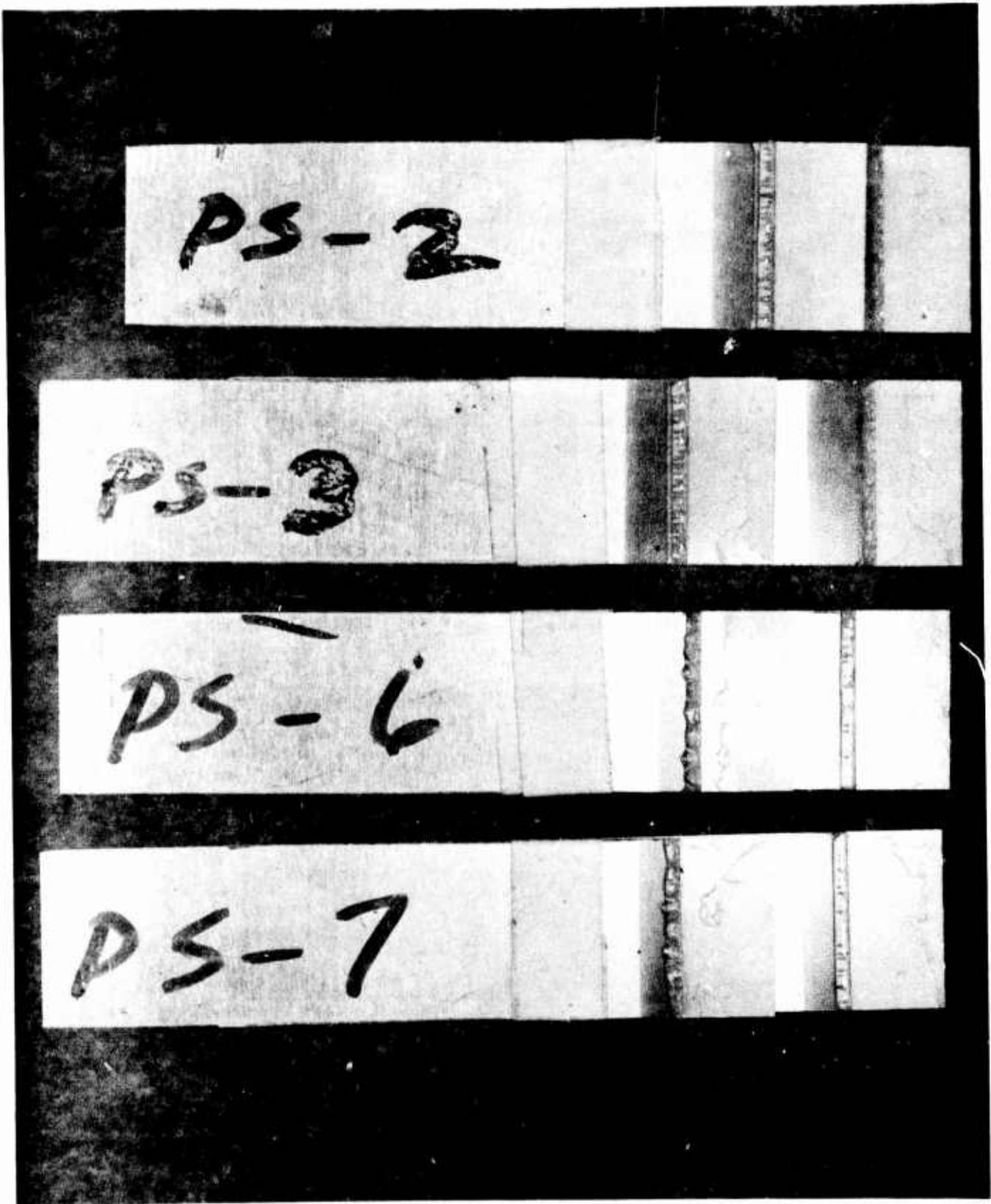


Figure 4: Tested Polysulfone Lap Shear Specimens



Figure 5: Tested PKXA Lap Shear Specimens

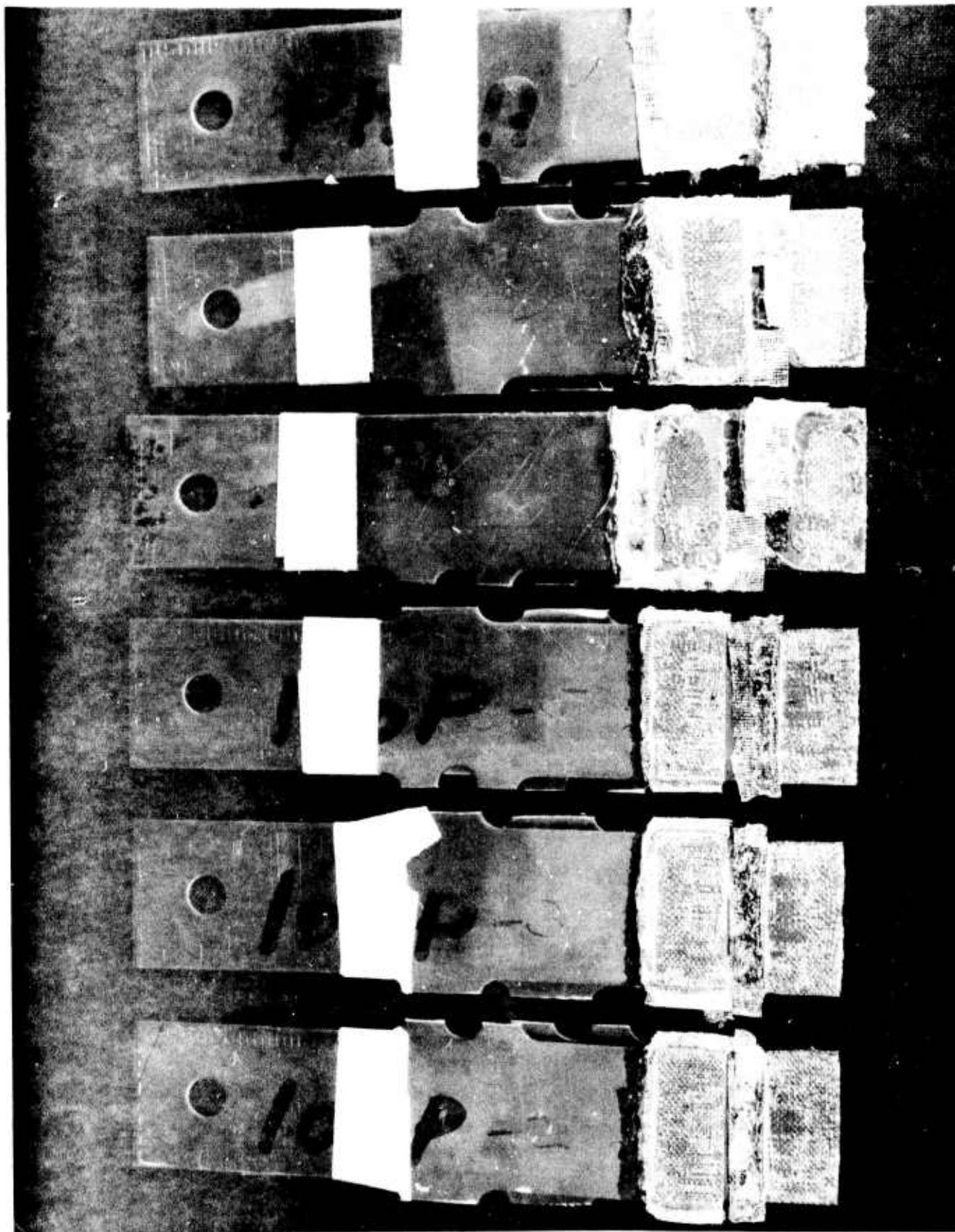
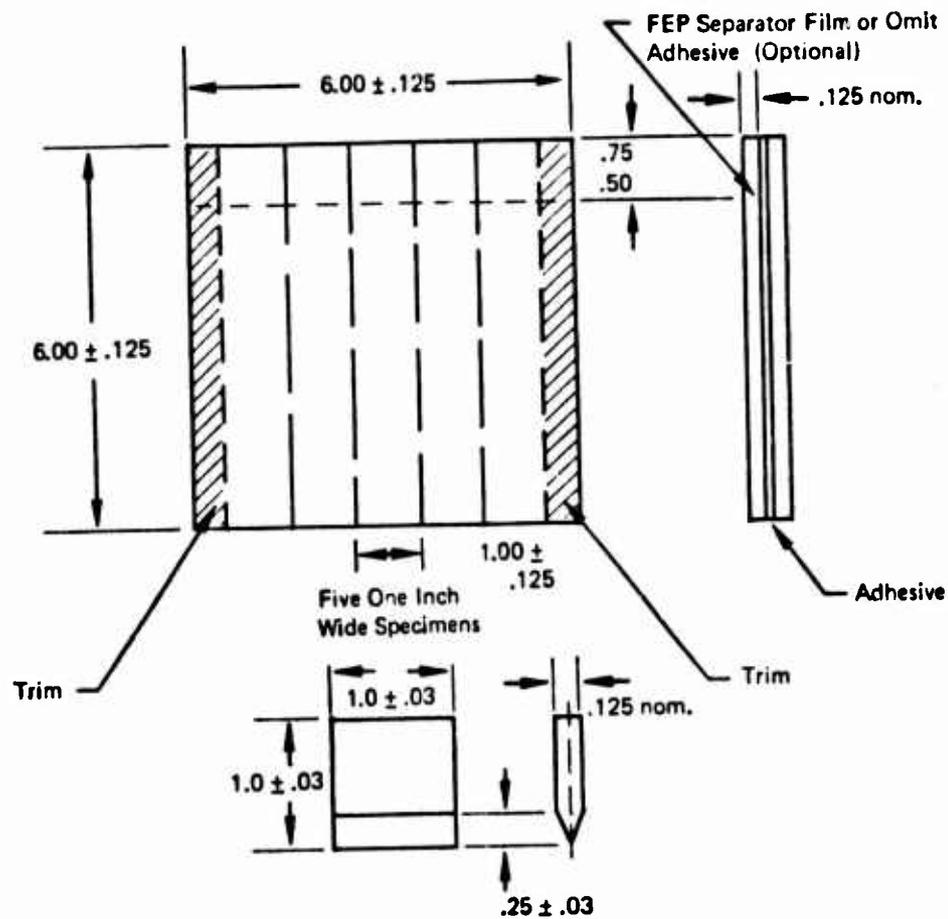


Figure 6: Failed Stainless Steel Lap Shear Specimens



Wedged Crack Extension Specimen - The End and Sides of the Wedge Shall be Approximately Flush with Specimen End and Sides

Figure 7: Crack Extension Specimen Configuration

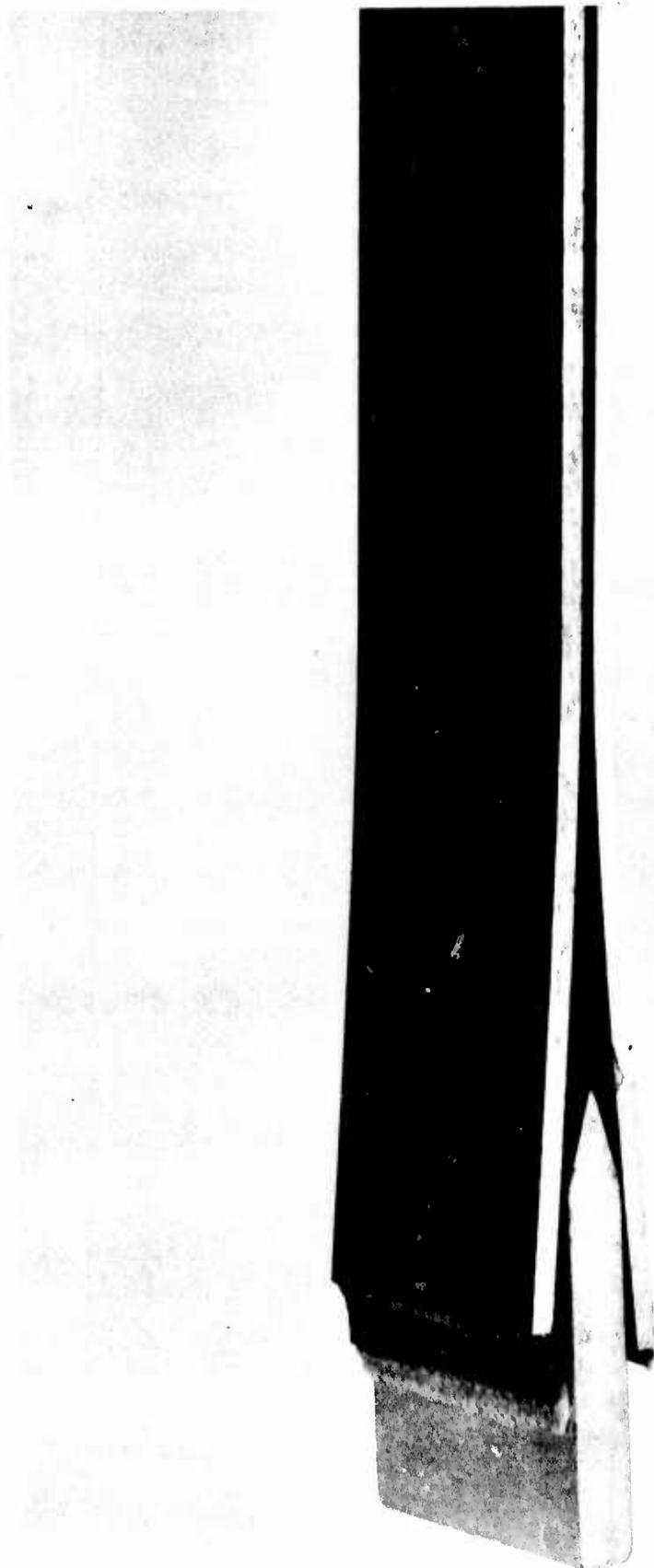
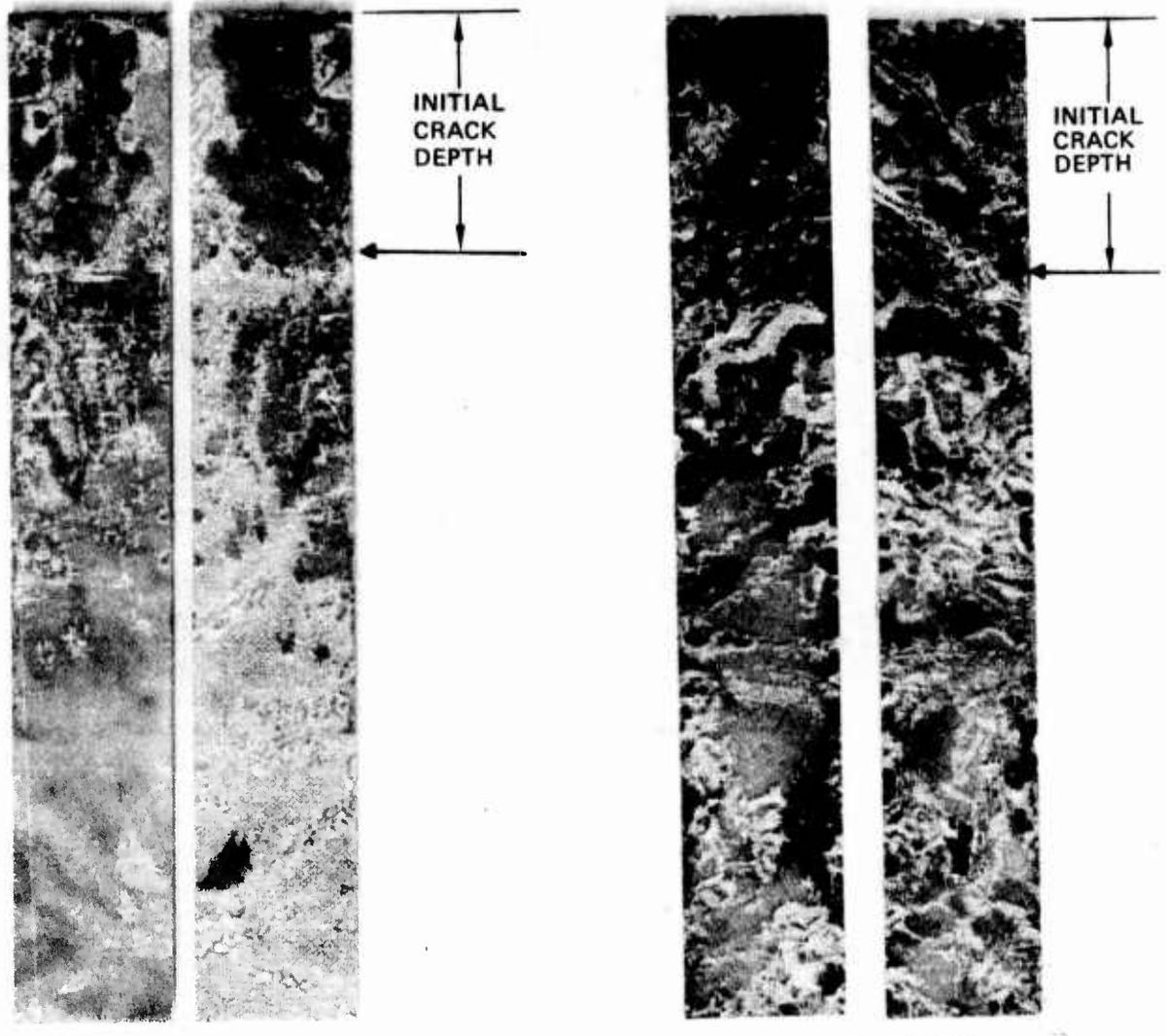


