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MAR 77 J T HOGGATT, S G HILL N00019-76-C-0171
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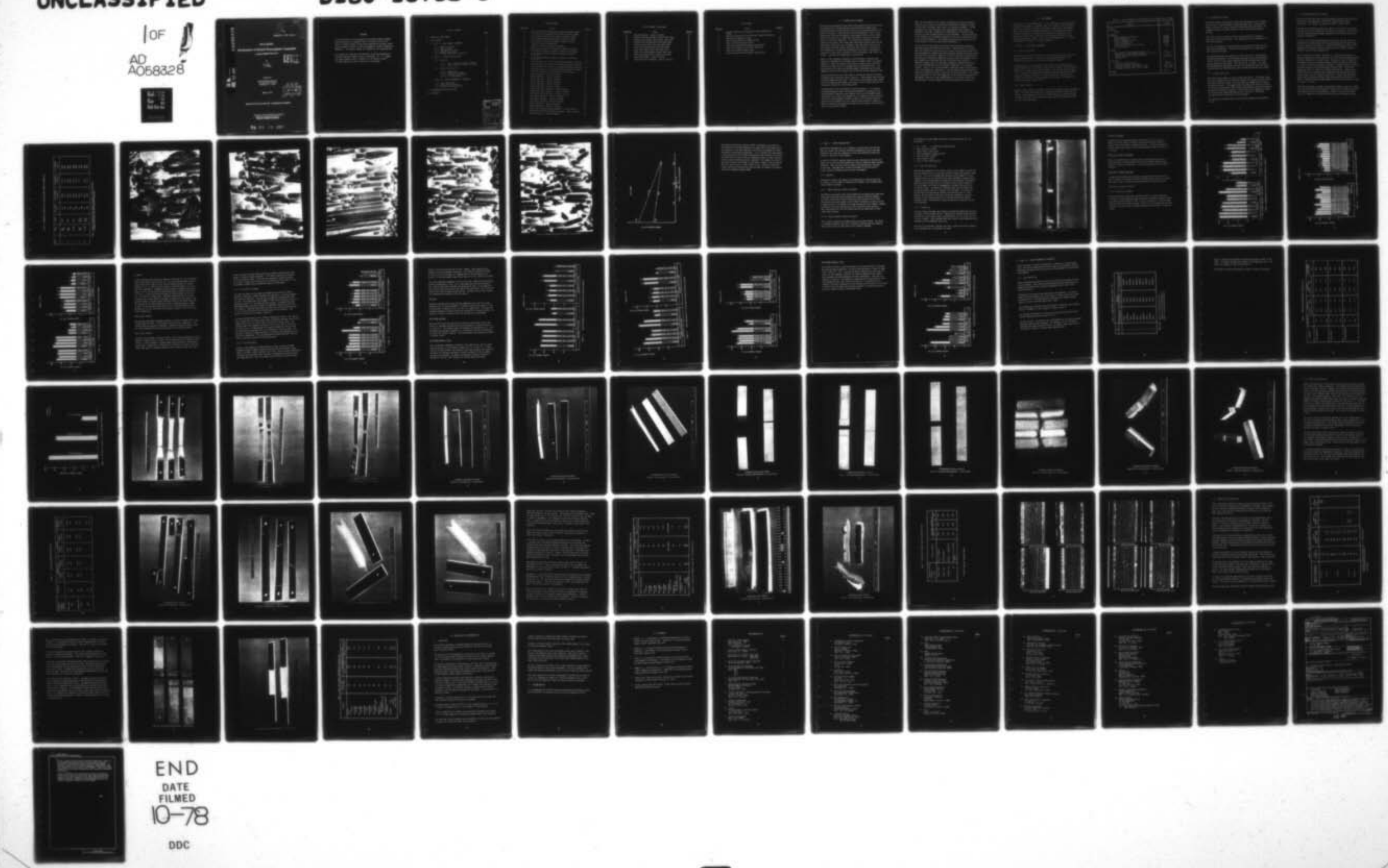

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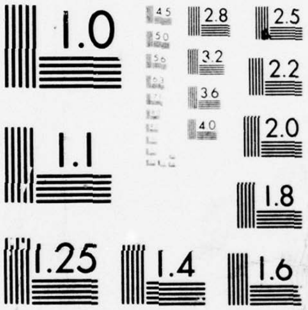

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Report No. D180 - 18752 - 3

FINAL REPORT

Development of Hybrid Thermoplastic Composites

Contract N00019-76-C-0171

By

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Prepared For

Naval Air Systems Command
U.S. Department of the Navy
Washington D. C. 20360

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APPROVED FOR PUBLIC RELEASE: DISTRIBUTION UNLIMITED

RESEARCH AND ENGINEERING DIVISION
BOEING AEROSPACE COMPANY
SEATTLE, WASHINGTON 98124

FOREWORD

This report covers the work conducted by the Boeing Aerospace Company during the period of 19 December 1975 to 28 February 1977 for the Naval Air Systems Command, United States Department of the Navy, under Contract N00019-76-C-0171, entitled "Development of Hybrid Thermoplastic Composites." Mr. Maxwell Stander (Air 52032D) was the Program Monitor.

This program was conducted by the Materials Technology Department of the Boeing Aerospace Company, Seattle, Washington. Mr. J. T. Hoggatt was Program Manager and Mr. Sylvester Hill was Technical Leader. Mr. Ralph Hodges was a major contributor to the program.