Figure 8: Titanium Crack Extension Specimen

TABLE IV - CRACK EXTENSION TEST RESULTS

ADHEREND	SURFACE TREATMENT	ADHESIVE	TYPE OF FAILURE		INITIAL CRACK LENGTH	CRACK GROWTH
			ROOM TEMPERATURE	ELEVATED TEMPERATURE		
Al	Phosphoric Acid Anodize	PKXA NR150A2	Cohesive (98-100)	Cohesive 98-100	1.54	None
			Cohesive (98-100)	Cohesive 98-100	1.52	None
	Sodium Dichromate Anodize	PKXA NR150A2	Cohesive (98-100)	Cohesive 98-100	1.54	None
			Cohesive (98-100)	Cohesive 98-100	1.47	None
	Sodium Dichromate - Sulfuric Acid Solution	PKXA NR150A2	Cohesive (98-100)	Cohesive 98-100	1.36	None
			Cohesive (98-100)	Cohesive 98-100	1.75	None
Stainless Steel	Sulfuric Acid Anodize	PKXA NR150A2	Cohesive (98-100)	Cohesive 98-100	1.7	None
			Cohesive (98-100)	Cohesive 98-100	1.46	.03
	Nitric Acid	PKXA NR150A2	Cohesive (40%)	Cohesive (20%)	1.71	.46
			Cohesive (98-100)	Cohesive (70%)	1.74	.15
	Alkaline	PKXA NR150A2	Cohesive (65%)	Cohesive (40%)	2.0	.8 Avg.
			Cohesive (85%)	Cohesive (55%)	3.16	None
Titanium	Chromic Acid Anodize	PKXA NR150A2	Cohesive (98-100)	Cohesive (95%)	.92	0.2
			Cohesive (25%)	Cohesive (5%)		
	Pasa-Gel	PKXA NR150A2	Cohesive (25%)	Cohesive (5%)	1.84	 .38
			Cohesive (75%)	Cohesive (65%)		
	Nitric-Hydrofluoric Acid Etch	PKXA NR150A2	Cohesive (75%)	Cohesive (65%)	2.24	.21
			Cohesive (85%)	Cohesive (25%)	1.74	.38

-  Initial crack with wedge
-  +300°F for PKXA and +500°F for NR150A2
-  Specimens fell apart during machining operations



200 HOURS AT R.T.

200 HOURS AT 300°F

Figure 9: Aluminum - Phosphoric Acid Anodize (PKXA)

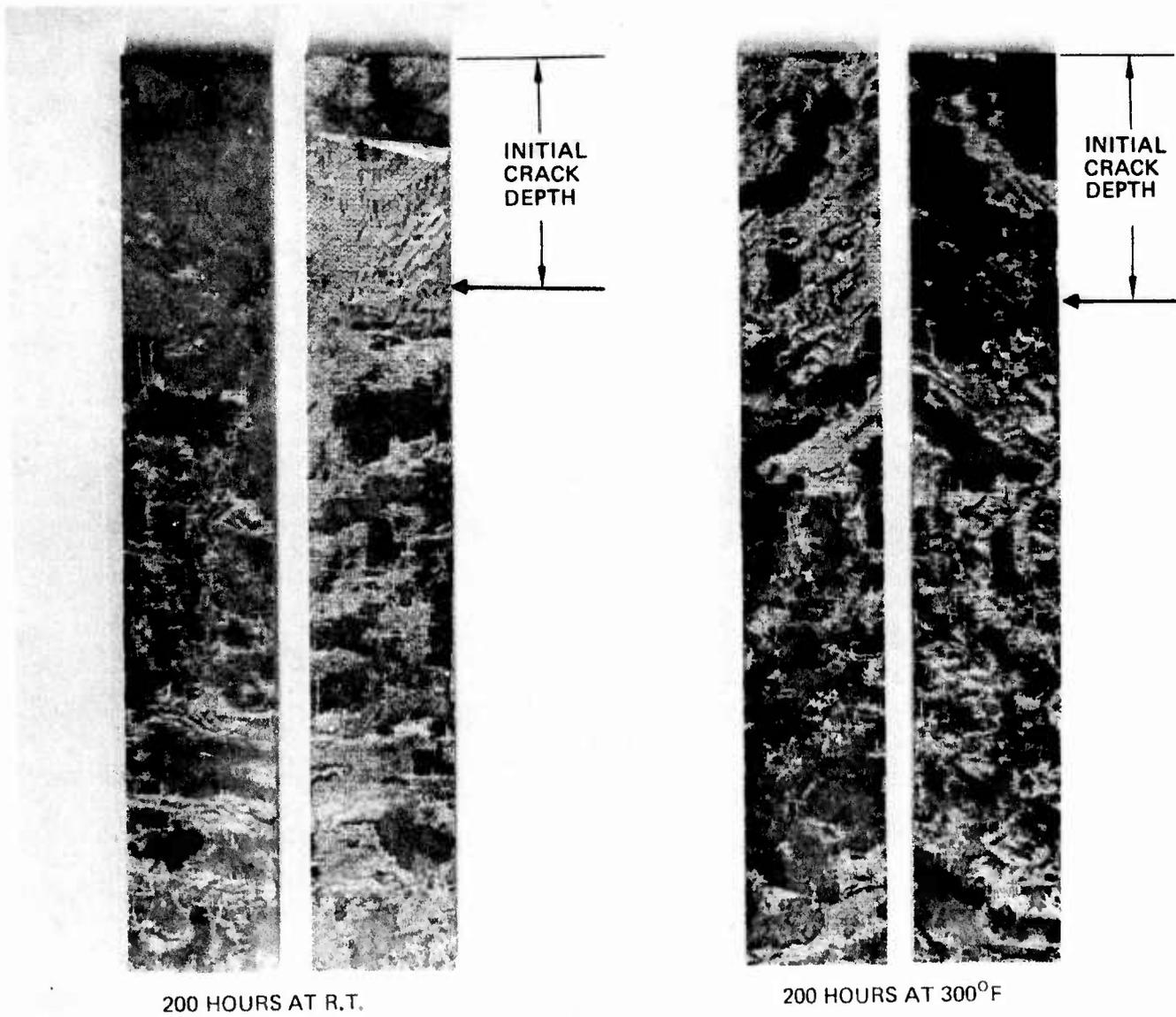
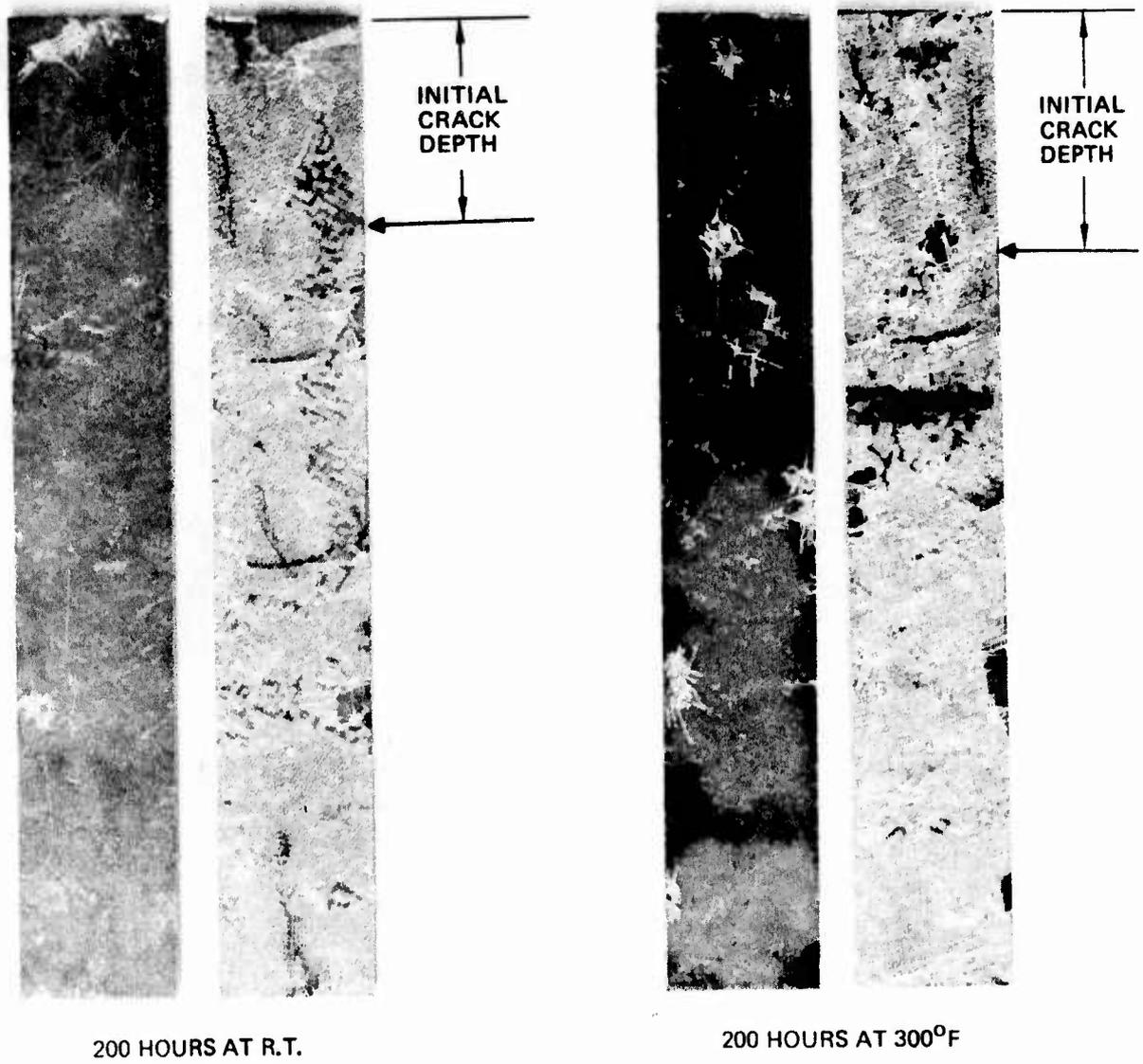


Figure 10: Aluminum - Chromic Acid Anodize (PKXA)



200 HOURS AT R.T.

200 HOURS AT 300°F

Figure 11: Aluminum - Sulfuric Acid - Sodium Dichromate ETCH (PKXA)



200 HOURS AT R.T.



200 HOURS AT 500°F

Figure 12: Aluminum - Chromic Acid Anodize (NR150A2)

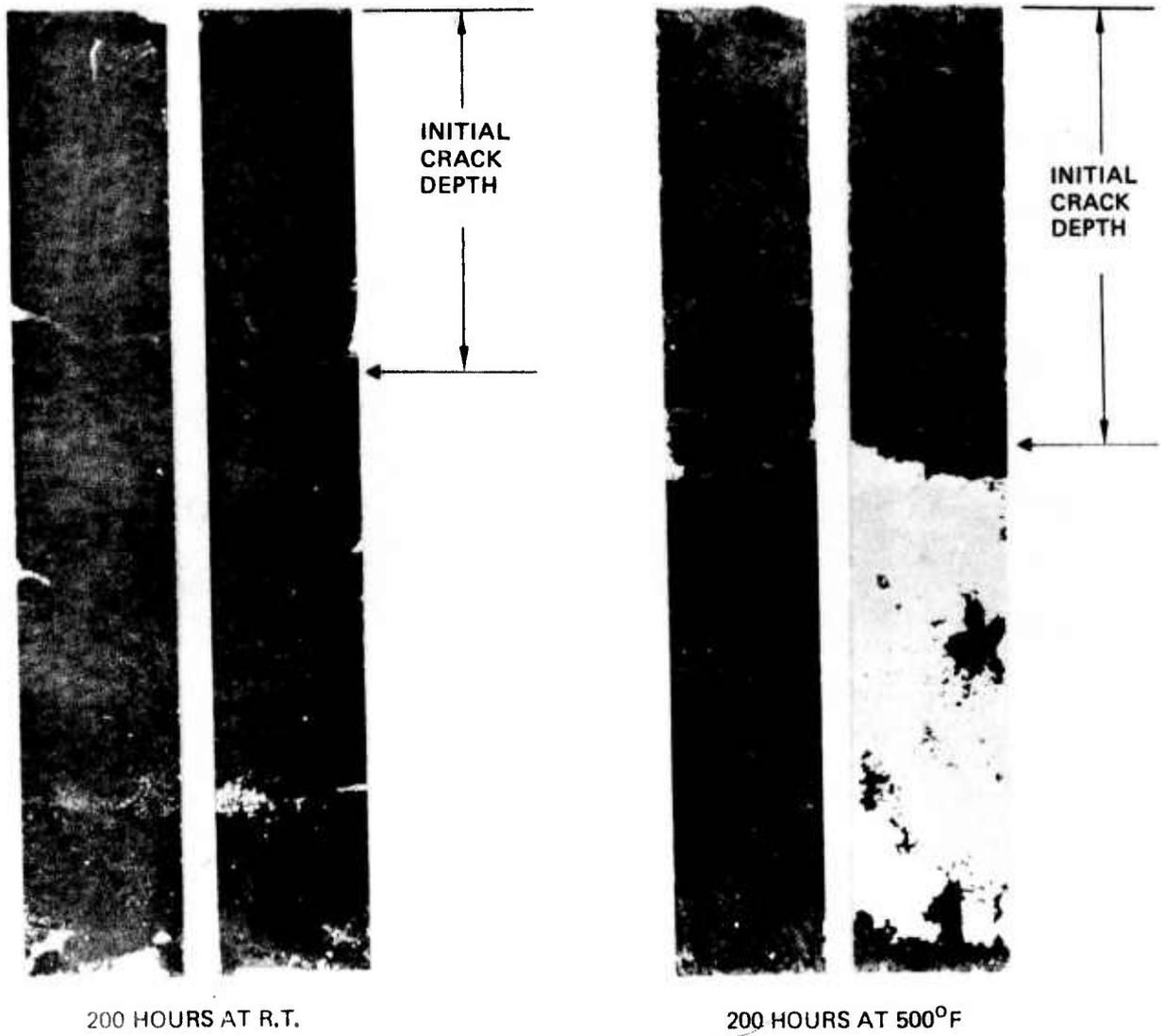


Figure 13: Aluminum - Sulfuric Acid - Sodium Dichromate ETCH (NR 150A2)