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1.0 INTRODUCTION AND SUMMARY

With more and more emphasis being placed upon manufacturing military aircraft and hardware with increased payloads and performance at reduced costs, advanced composites have become more attractive as replacement materials for metallic structures. Under previous contracts (References 1 and 2) it was demonstrated with glass and graphite reinforcements that the use of thermoplastic resin matrix was a viable and cost-effective process for the manufacture of structural components. The thermoplastic composites are less expensive than epoxies, have longer open time permitting more layup time and manufacturing operations, less scrappage, lower fabrication costs, can be post-formed, and permit shorter processing times (no cure). The intent of this program was to increase the technological data base for thermoplastic usage and to assess their resistance to fluid environments under stress conditions. This work was accomplished in three phases.

Phase I of this program was devoted to evaluating ways of improving the adhesion of thermoplastic polysulfone resins to graphite fibers in advanced composites. Under this phase of the program, an epoxy surface finish was placed on the fibers prior to prepreging with the polysulfone resin. The epoxy surface finish which was applied showed no advantage over the epoxy compatible surface finish normally used by Hercules on their graphite fibers.

Three polysulfone resins (P-1700, PKXA.41, and PKXA.24) from Union Carbide Corporation were evaluated as matrix materials. These polymers were evaluated at ambient temperature and 300°F. The flexural and interlaminar shear properties of the PKXA polymers appear to be similar to the P-1700 properties with increased resistance to chlorinated solvents.

The objectives of the second phase of this program were: (1) to examine polysulfone/graphite, metal-clad polysulfone/graphite, and epoxy-clad polysulfone/graphite composites under combined stress and environmental aging conditions; (2) to assess the compatibility of cladding with polysulfone composites with epoxy/graphite and metals without sacrificing the structural properties of the composites; (3) to evaluate epoxy and metal cladding as a means of protecting the polysulfone composites from solvents and fluids which attack the polysulfone polymer.

Under the second phase of this program, thermoplastic polysulfone graphite composites with various clad surfaces were evaluated for mechanical properties and their resistance, under stress, to selected environments. P-1700, aluminum-clad P-1700, and epoxy-clad P-1700 composites were exposed to weathering, salt water, humidity, MIL-H-5606 hydraulic fluid, MIL-L-7808 lubricant, and MIL-H-83306 hydraulic fluid environments. With the exception of MIL-H-83306, the environmental exposures did not have a significant effect on the stressed composites. The mechanical properties were very similar to those reported in an earlier Navy program (Reference 1).

The last phase of the program was devoted to hybridizing the polysulfone composites by mixing the resins and carriers. The objective of this phase, which was divided into three tasks, was to assess the structural potential of hybrid thermoplastic composites and the processing problems that may be associated with their processing. Under the first task, mechanical properties were determined on P-1700 hybrid composites of glass, Kevlar and graphite fabrics. Each composite was fabricated from two of the three fabric materials. Mechanical property tests on these hybrid composites showed no deficiencies that would preclude their use on structural components in the aerospace industry. Tasks II and III were devoted to fabricating metal-clad and epoxy-clad P-1700 composites, respectively. The clad laminates were evaluated for mechanical properties and their resistance to fluid environments. The cladding offers protection for the P-1700 composites on the surfaces as expected, but detrimental fluids such as MIL-H-83306 hydraulic fluid readily attack the exposed edges of the clad composites even when sealed with an epoxy resin.

2.0 STUDY PROGRAM

The objectives of this program were: (1) to study ways of improving adhesion of polysulfone polymers to graphite fibers, (2) to evaluate the structural integrity of graphite reinforced thermoplastics composites in environments under stress, and (3) to evaluate the feasibility of protecting graphite reinforced thermoplastic composites from exposure by hybridizing. As a method of achieving these objectives, the program was divided into three major study programs: Phase I - Fiber Surface Treatment; Phase II - Stress Cracking Study; and Phase III - Hybrid Composites.

2.1 PHASE I - FIBER SURFACE TREATMENTS

2.1.1 Material Selection

The fibers selected for this portion of the program were Hercules' HM-S and Type A-S graphite fibers. These fibers were selected because of their availability and wide use in other Navy development programs.

The thermoplastic resin selected for this phase of the program was Union Carbide Corporation's polysulfone (P-1700). The P-1700 was selected primarily because of its extensive data base developed in earlier Navy contracts (Ref. 1, 2, 4). The resin has demonstrated excellent properties with various fibers in earlier programs and there is a large data base with standard fiber finishes for comparison of data developed in this phase. The properties of this polysulfone are shown in Table 1.

2.1.2 Fiber Finishes

An epoxy surface finish of Fiberite's 934 resin was placed on Hercules Type A-S fiber. The fibers were dipped into a one-percent solution of 934 resin and acetone. After allowing the fibers to become completely saturated with the solution, the fibers were dried and the epoxy cured for 90 minutes at 350°F in an air circulating oven.

Table 1: Typical Properties of Polysulfone (P-1700) Thermoplastic Polymer

Property	P-1700
General	
Density	1.24
Mechanical	
Tensile Strength at Yield, psi	10,200
Tensile Modulus, psi	360,000
Tensile Elongation at Break, %	50-100
Flexural Strength, psi	15,400
Flexural Modulus, psi	390,000
Izod Impact at 72°F, ft-lb/in. Notch	1.3
Rockwell Hardness	R120
Thermal	
Heat Distortion Temperature at 264 psi, °F	345
Coefficient of Linear Thermal Expansion, in./in./°F	3.1×10^5
Thermal Conductivity, Btu/hr/ft^2/°F/in.	1.8
Flammability	Non-Burning
Electrical	
Dielectric Strength, V/mil	425
Volume Resistivity, 72°F, ohm-cm	5×10^{16}
Dielectric Constant, 72°F, 60 Hz - 1 MHz	3.07 - 3.03
Dissipation Factor, 72°F, 60 Hz - 1 MHz	.008-.0034
Source	Ref. 5

2.1.3 Impregnation of Fibers

The graphite fibers with the epoxy surface were impregnated with the thermoplastic resins in the laboratory on a batch-to-batch basis. Unidirectional sheets of prepreg, 34 inches long and 26 inches wide, were fabricated. The impregnating resins were Union Carbide's P-1700 and two PKXA polysulfone polymers.

The P-1700 resin was received in pellet form and dissolved in methylene chloride to make a stock solution. The impregnating solution contained 15 to 20% solids by weight.

The PKXA, an endcapped reactive polysulfone, was received in flake form from Union Carbide Corporation. The solution was prepared in the same manner as the P-1700 solution above.

The impregnated fibers were placed into a hood until most of the methylene chloride had evaporated. The dry prepreg was placed into an oven maintained at 180 - 200°F until the residual methylene chloride had evaporated and the prepreg reached constant weight. This required 16 - 24 hours. The fibers were weighed before impregnation and after the impregnated fibers had reached constant weight and these data were used to achieve the desired resin content.

2.1.4 Laminate Fabrication

The laminates were molded in a press at 650°F and 200 psi. The prepreg sheets were cut to appropriate sizes and placed between 5-mil Kapton film and titanium caul sheets (.040 inch thick). The caul sheets were bagged and full vacuum was drawn on the part. The bagged assembly was placed into the press at 650°F for 30 minutes under contact press pressure (approximately 300 lbs ram pressure) and 200 psi was applied to the part. The part was held at temperature (650°F) for 20 minutes and cooled under pressure at a rate of 10 - 20°F/minute. Cool-down rate varies with part size and thickness.

The composite was removed from the press when the temperature reached 200°F or less.

2.1.5 Specimen Fabrication and Testing

Flexure and interlaminar shear (short beam method) specimens were fabricated and tested at room temperature and 300°F per ASTM D-790 and D-2344, respectively. Test results are reported in Table 2, Items 4 and 5.

Scanning electron photomicrographs were taken of a failed flexural specimen from each of the resin systems at a 45° angle to the fracture. Figures 1 and 2 are the scanning electron photomicrographs of the failed PKXA and P-1700 specimens, respectively.

A study of the P-1700 microphotographs discloses that the epoxy surface on the Hercules A-S fibers appears to offer no advantage over the normal fiber finish for adhesion (see Figure 5). Properties of the epoxy surface finish specimens were slightly less than properties of the P-1700/A-S specimens reported in previous work (Reference 2).

Under a previous Navy contract (Reference 1), P-1700 polymer appeared to have poor adhesion to an HM-S type fiber, and it was felt that better adhesion could be achieved with a surface finish. The search for ways of improving the adhesion of resins to the HM-S graphite fibers continued with the idea of reactive surfaces on end groups of polymers. Two PKXA resins with reactive end groups were used for making prepregs with HM-S fibers. HM-S/P-1700 prepreg was used as a control for property determination. Laminates were molded from each of the three resin systems using the same molding process that was used in earlier composite fabrication. Flexural and interlaminar shear (short beam method per ASTM D-2344) specimens were fabricated and tested. Scanning electron photomicrographs of the failed specimens are shown in Figures 3, 4, and 5.

The PKXA resins appear to adhere to the graphite fibers as well as, or better than, the P-1700 resin but the mechanical properties are slightly less than the properties of the P-1700 composites. Figure 6 is a plot of Flexural Stress vs. Temperature for PKXA and P-1700 resins on HM-S graphite fibers.

Table 2: MECHANICAL PROPERTIES OF UNIDIRECTIONAL GRAPHITE COMPOSITES

ITEM	RESIN	FIBER	TEST TEMP. (^oF)	FLEX STRENGTH (10^3 psi)	FLEX MODULUS (10^6 psi)	ILS STRENGTH (psi)
1	PKXA .24	HM-S	RT	89.0	14.4	6735
			300	71.9	12.4	4345
2	PKXA .41	HM-S	RT	103.4	20.5	5645
			300	76.3	20.0	3610
3	P-1700	HM-S	RT	167.5	17.5	7630
			300	80.0	15.9	5420
4	PKXA .24	A-S/ Epoxy	RT	116.5	7.9	7335
			300	5.5	--	865
5	P-1700	A-S/ Epoxy	RT	101.0	10.5	8565
			300	64.5	11.1	5740

NOTE: PKXA.24 and PKXA.41 were numbers assigned to the proprietary N'capped polysulfone by Union Carbide Corporation.

Figure 1: PKXA .24/A-S-Epoxy Unidirectional Graphite Composite (600X)

Figure 2: P-1700/A-S-Epoxy Unidirectional Graphite Composite (600X)

Figure 3: PKXA .24/HM-S Unidirectional Graphite Composite (600X)

Figure 4: PKXA .41/HM-S Unidirectional Graphite Composite (600X)