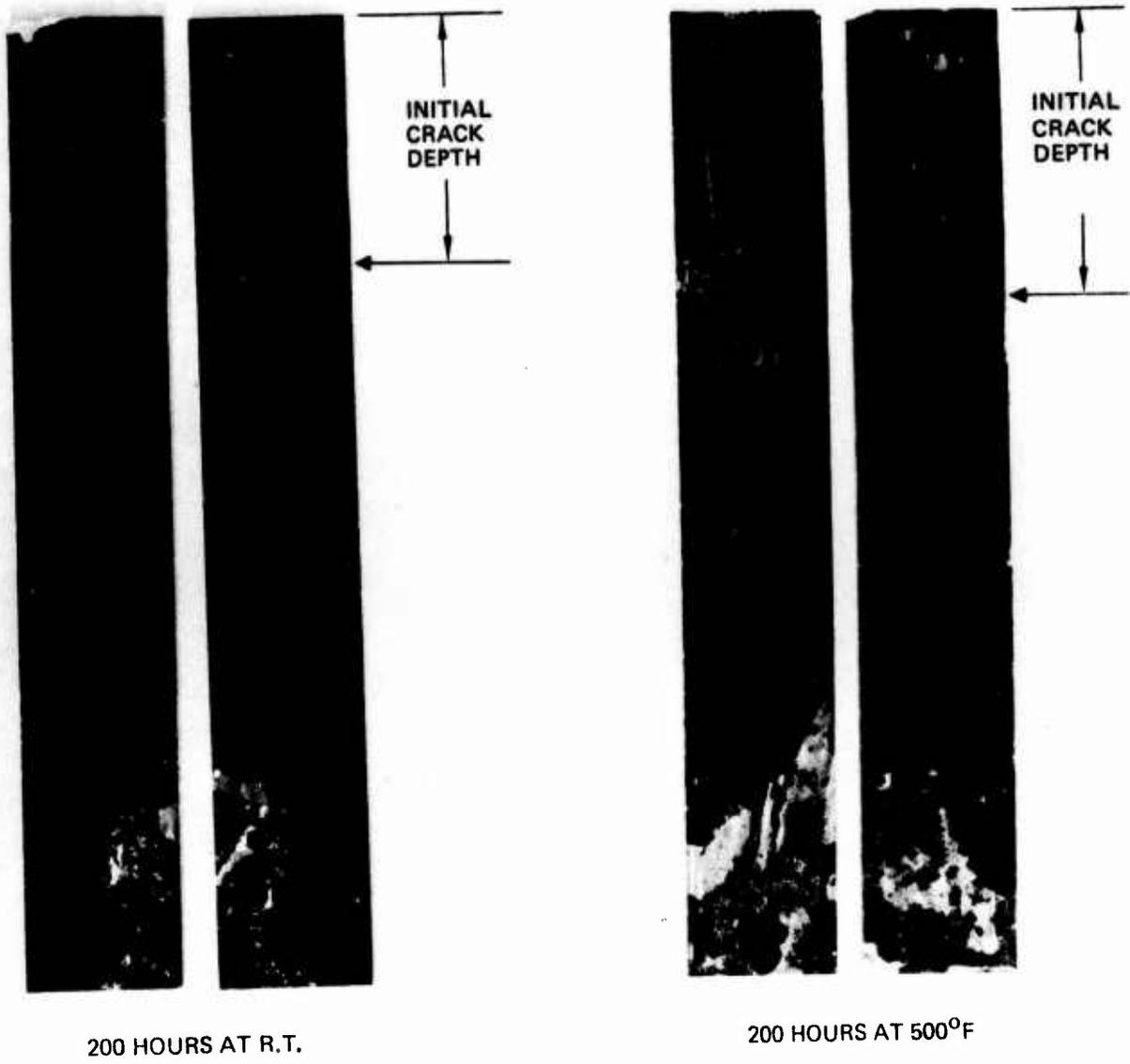


Figure 14: Aluminum - Phosphoric Acid Anodize (NR 150A2)

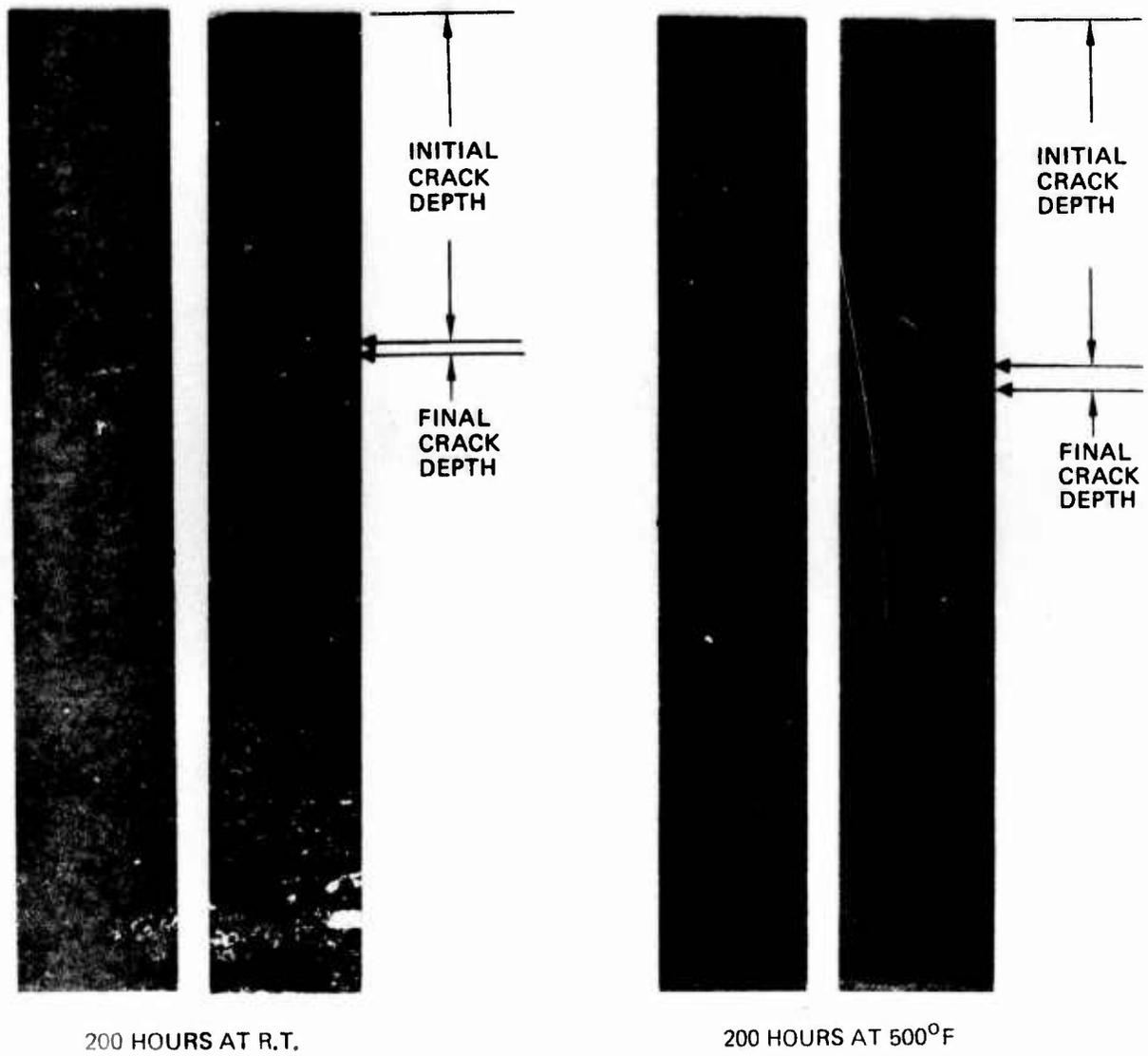


Figure 15: Stainless Steel - Sulfuric Acid Anodize (NR 150A2)

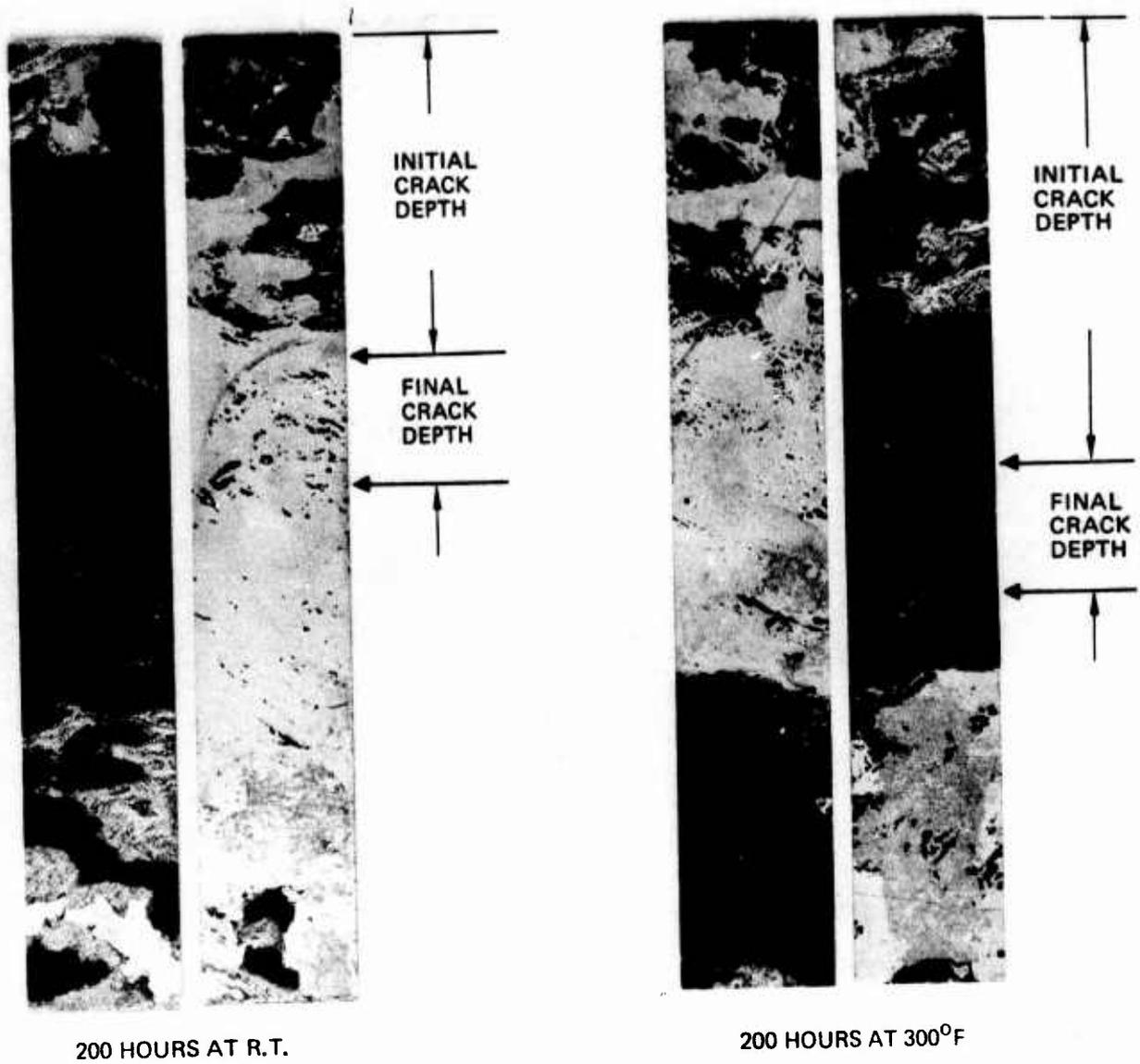
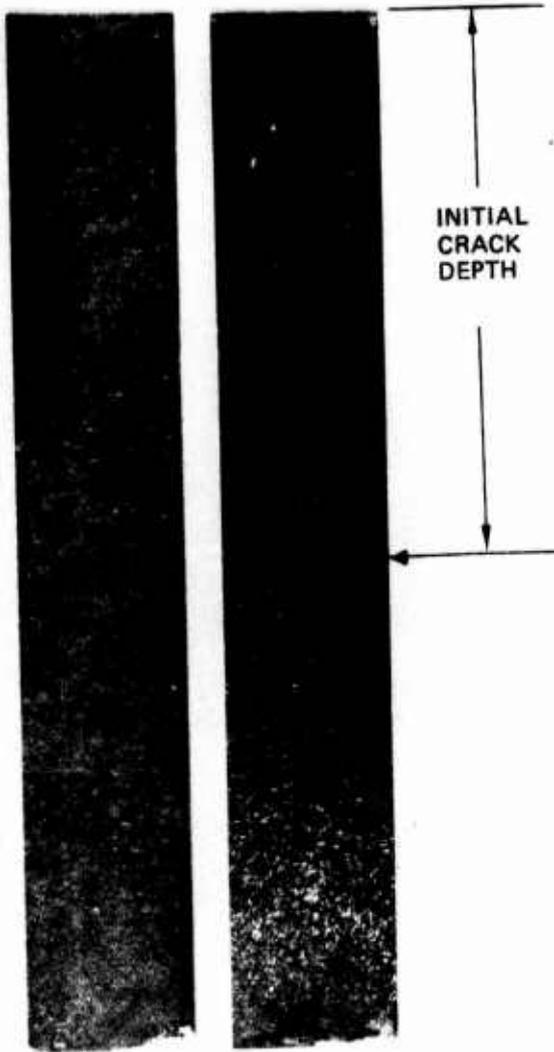
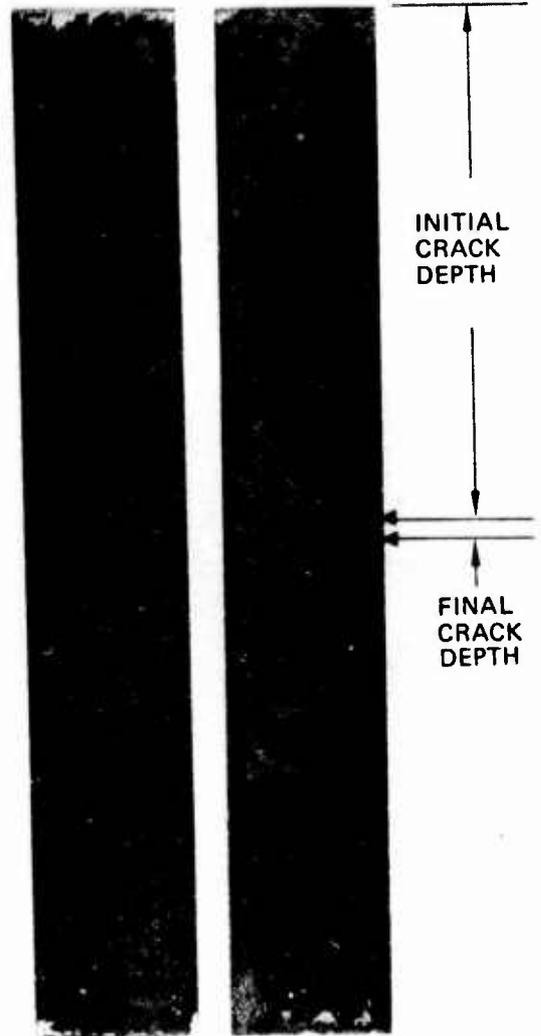


Figure 16: Stainless Steel - Alkaline Clean (PKXA)

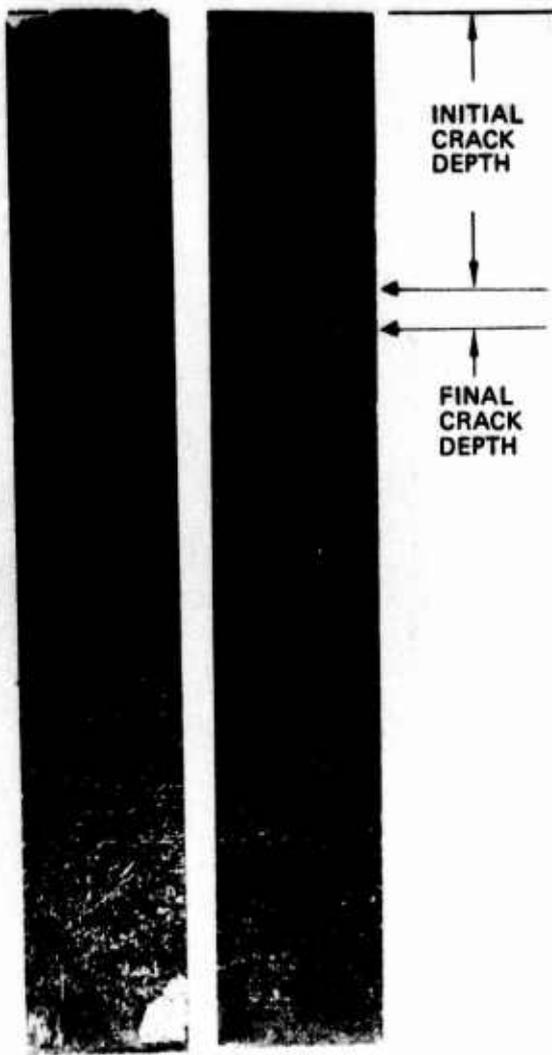


200 HOURS AT R.T.

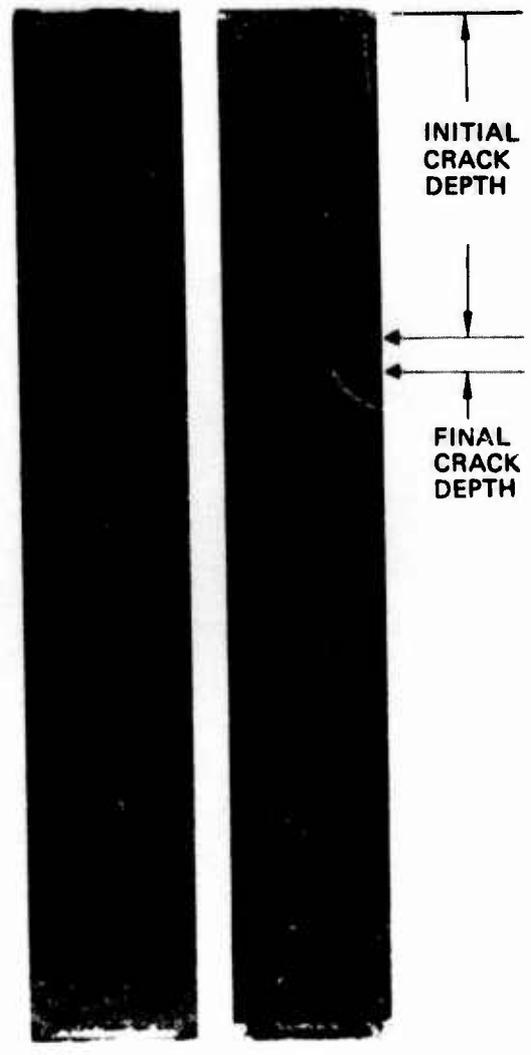


200 HOURS AT 500°F

Figure 17: Stainless Steel - Alkaline Clean (NR 150A2)