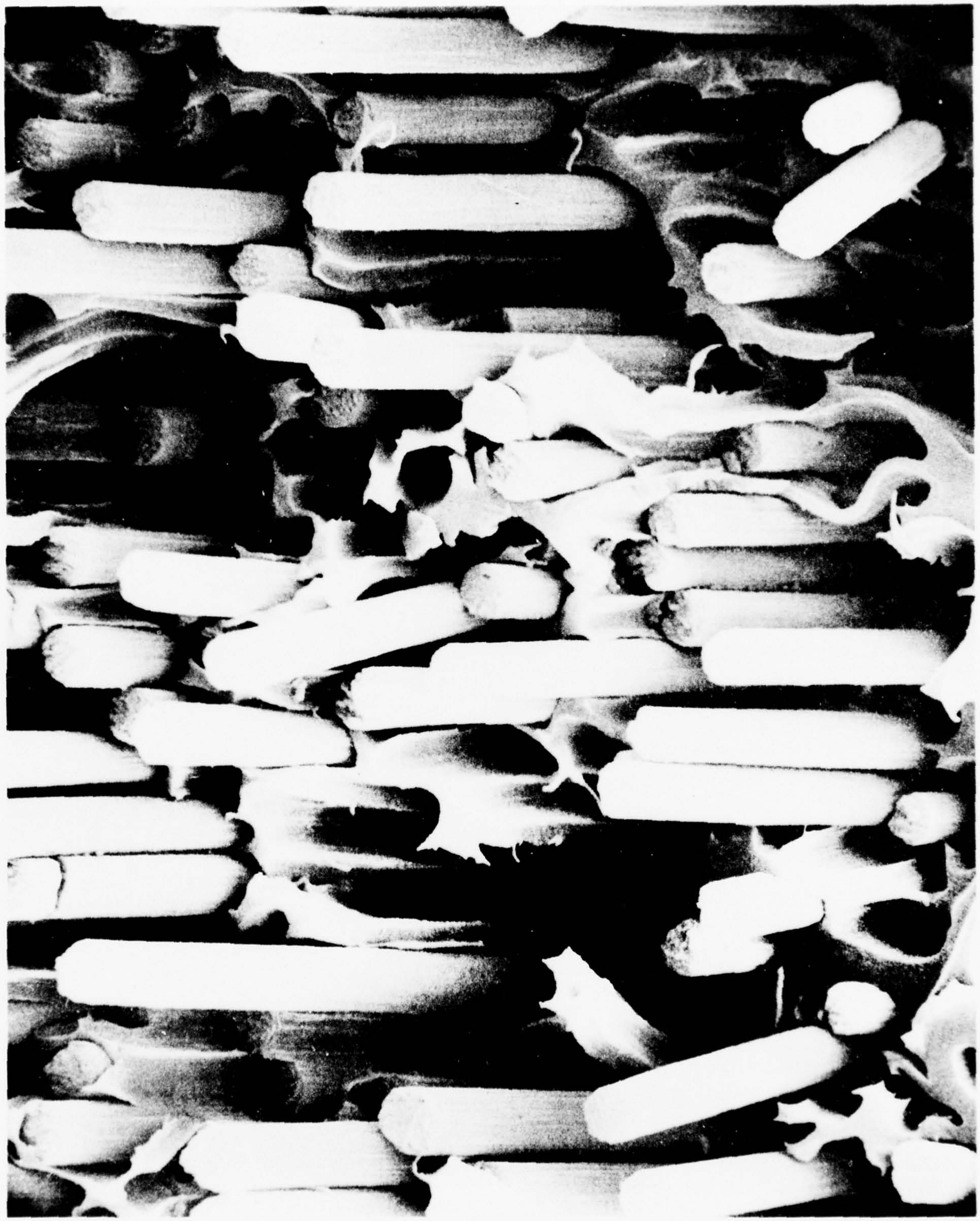

Figure 5: P-1700/HM-S Unidirectional Graphite Composite (600X)

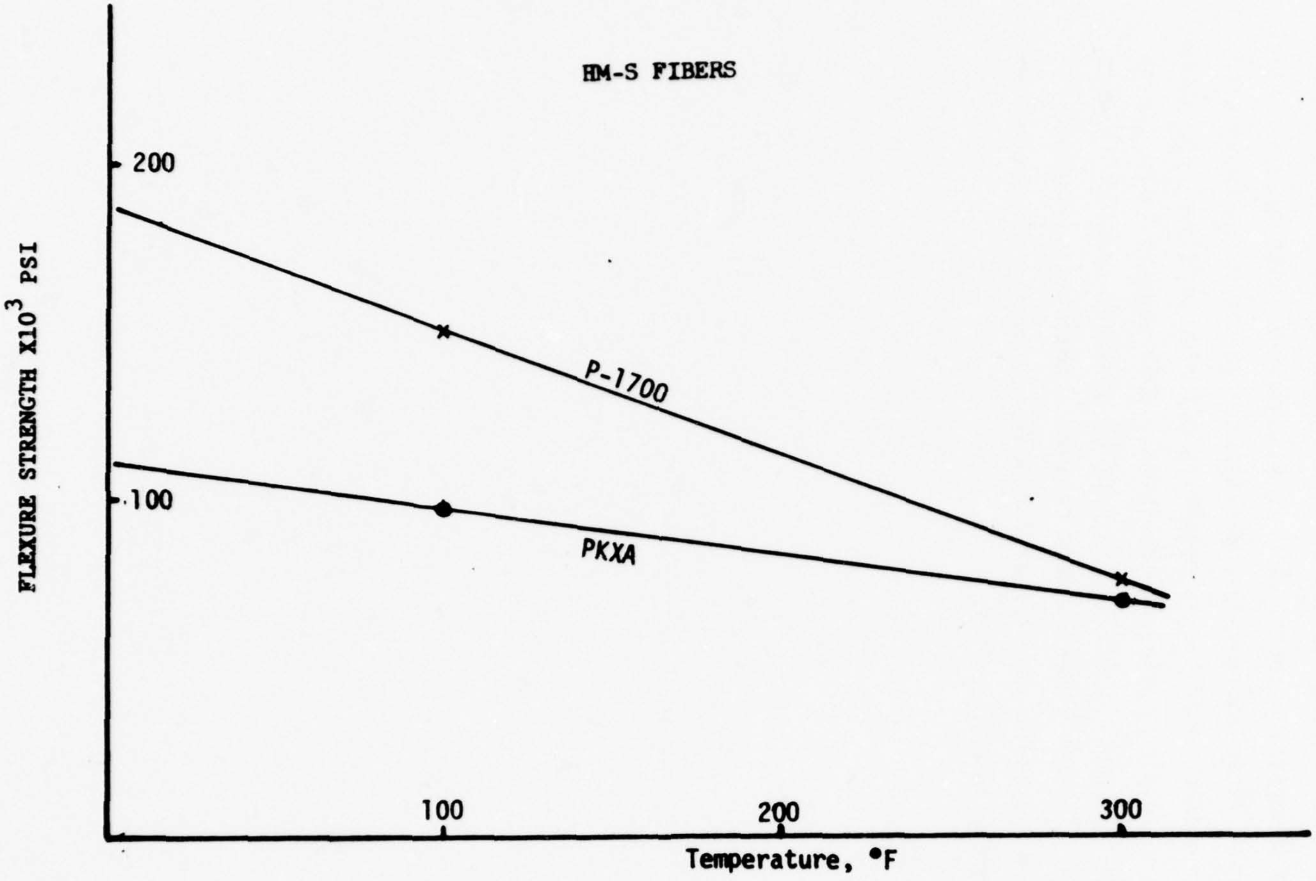

FIGURE 6 FLEXURAL STRESS VS. TEMPERATURE

Processing of PKXA resins warrants further investigation and evaluation. The properties of the PKXA composites are lower than P-1700 properties but the properties of the PKXA composites can possibly be upgraded with added impregnation and processing experience. It is a polymer with good potential. It was noted during resin content determinations that laminates fabricated from PKXA resins did not dissolve in methylene chloride or other laboratory solvents after the molding cycle was completed. This appears to be due to some crosslinking of the polymer or some type of chemical reaction between the resin and the fiber or fiber surface finish. Laminates showed only slight cracking and discoloration after two weeks of exposure in methylene chloride and Dimethylformamide (DMF).

2.2 PHASE II - STRESS CRACKING STUDY

Unreinforced thermoplastics have a tendency to stress crack and craze and there was concern that graphite reinforced thermoplastics may do the same. Therefore, this study was undertaken to assess the problem and ensure the structural integrity of the laminates.

A series of P-1700/A-S graphite composites were fabricated to determine the effects of various fluid environments upon flexural properties under stressed conditions. Non-clad, metal-clad and epoxy-clad specimens were evaluated in the study. Specimens were stressed and exposed to aircraft service fluids, moisture, weathering and salt water environments.

2.2.1 Materials

The materials used in this phase of the program were P-1700/A-S (62% fiber by volume), .011-inch 2024-T3 aluminum-clad P-1700/A-S, and T-300/934 epoxy-clad P-1700/A-S laminates.

2.2.1.1 Metal Cladding P-1700/A-S Laminates

The 2024-T3 clad-aluminum skins were phosphoric acid anodized and primed with BR127 (BMS 5-89) epoxy primer manufactured by American Cyanimid Company. P-1700/A-S laminate was solvent cleaned with ethyl alcohol and air dried. The aluminum skins were bonded to the P-1700/A-S laminate by placing a 10-mil P-1700/112 E-glass film between the primed aluminum and the P-1700/A-S laminate and heating in a press under vacuum to 525°F with 50 psi part pressure. Parts were heated until the bond reached equilibrium temperature and then cooled under pressure.

2.2.1.2 Epoxy Cladding P-1700/A-S Laminates

The P-1700/A-S laminate was abraded lightly and solvent cleaned. Four plies of T-300/934 (Fiberite's 934 resin on Thornel T-300 unidirectional graphite fibers) were laid up on each side of the P-1700/A-S laminates.

The laminate was vacuum bagged and cured in an autoclave using the cure cycle below:

a. R.T. - 250°F 1 - 5°F/minute, vacuum pressure
b. Hold 15 minutes at 250°F
c. Apply 100 psi, vent vacuum
d. Hold 45 additional minutes at 250°F
e. Heat from 250°F to 350°F
f. Hold 2 hours at 350°F
g. Cool to +140°F under pressure.

2.2.2 Stress Cracking Tests

Specimens were mounted in test fixtures (Figure 7) and loaded to the desired stress concentration prior to placing into the different environmental aging solutions. The initial specimens were placed into environments at stress concentrations of 10, 25, and 50% of the control specimen's average flexure strength. After testing several specimens from the different environmental aging solutions which had little or no effect on the flexural properties of the specimens, the flexural stresses were increased to 60 and 75% of the control values. The percent of ultimate stress was calculated using the average control data for a laminate as the ultimate. To achieve the desired stress the specimen was loaded to the desired stress in a test machine, then the bolts on the test fixture were tightened to clamp the specimen in the stressed condition until the load on the test machine returned to zero. At that point we were assured that the desired load was on the specimen.

2.2.2.1 Weathering

Stressed flexural specimens were exposed to accelerated weathering conditions according to ASTM procedure E42-65. Specimens were exposed to 600 20-minute sunlight/rain cycles under stress concentrations of 10, 25, 50, 60, and 75% of their ultimate. These specimens were then tested at room temperature after completing the environmental aging cycle.

The epoxy and polysulfone laminates lost their surface gloss during exposure but no permanent set or deformation was noted.