200 HOURS AT R.T.



200 HOURS AT 500°F

Figure 18: Stainless Steel - Nitric - Hydrofluoric Acid ETCH (NR 150A2)

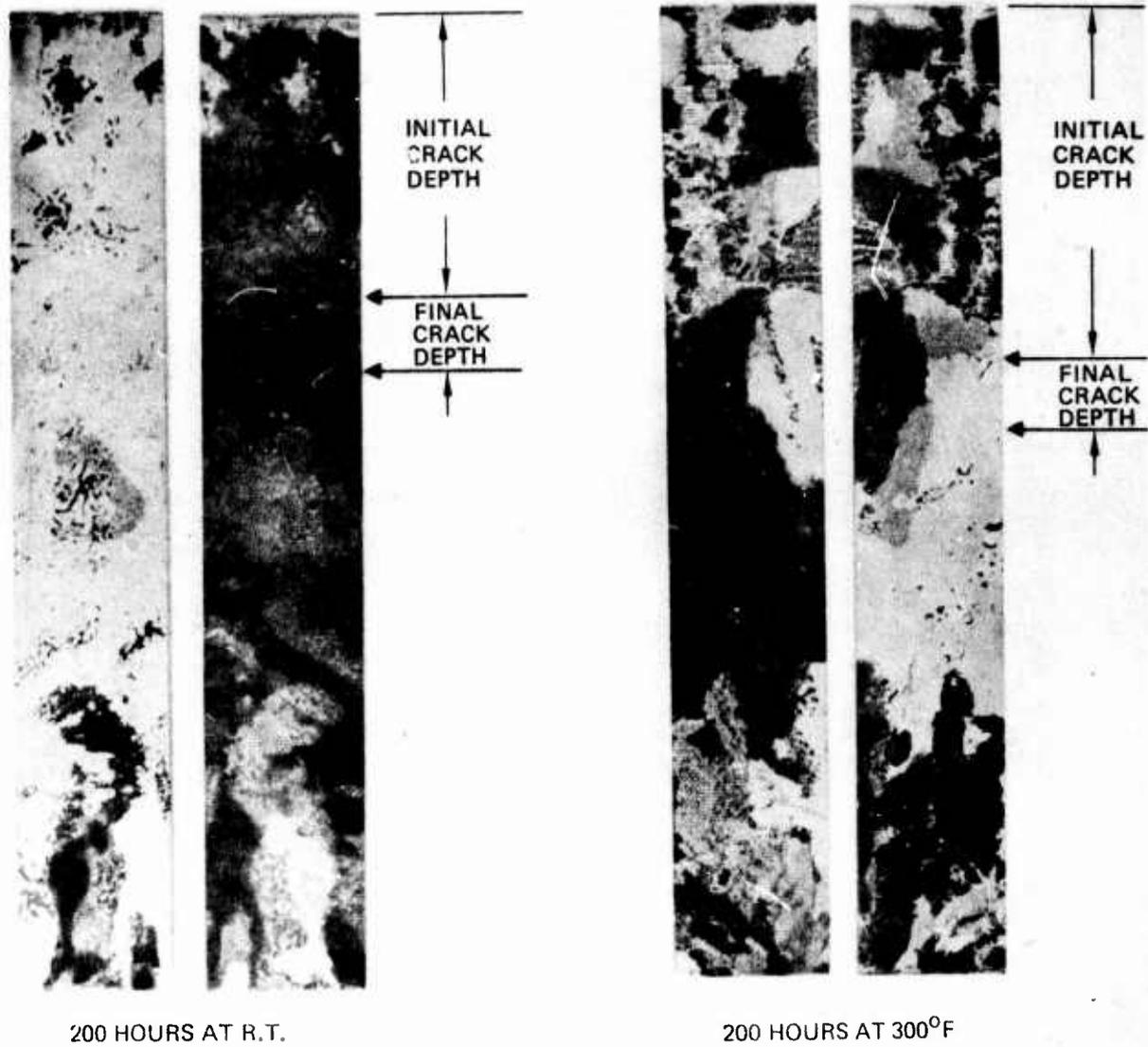


Figure 19: Stainless Steel - Nitric - Hydrofluoric Acid ETCH (PKXA)

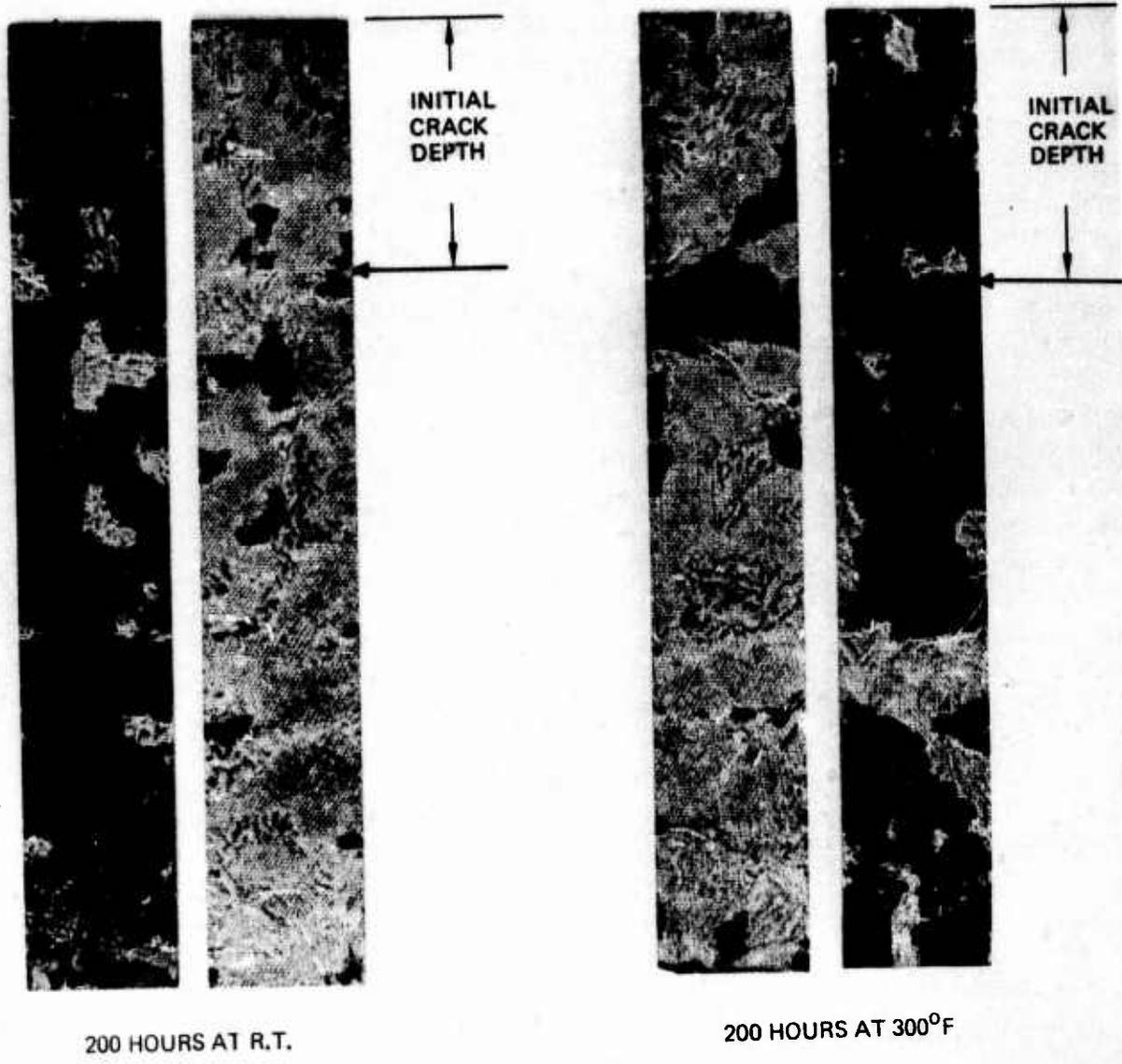
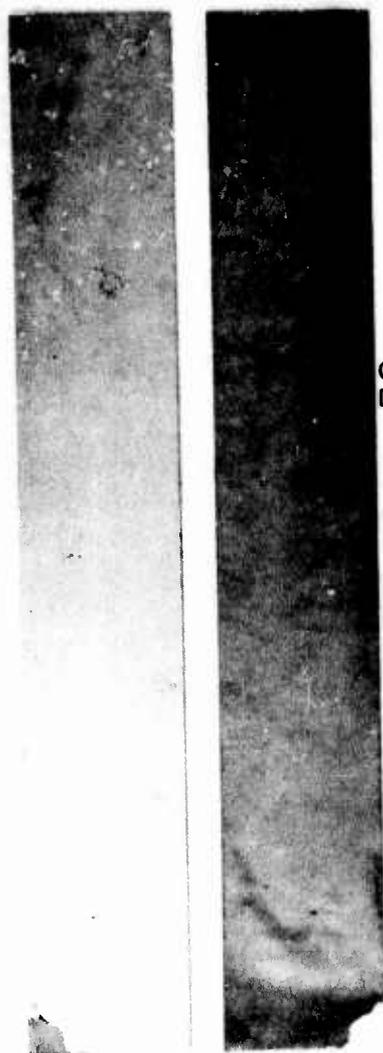
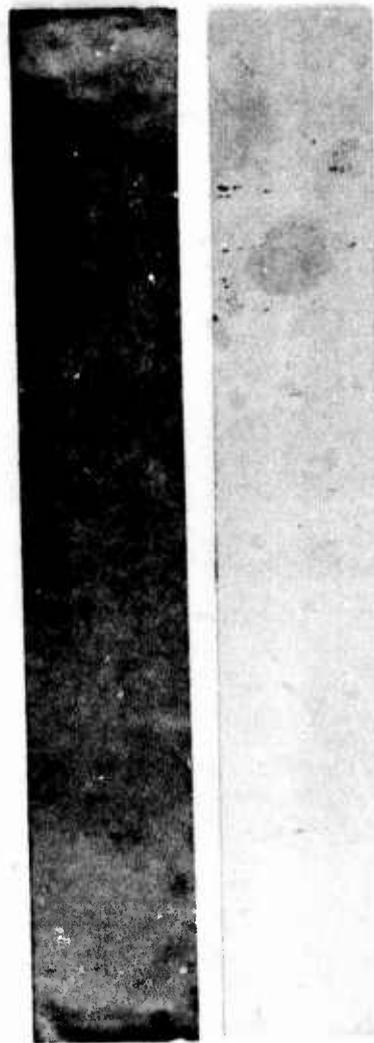


Figure 20: Stainless Steel - Sulfuric Acid Anodize (PKXA)



COMPLETE
DELAMINATION

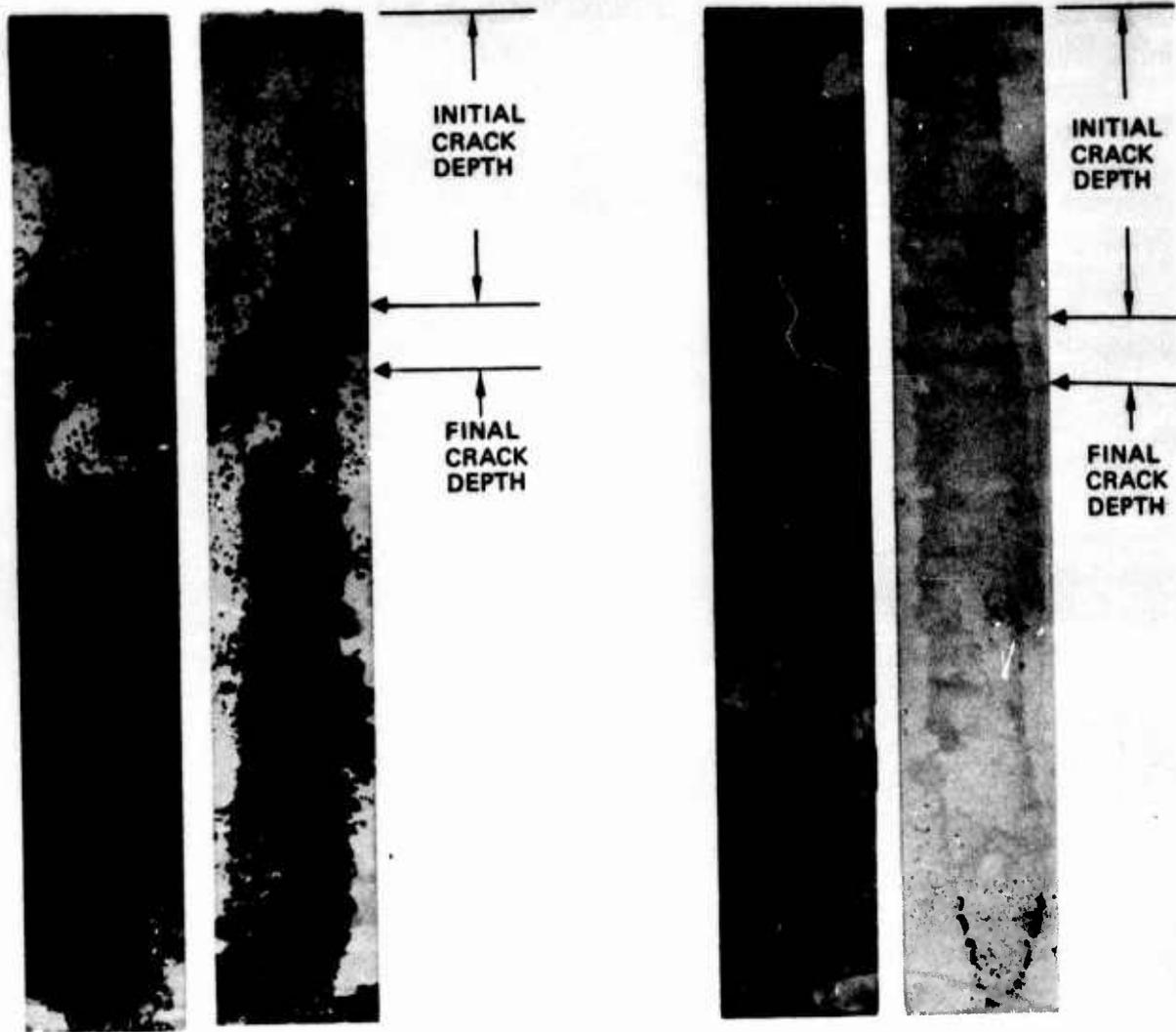
200 HOURS AT R.T.