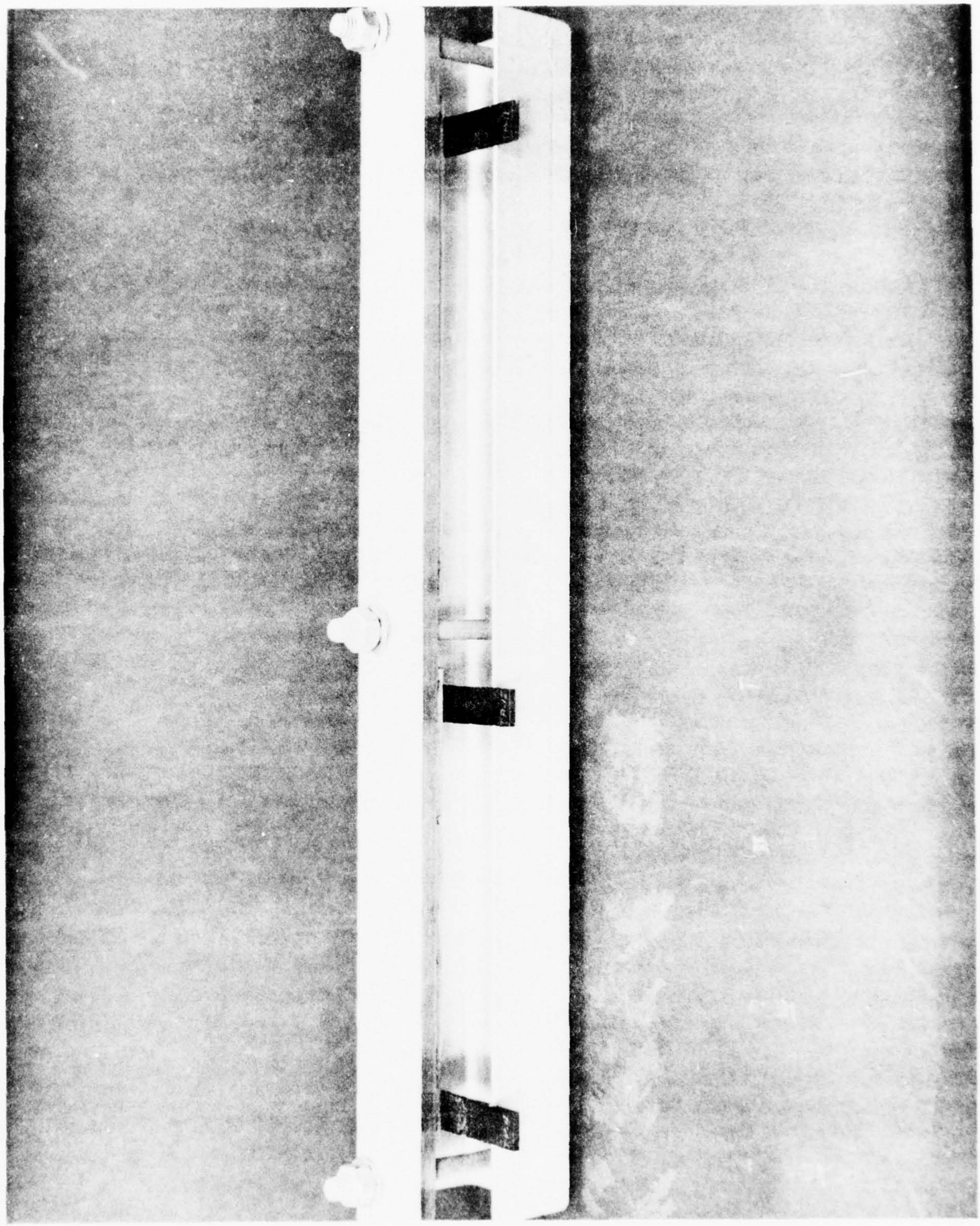

Figure 7: Fixture For Stressing Flexural Specimens

P-1700/A-S Specimens

Specimens exposed to weathering under stress loads of 10, 25, and 50% showed very little change in flexural stresses but definite increase in flexural moduli when tested at ambient conditions. A definite decrease in flexural stresses was noted for the exposed specimens loaded to 60 and 75% of ultimate.

Metal-Clad P-1700/A-S Specimens

Metal-clad specimens were stressed to 60% of ultimate during exposure. A slight decrease in flexural moduli when tested at ambient conditions was noted for the metal-clad specimens. This is the reverse of the P-1700/A-S specimens.

Epoxy-Clad P-1700/A-S Specimens

The epoxy-clad specimens were exposed to weathering under stress loads of 60% of ultimate. A decrease in flexural strength and an increase in flexural moduli were noted for these specimens when tested at ambient conditions.

These data are shown in Figure 8.

2.2.2.2 Moisture Environment

Moisture tests were conducted to determine effect on stressed composites as follows: Stressed specimens were placed into a condensing humidity chamber with 100% relative humidity at 120°F ± 5°F for a period of 7, 30 and 90 days. Specimens were also placed into water at 180°F ± 5°F and tested after two days of aging. The stress levels, exposure times and data are shown in Figures 9A and 9B.

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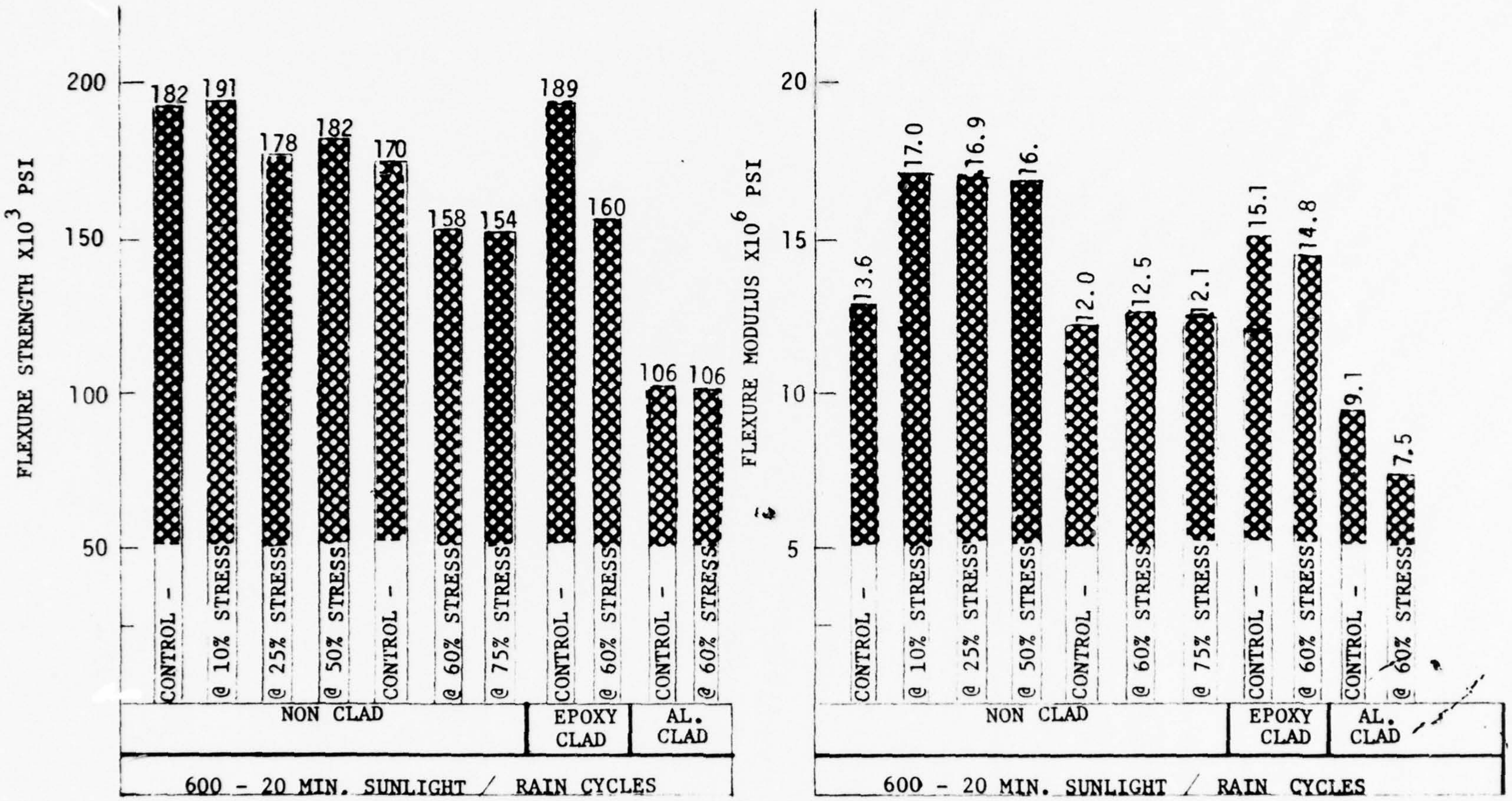

FIGURE 8 WEATHERING OF P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES

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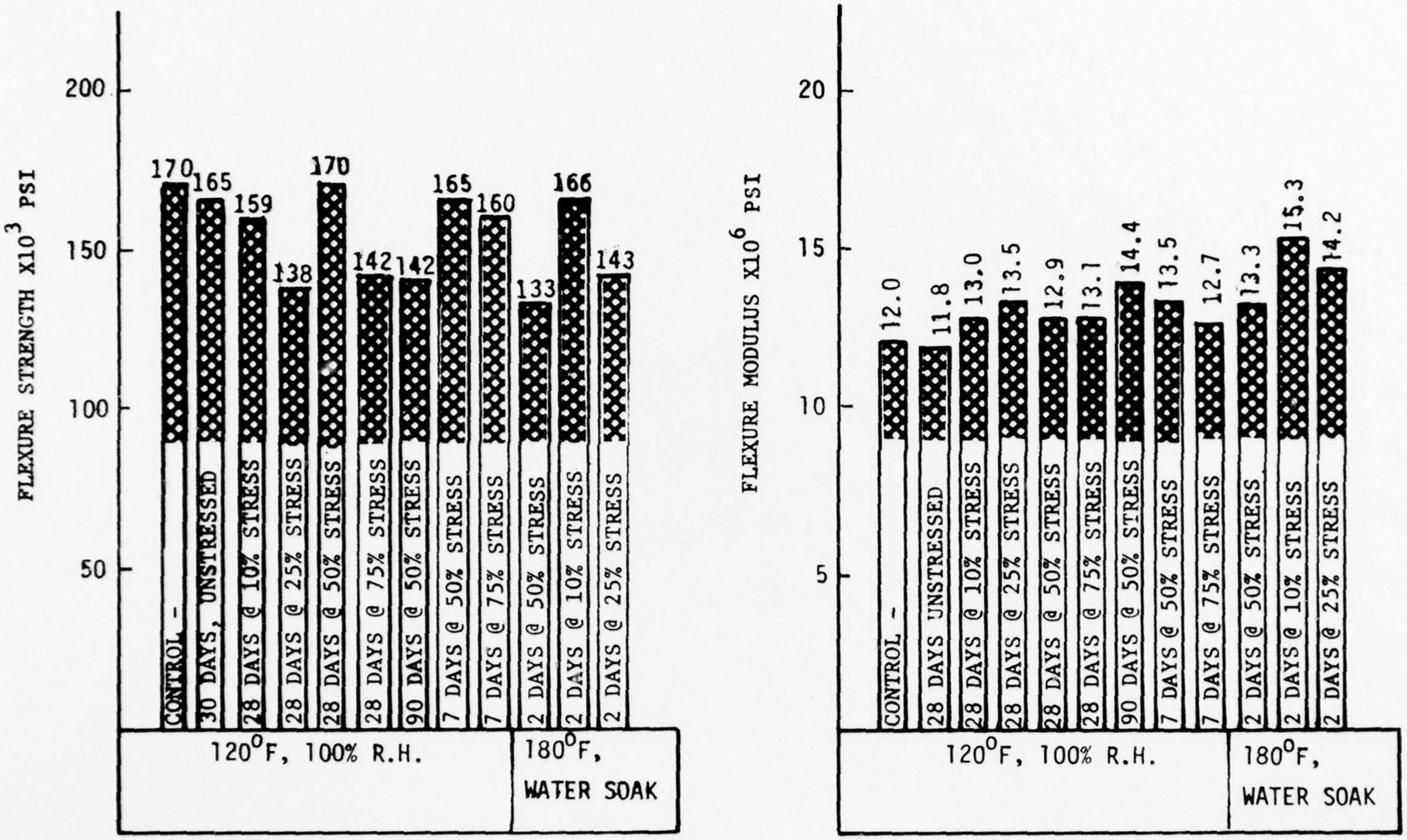