COMPLETE
DELAMINATION

200 HOURS AT 500°F

Figure 21: Titanium - Chromic Acid Anodize (NR 150A2)



200 HOURS AT R.T.

200 HOURS AT 500°F

Figure 22: Titanium - PASA - Gel Surface Treatment (NR 150A2)

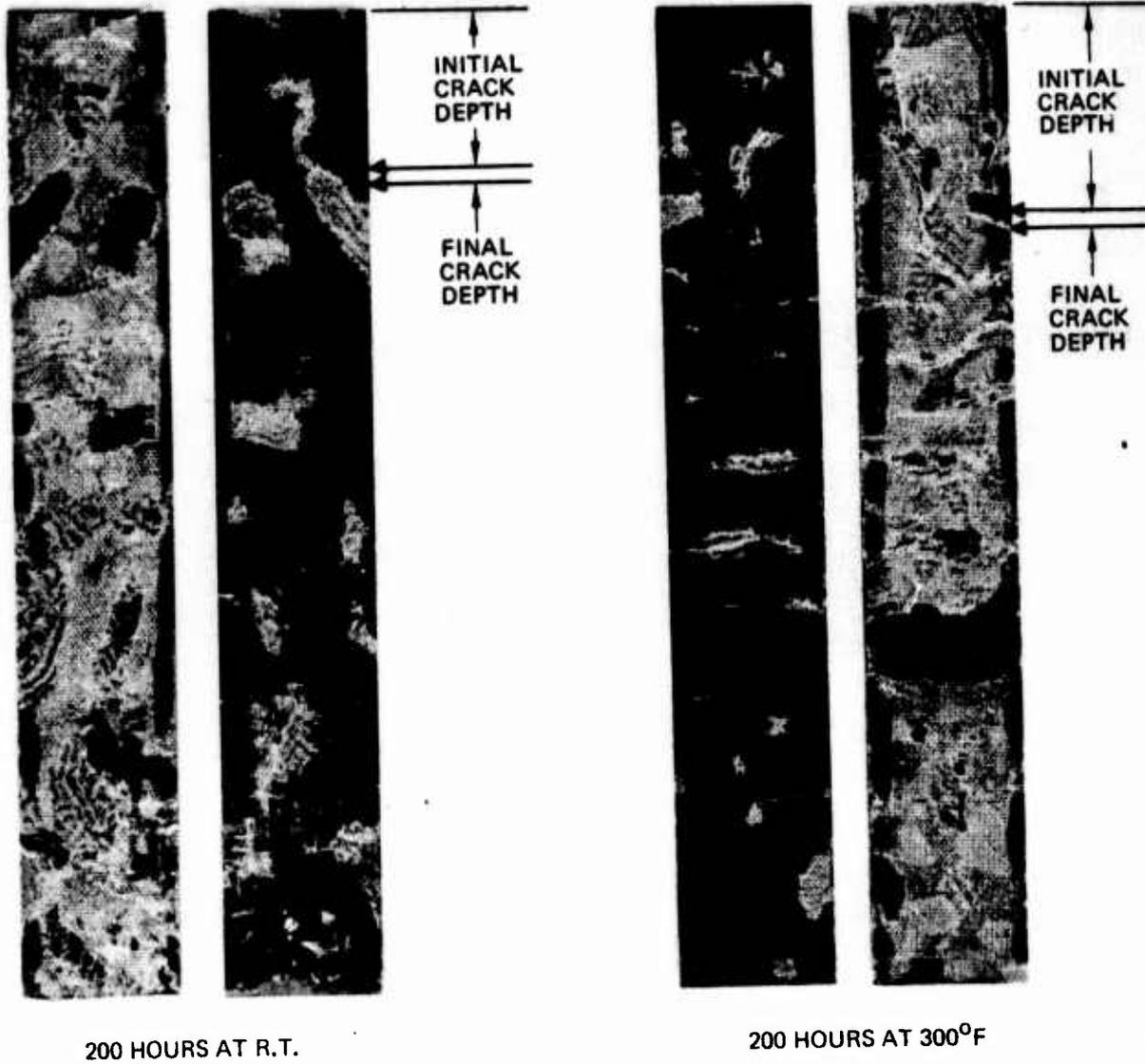
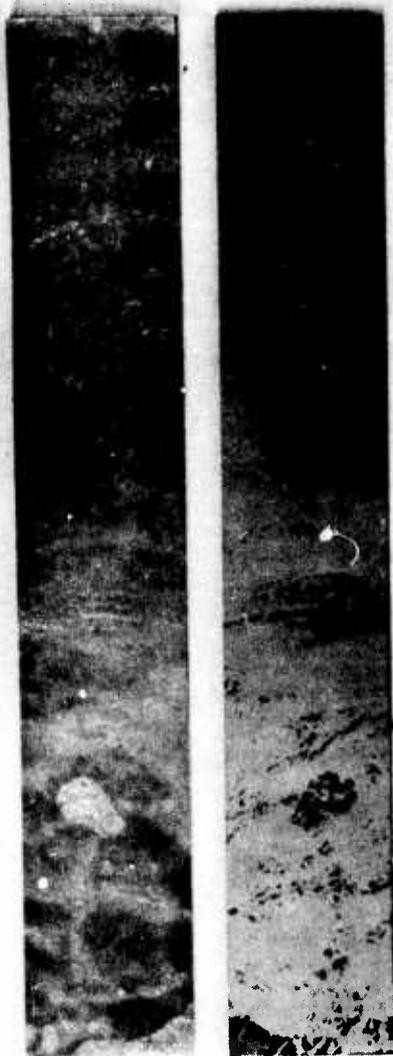
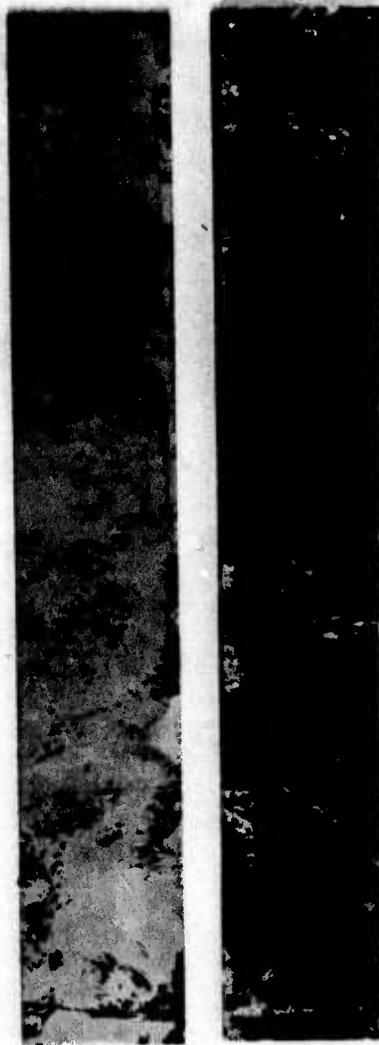


Figure 24: Titanium - Chromic Acid Anodize (PKXA)



COMPLETE
DELAMINATION

200 HOURS AT R.T.



COMPLETE
DELAMINATION

200 HOURS AT 300°F

Figure 25: Titanium - PASA - Gel Surface Treatment (PKXA)

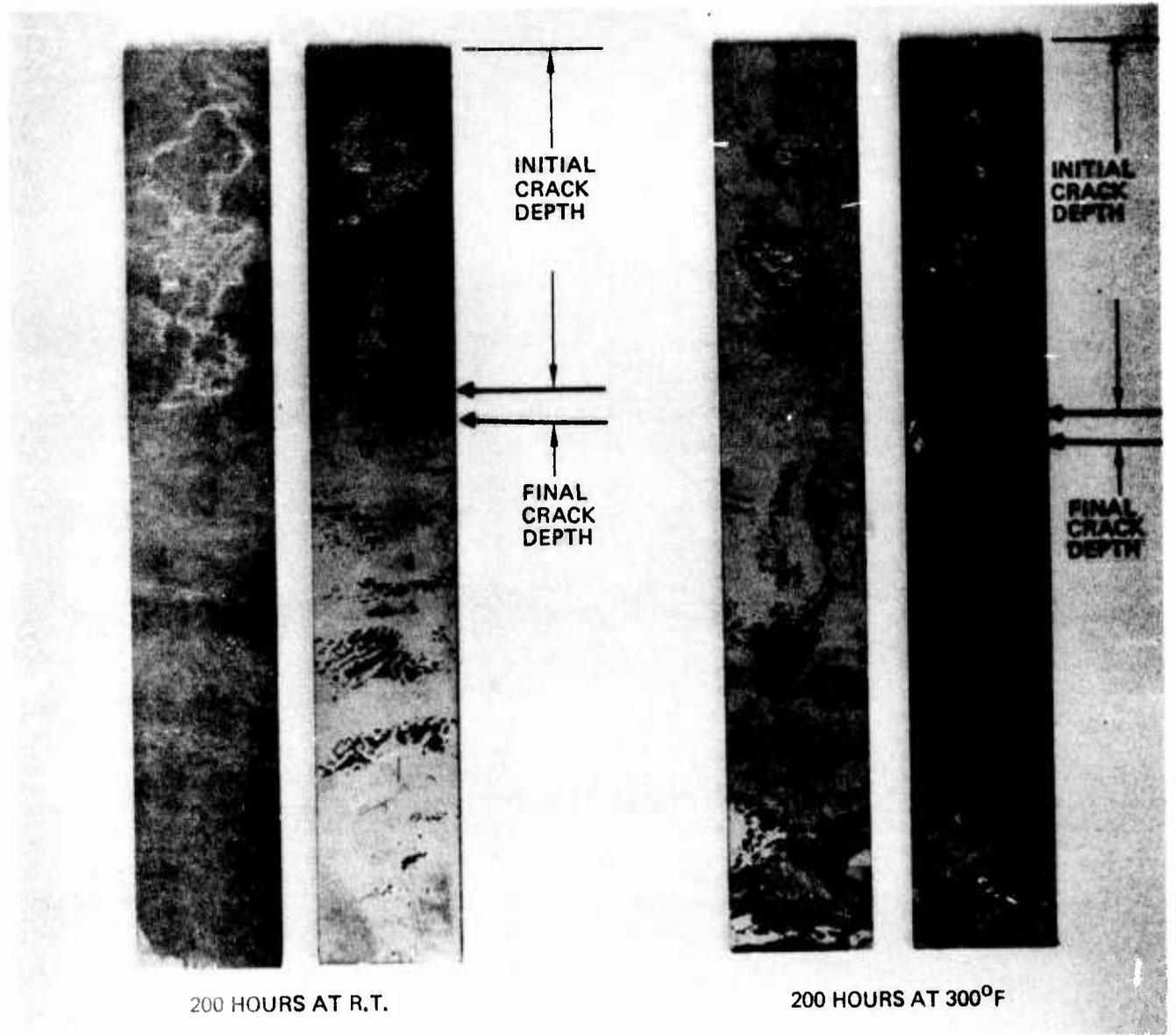


Figure 26: Titanium - Nitric - Hydrofluoric Acid ETCH (PKXA)

ADHEREND: 2024-T3 ALUMINUM

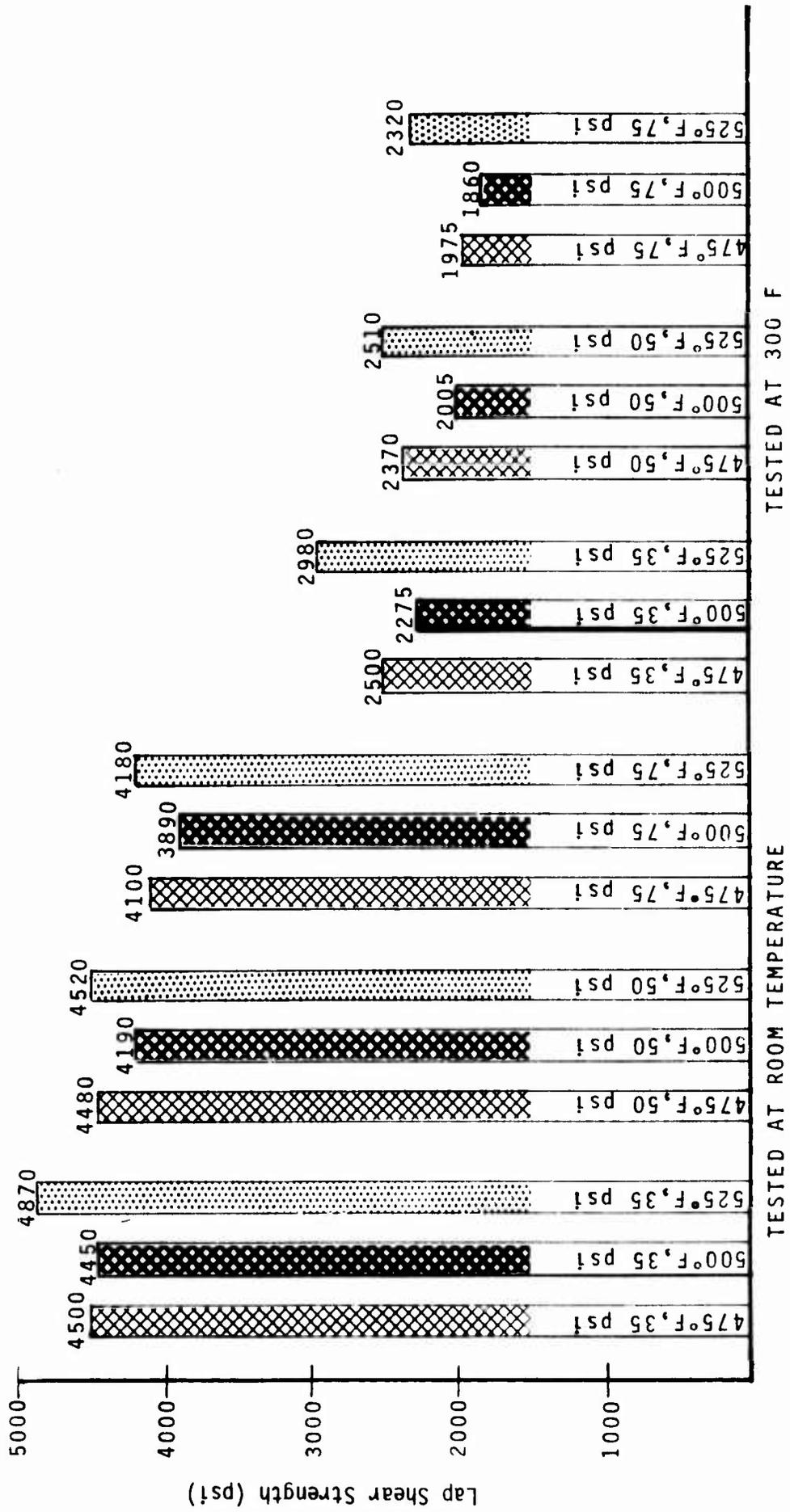


FIGURE 27: PKXA PROCESSING STUDIES-- PRESSURE AND TEMPERATURE

ADHERENDS: 2024-T3 ALUMINUM

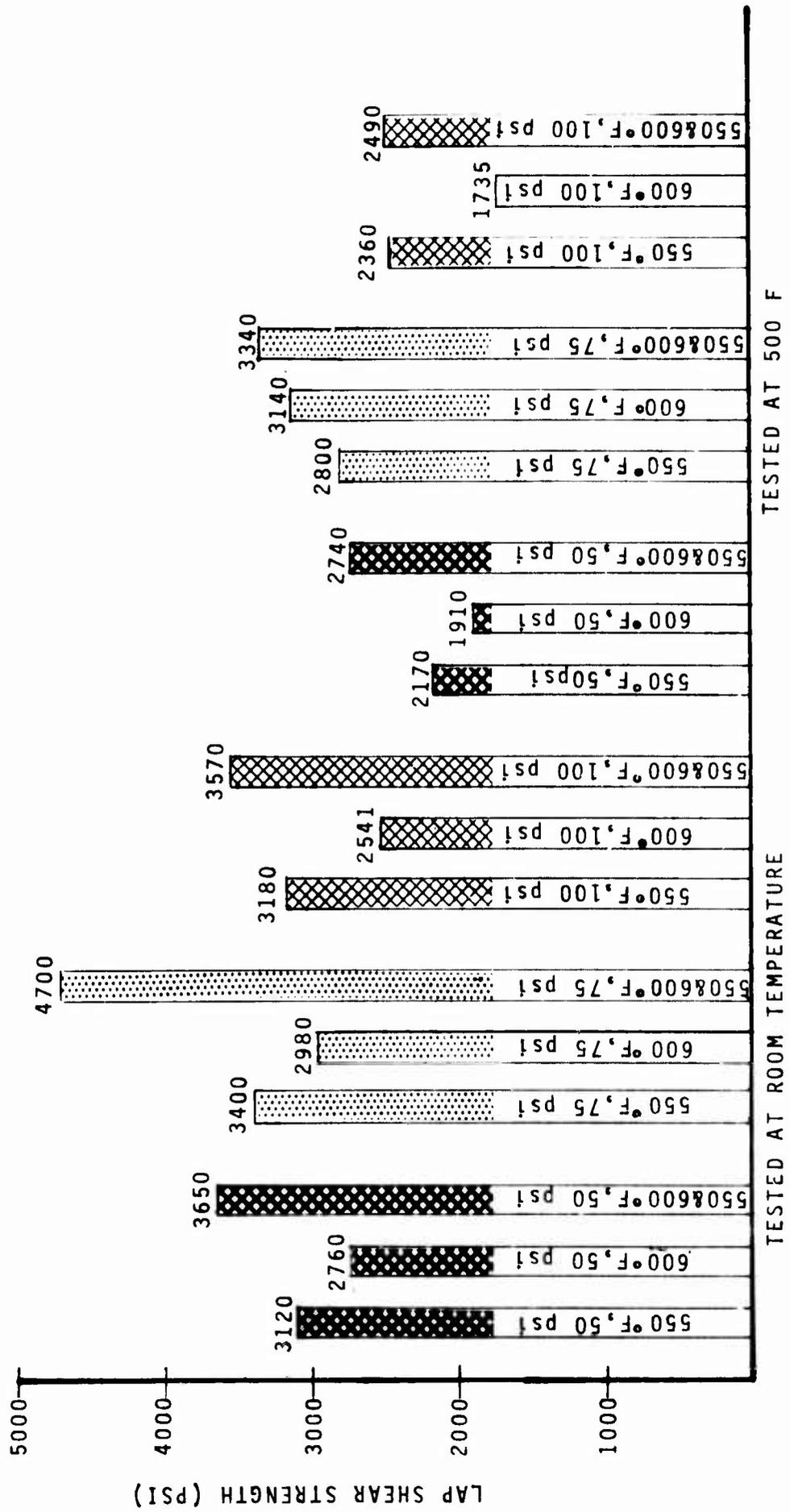


FIGURE 28: NR150A2 PROCESSING STUDIES-- PRESSURE AND TEMPERATURE

TABLE V - ADHESIVE MODIFICATION LAP SHEAR DATA - 2024-T3 ALUMINUM

Material	Bonding Temp. (°F)	Bonding Pressure (psi)	Average Shear Stress (psi)		Type of Failure % Cohesive
			RT	300°F	
A	475	35	1495	1515	85 - 95
B			2465	980	
C			1925	960	
D			2150	1645	
E			4525	1485	
F			2740	910	
G			2335	1325	
A	500		4200	1945	
B			1635	1745	
C			1575	1305	
D			2565	1955	
E			4400	835	
F			2850	350	
G			1835	815	
A	525		4215	3065	
B			1745	735	
C			1785	1325	
D			2770	1825	
E			4505	555	
F			2925	585	
G			2465	790	

A = PKXA

B = PKXA with 15% Amorphous Boron

C = PKXA with 15% Amorphous Boron and 5% PPS

D = PKXA with 15% Powdered Aluminum

E = PKXA with 15% Powdered Aluminum and 5% PPS

F = PKXA with 15% Amorphous Boron and 3% Powdered Aluminum

G = PKXA with 15% Amorphous Boron and 3% PPS

TABLE VI - ADHESIVE MODIFICATION LAP SHEAR DATA

Material	Bonding Temp. (°F)	Bonding Pressure (psi)	Average Shear Stress (psi) RT	Average Shear Stress (psi) 300°F	Type of Failure % Cohesive
A	475	50	4680	1505	85 - 95
B			3025	1185	
C			1915	815	
D			4695	2445	
E			2445	1065	
F			2740	910	
G			1378	745	
A	500	50	4160	1775	85 - 95
B			2225	1995	
C			1005	770	
D			4745	2555	
E			2053	1215	
F			3040	445	
G			1948	420	
A	525	50	3935	1715	85 - 95
B			2235	2215	
C			1615	1185	
D			4150	1885	
E			1883	650	
F			2950	1215	
G			1854	935	

A = PKXA

B = PKXA with 15% Amorphous Boron

C = PKXA with 15% Amorphous Boron and 5% PPS

D = PKXA with 15% Powdered Aluminum

E = PKXA with 15% Powdered Aluminum and 5% PPS

F = PKXA with 15% Amorphous Boron and 3% Powdered Aluminum

G = PKXA with 15% Amorphous Boron and 3% PPS

ADHEREND: ALUMINUM

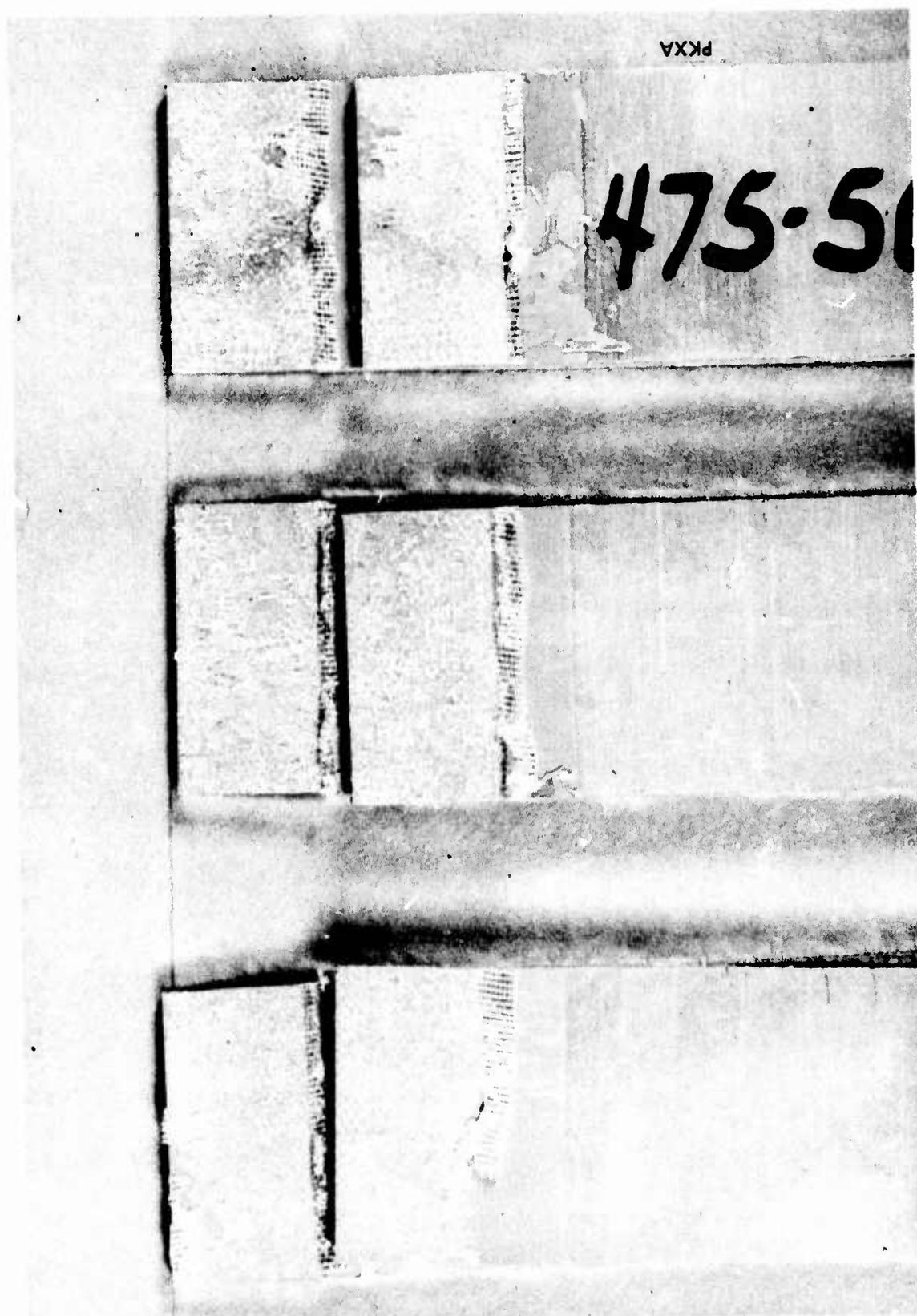
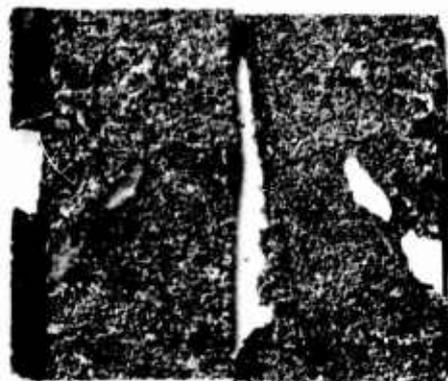


Figure 29: Modification A - Typical Lap Shear Specimens

BORON - FILLED PKXA



0-1



Figure 30: Modification B - Typical Failed Lap Shear Specimens

BORON + PPS FILLED

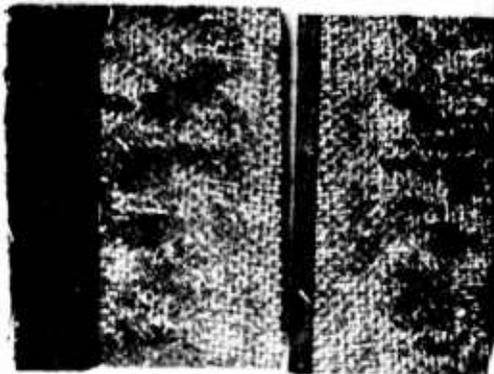
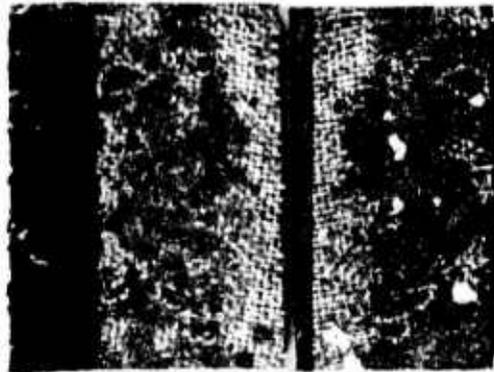
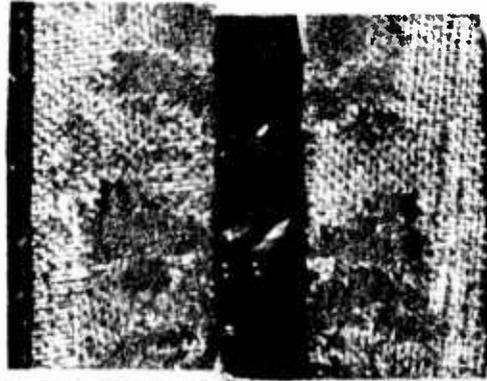


Figure 31: Modification C - Typical Failed Lap Shear Specimens

ALUMINUM - FILLED PKXA

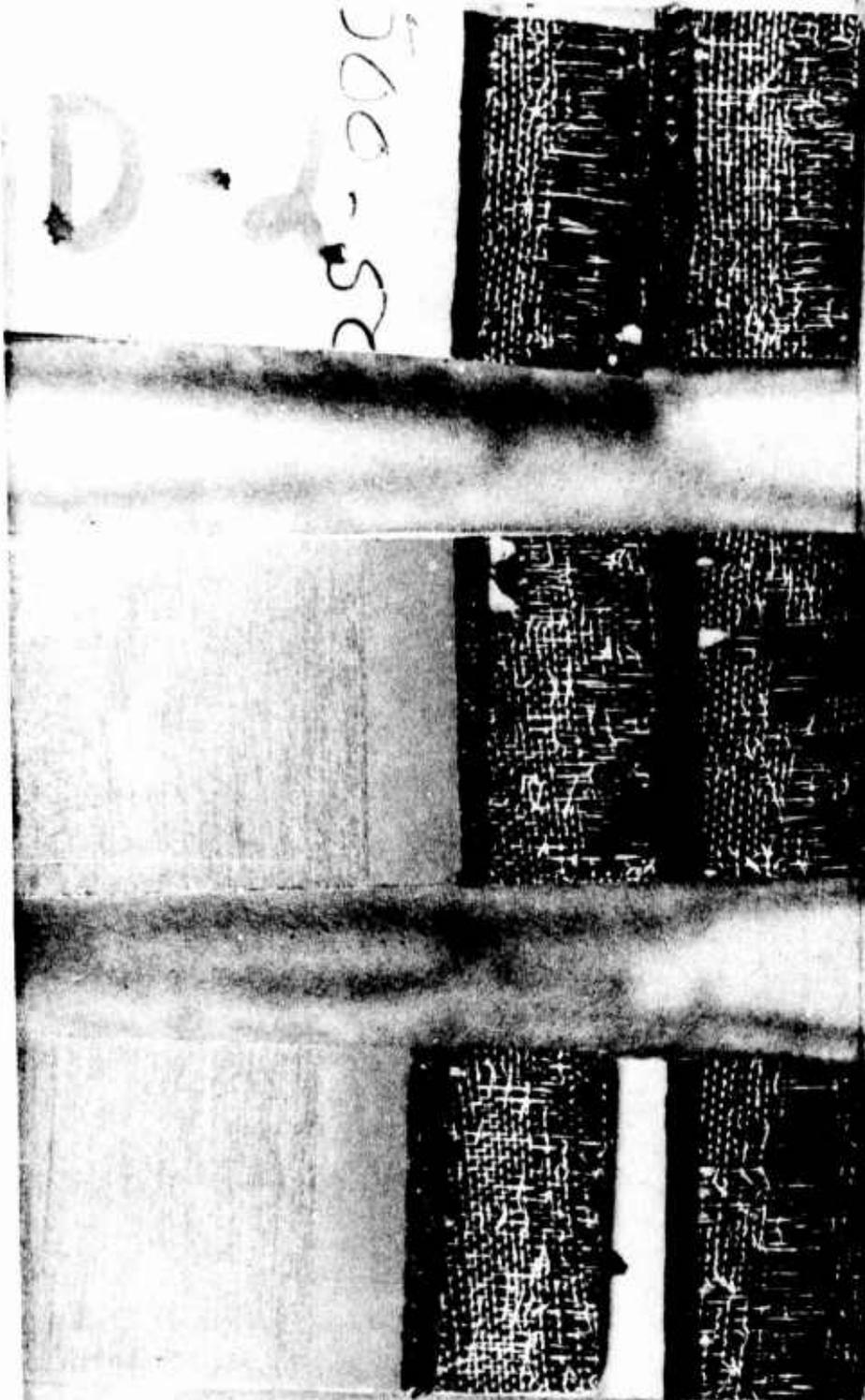


Figure 32: Modification D - Typical Failed Lap Shear Specimens

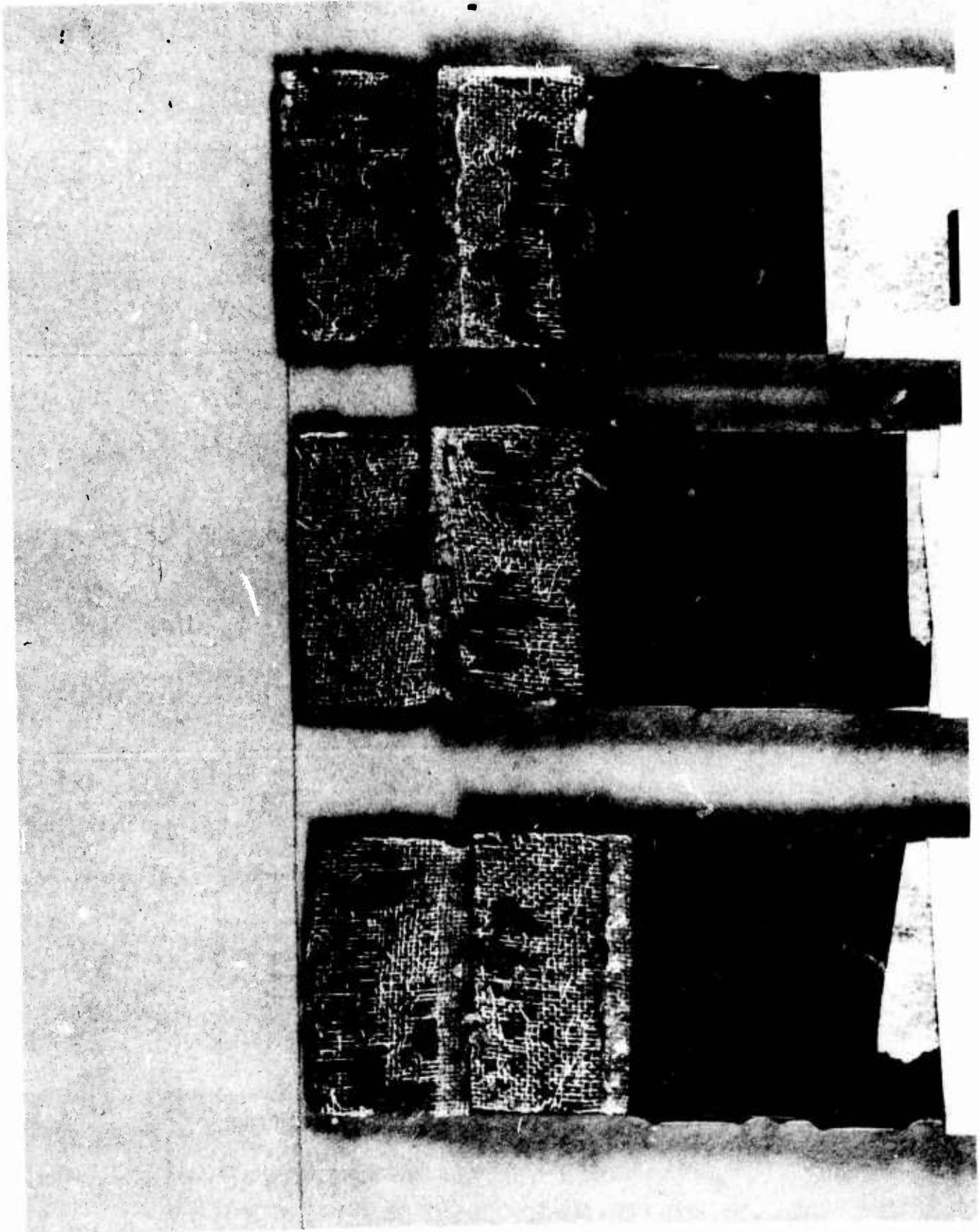
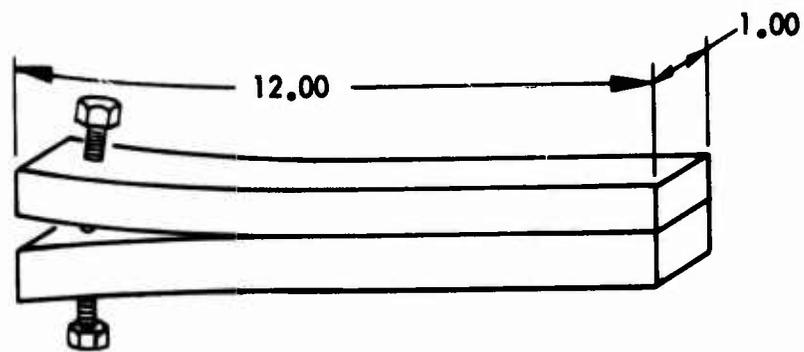
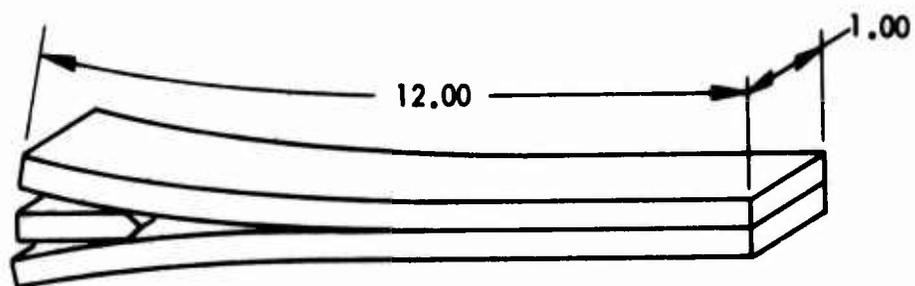


Figure 33: Modification E - Typical Failed Lap Shear Specimens



a: BOLT LOADED SPECIMEN



b: WEDGE LOADED SPECIMEN

Figure 34 : SELF-CONTAINED DCB SPECIMENS WITH CONSTANT DISPLACEMENT LOADING

TABLE VII - THE EFFECTS OF THERMAL AGING ON PKXA AND NR150A2 BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Aging Temp., °F	Adhesive	Hours Aging	Average Crack Growth (inches)	Crack Range (inches)
Steel	300	PKXA	10	NC	-
	300		100	NC	-
	300		1000	NC	-
Aluminum	300	PKXA	10	NC	-
	300		100	1.16	NC to 2.00
	300		1000	NC	-
Titanium	300	PKXA	10	NC	-
	300		100	NC	-
	300		1000	.16	NC to .32
Steel	500	NR150A2	10	.33	NC to 1.00
	500		100	1.50	.50 to 2.50
	500		1000	1.16	1.00 to 1.25
Aluminum	500	NR150A2	10	1.60	1.00 to 2.25
	500		100	3.67	3.00 to 4.00
	500		1000	1.67	1.25 to 2.00
Titanium	500	NR150A2	10	.83	NC to 1.50
	500		100	2.16	1.75 to 2.75
	500		1000	2.05	.60 to 3.00

NC = No Change

- = No Range

* Represents Avg. Control Value for Both Systems

Aging Temp.: PKXA = 300°F
NR150 = 500°F

Tested @ +70°F

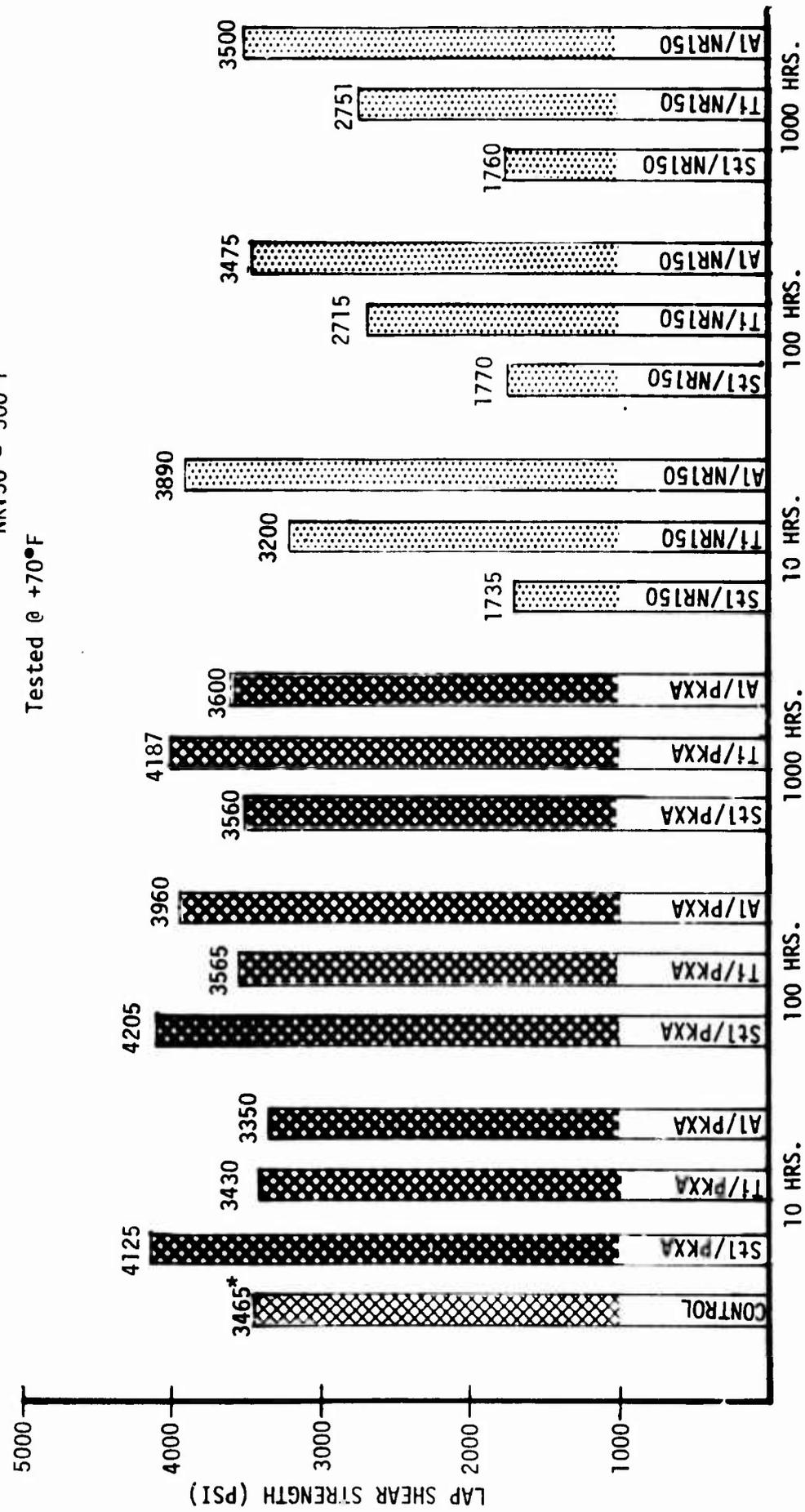


Figure 35: THERMAL AGING OF PKXA AND NR150A2 ADHESIVES

TABLE VIII - THE EFFECT OF SALT WATER ON NR150A2 and PKXA BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Adhesive	Aging Temp. (°F)	Hours Aging	Average Crack Growth (inches)	Crack Range (inches)
Steel	NR150A2	100 ± 5°F	200	0.25	NC to 0.50
Titanium			200	NC	NC
Aluminum			200	NC	NC
Steel	PKXA	100 ± 5°F	200	NC	-
Titanium			200	NC	-
Aluminum			200	0.58	NC to 1.75

NC = No Change

- = No Range

* Represents Avg. Control Value for Both Systems

Temp. = $100 \pm 5^\circ\text{F}$

Conc. = $5 \pm .5\%$

Tested at $+70^\circ\text{F}$

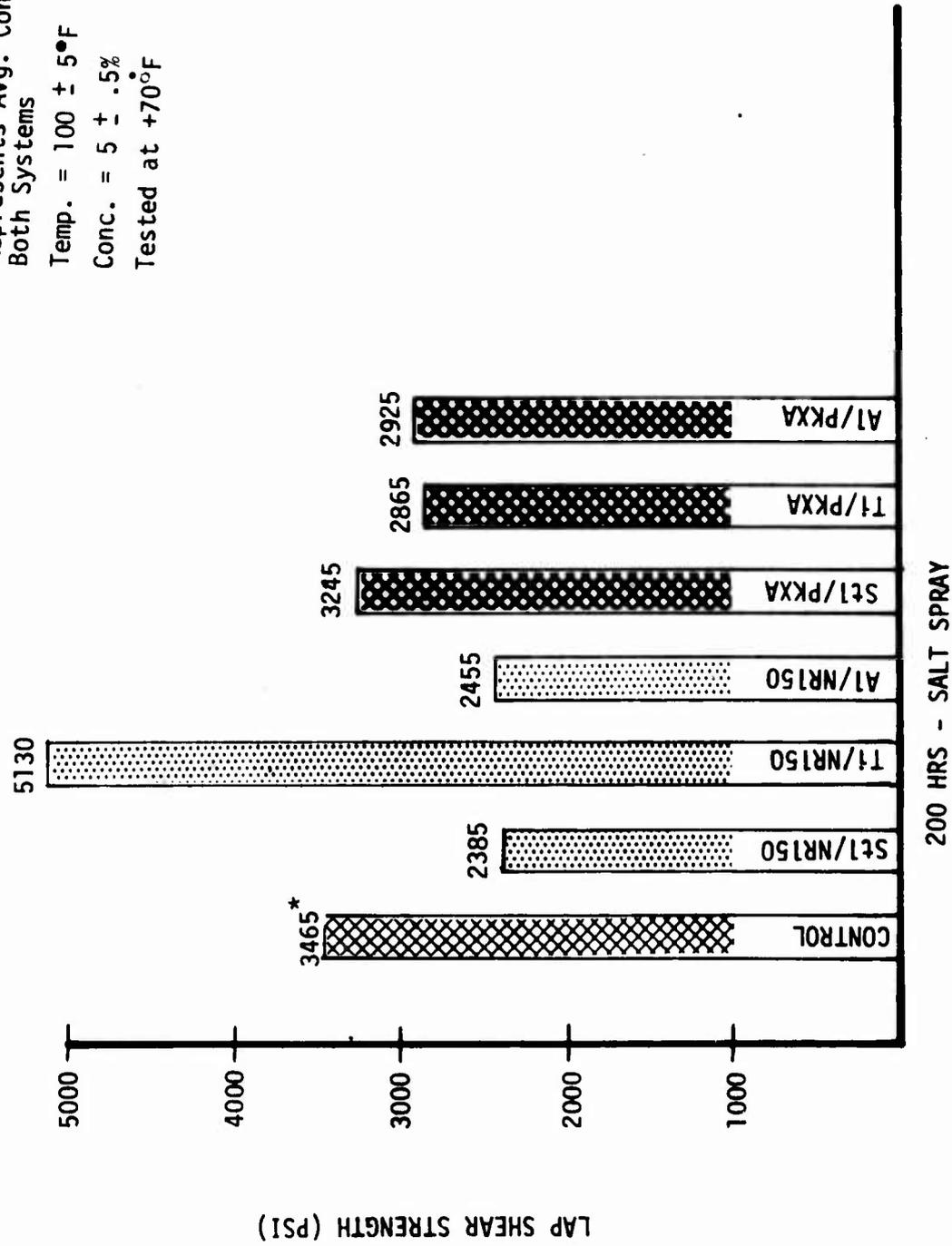


Figure 36: THE EFFECT OF SALT ENVIRONMENT ON PKXA AND NR150A2 ADHESIVES

TABLE IX - THE EFFECTS OF HUMIDITY ENVIRONMENT ON PKXA AND NR150A2 BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Adhesive	Aging Temp. (°F)	Days Aging	Average Crack Growth (inches)	Crack Range (inches)
Steel	PKXA	120	10	NC	-
Aluminum			24	NC	-
Titanium			10	0.33	-
			24	1.28	0.50 to 2.00
			10	NC	-
			24	0.25	-
Steel	NR150A2		10	0.87	0.75 to 1.00
Aluminum			24	1.12	1.00 to 1.25
Titanium			10	0.33	0.25 to 0.50
			24	0.58	NC to 1.00
			10	0.37	0.25 to 0.50
			24	0.60	0.40 to 0.80

NC = No Change
 - = No Range

* Represents Avg. Control Value for both systems
Tested at +70°F

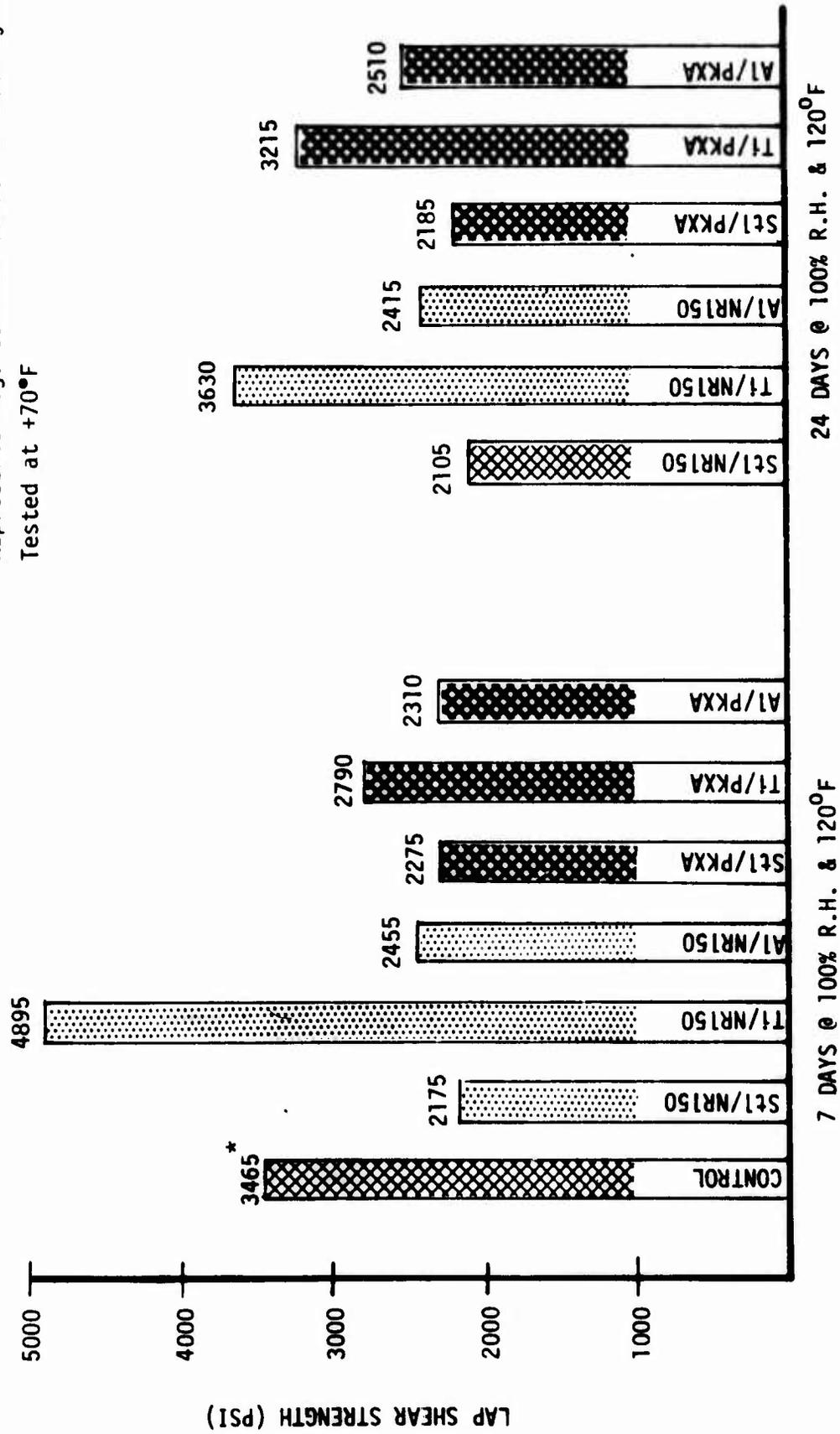


Figure 37: THE EFFECT OF HUMIDITY ON PKXA AND NR150A2 ADHESIVES

TABLE X - THE EFFECT OF JP-4 ON NR150A2 BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	3.30	2.50 to 4.00
	24	RT	3.00	2.00 to 4.00
	10	160	2.20	2.00 to 2.52
	24	160	2.70	1.50 to 3.00
Aluminum	10	RT	2.70	1.00 to 3.00
	24	RT	Delamination	-
	10	160	3.80	3.00 to 4.50
	24	160	3.40	2.75 to 4.00
Titanium	10	RT	2.20	1.50 to 3.00
	24	RT	0.50	0.30 to 0.75
	10	160	1.00	-
	24	160	2.62	1.25 to 4.00

- = No Range

* Represents Avg Control Value for both systems
 Testd Tested at +70°F

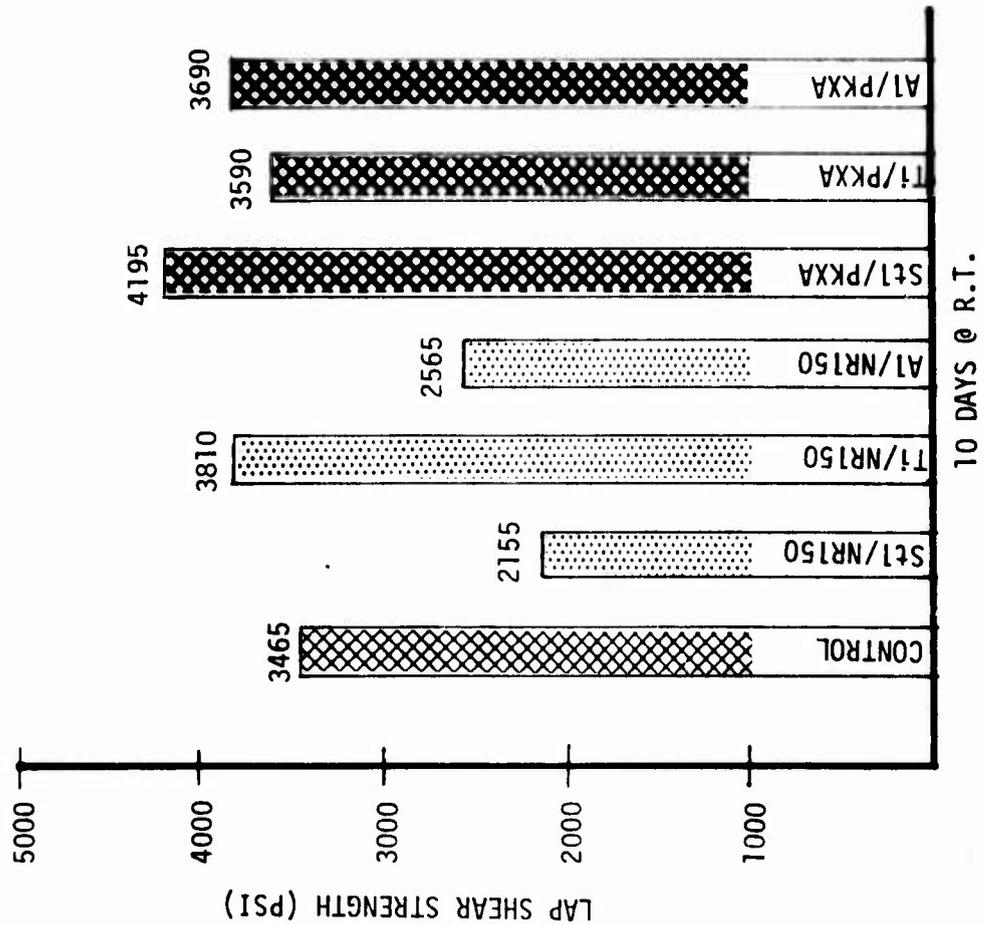
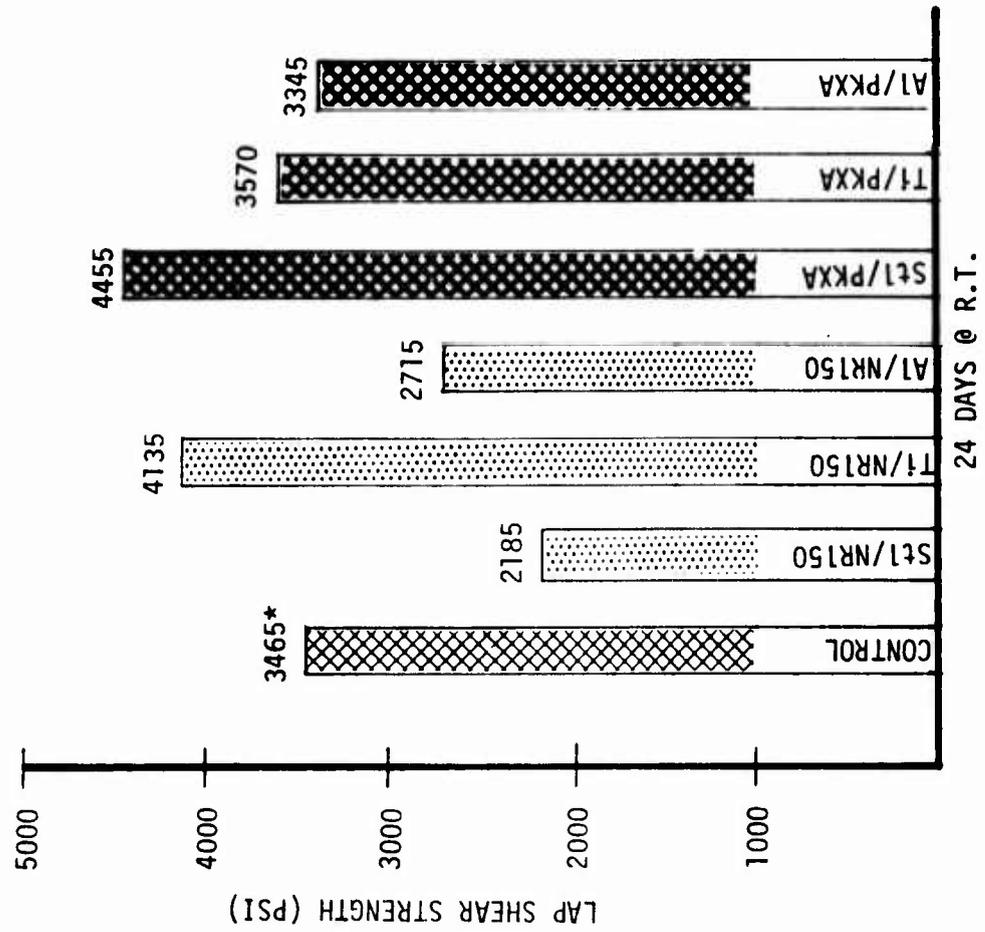


Figure 38: THE EFFECT OF JP-4 ON PKXA AND NR150A2 LAP SHEAR SPECIMENS

* Represents Avg. Control Value for Both Systems
Tested at +70°F

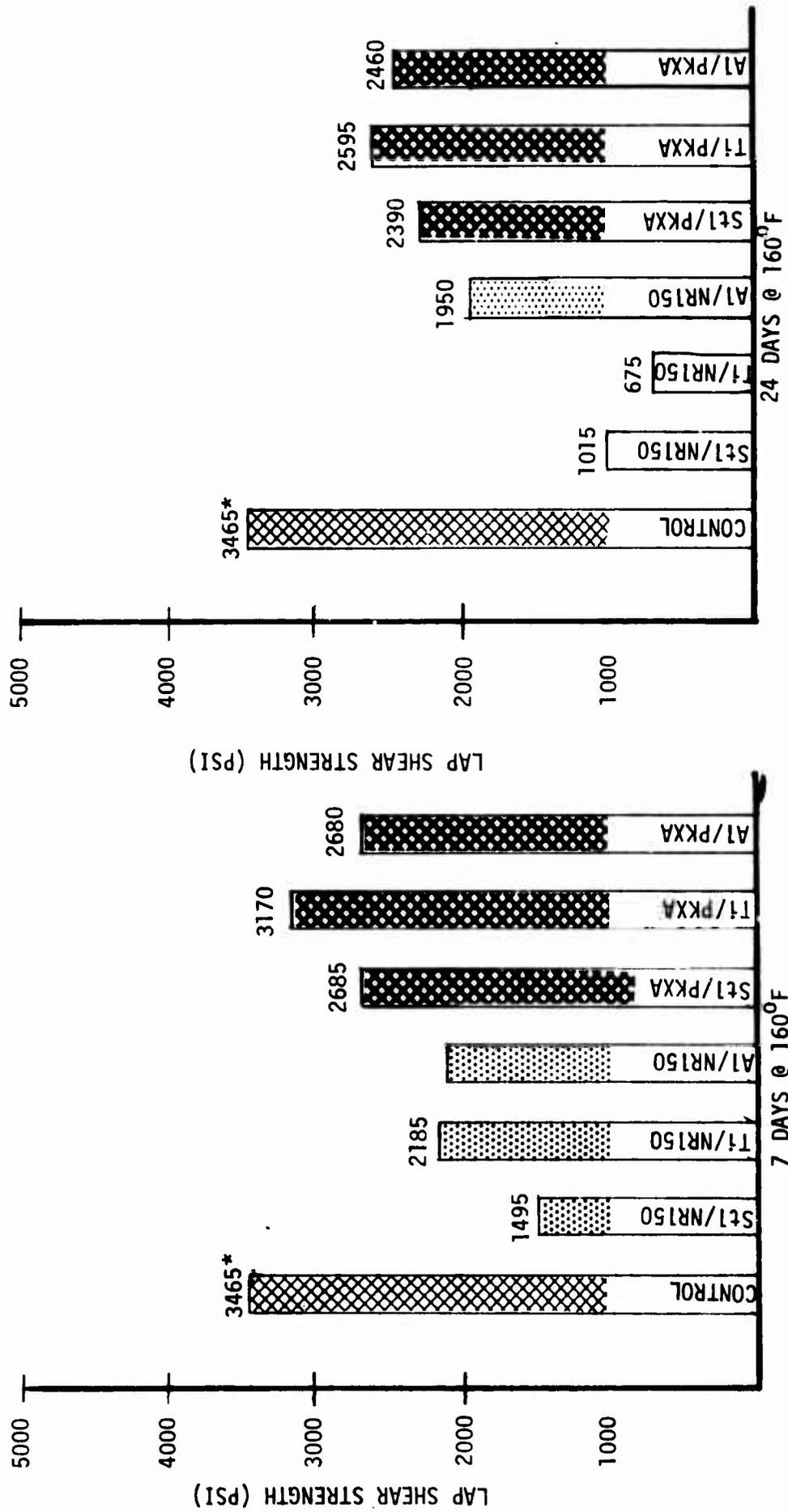


Figure 39: THE EFFECT OF HOT JP-4 ON PKXA AND NR150A2 LAP SHEAR SPECIMENS

TABLE XI - THE EFFECT OF JP-4 ON PKXA BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	NC	-
	24	RT	NC	-
	10	160	0.375	-
	24	160	1.00	-
Aluminum	10	RT	0.50	0.25 to 0.75
	24	RT	0.26	0.25 to 0.30
	10	160	0.71	0.50 to 1.25
	24	160	1.50	1.00 to 1.75
Titanium	10	RT	0.25	-
	24	RT	1.12	0.50 to 1.75
	10	160	0.50	-
	24	160	0.75	0.50 to 1.00

NC = No Change

- = No Range

TABLE XII - THE EFFECT OF MIL-L-7808 ON NR150A2 BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	1.24	0.78 to 1.60
	24	RT	1.46	0.84 to 1.75
	10	160	2.13	2.10 to 2.15
	24	160	2.47	1.98 to 2.86
Aluminum	10	RT	2.79	2.63 to 2.98
	24	RT	2.83	2.69 to 3.04
	10	160	3.46	3.41 to 3.63
	24	160	Delamination	-
Titanium	10	RT	1.83	1.69 to 1.98
	24	RT	0.49	0.43 to 0.64
	10	160	0.53	0.46 to 0.69
	24	160	1.92	1.83 to 2.00

- = No Range

TABLE XIII - THE EFFECT OF MIL-L-7808 ON PKXA BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	NC	-
	24	RT	NC	-
	10	160	0.43	0.34 to 0.52
	24	160	0.68	0.61 to 0.78
Aluminum	10	RT	NC	-
	24	RT	0.23	0.19 to 0.27
	10	160	0.51	0.47 to 0.54
	24	160	0.73	0.63 to 0.90
Titanium	10	RT	0.13	0.08 to 0.21
	24	RT	0.42	0.08 to 0.84
	10	160	1.23	1.11 to 1.44
	24	160	0.76	0.63 to 0.94

NC = No Change

- = No Range

TABLE XIV - THE EFFECT OF MIL-H-5606 ON NR150A2 BONDED
DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	0.78	0.64 to 0.93
	24	RT	0.82	0.67 to 0.97
	10	160	1.04	0.98 to 1.15
	24	160	1.16	1.09 to 1.23
Aluminum	10	RT	0.62	0.53 to 0.78
	24	RT	0.76	0.64 to 0.93
	10	160	0.93	0.89 to 0.99
	24	160	0.87	0.68 to 1.04
Titanium	10	RT	1.98	1.91 to 2.14
	24	RT	0.76	0.74 to 0.84
	10	160	1.30	1.25 to 1.37
	24	160	2.64	2.59 to 2.73

TABLE XV - THE EFFECT OF MIL-H-5606 ON PKXA BONDED DOUBLE CANTILEVER BEAM SPECIMENS - STRESSED

Adherend	Days Aging	Test Temp. (°F)	Average Crack Growth (inches)	Crack Range (inches)
Steel	10	RT	.11	.10 to .14
	24	RT	NC	-
	10	160	.34	.28 to .39
	24	160	.43	.36 to .72
Aluminum	10	RT	NC	-
	24	RT	.14	.11 to .19
	10	160	.22	.16 to .31
	24	160	NC	-
Titanium	10	RT	.34	.31 to .37
	24	RT	.28	.23 to .34
	10	160	.47	.41 to .55
	24	160	.43	.38 to .51

NC = No Change

- = No Range

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Polyphenylene Sulfides	Bonding	DCB Specimens
Polyimides	Crack Extension	
Adhesives	Shear Strength	
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
<p>Six different classes of thermoplastic polymers having high temperature capabilities (+300°F service) were surveyed after studying available data; five polymers from three different classes were screened for adhesive shear properties. The screening was conducted on two polysulfones, PKXA and P-1700; one polyethersulfone, 100P; and two polyimides, NR150A2 and 2080D. Screening test results indicated that all five polymers were good potential adhesives. Two polymers, PKXA and NR150A2, were selected for in-depth studies in surface</p>		

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

preparation and environmental resistance.)

Three different surface preparation techniques were evaluated for each metal substrate (aluminum, titanium and stainless steel). Upon completion of the surface preparation study, phosphoric acid anodize, chromic acid anodize, and sulfuric anodize were selected for surface treating aluminum, titanium and steel in the environmental exposure study, respectively. Limited modifications of PKXA polymer were made to improve the adhesive quality of the material. Test results indicated that these limited modifications offered no structural advantages. The addition of glass scrim to the film adhesive was an improvement over the neat resin film.

The environmental resistance of PKXA and NR150A2 adhesively bonded DCB and lap shear specimens were evaluated in thermal, salt water, humidity and fluid environments. Test results indicated that the PKXA DCB specimens were resistant to significant crack growth in all environments. The shear strength of PKXA specimens was not significantly reduced in thermal or room temperature JP-4 environments. NR150A2 DCB specimens were not significantly affected by humidity and MIL-H-5606 environments, but the lap shear specimens were significantly affected in thermal, JP-4 (RT and 160°F) and MIL-L-7808 environments.