FIGURE 9A P-1700 NONCLAD GRAPHITE LAMINATES IN MOISTURE ENVIRONMENT

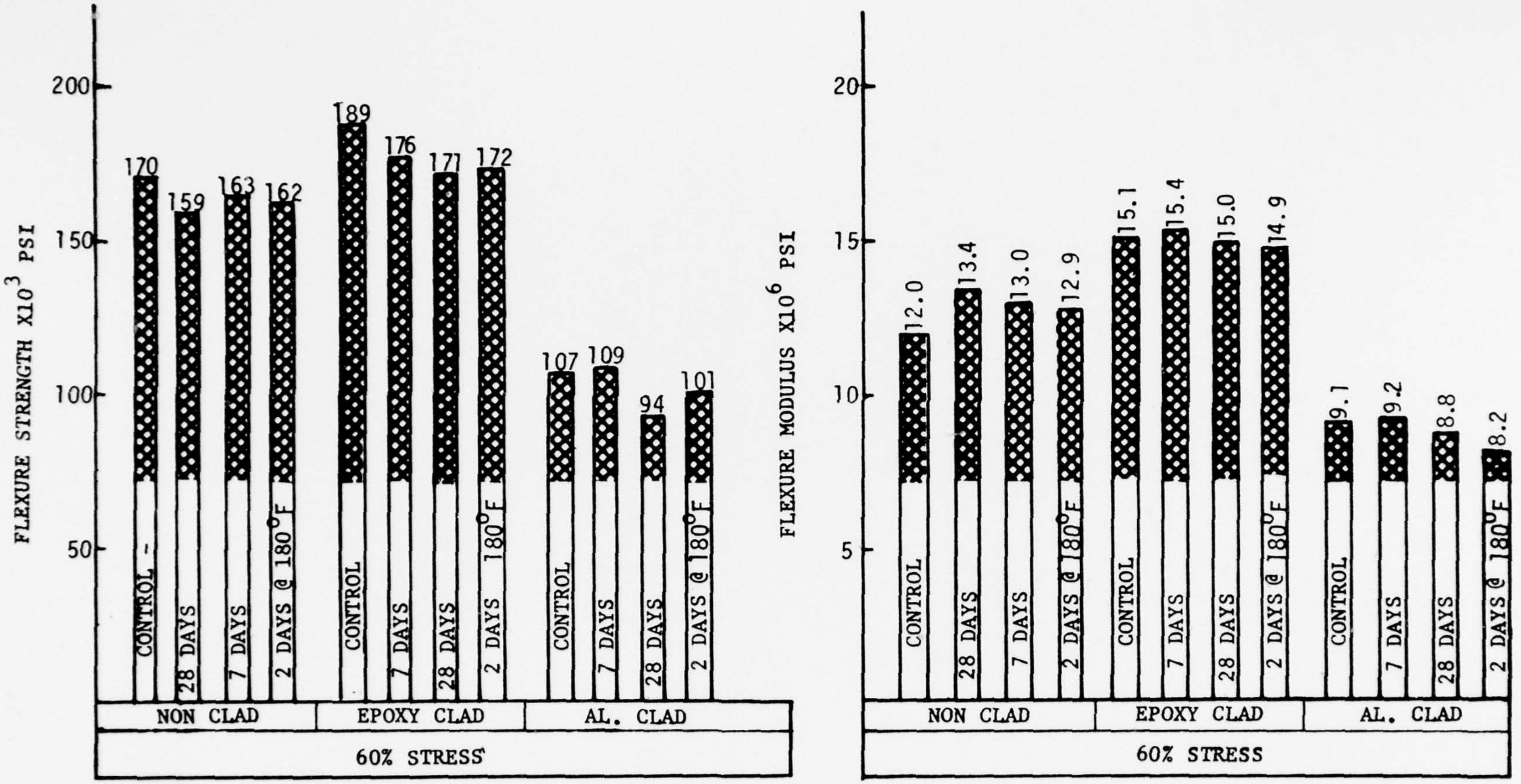

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FIGURE 9B P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES IN MOISTURE ENVIRONMENT

P-1700/A-S

Flexural specimens were stressed in humidity for periods of 7, 28, and 90 days under varying stress loads of 10 to 75%. Test data from specimens stressed 7 days under stress loads of 50, 60, and 75% showed decreases in flexural stresses of 2.9, 4.1 and 5.9% with increases in flexural moduli of 12.5, 8.3, and 5.8%, respectively. Specimens stressed 28 days in humidity with stress loads of 10, 25, 50, 60 and 75% displayed decreases in flexural stresses of 6.5, 18.8, 0, 6.5 and 16.5% with increases in flexural moduli of 8.3, 12.5, 7.5, 11.7, and 9.2%, respectively. Specimens stressed at 50% for 90 days displayed stress losses equal to specimens stressed at 75% for 28 days. The largest increase in flexural moduli (20%) was noted for the 90-day specimens. This was due to the long aging period at 120°F. Specimens soaked in water at 180°F for two days with stress loads of 10, 25 and 50% exhibited stress decreases of 2.3, 15.9 and 21.8% with increases in moduli of 27.5, 18.3, and 10.8%, respectively.

Epoxy-Clad P-1700/A-S

The epoxy-clad specimens displayed a decrease in flexural strength after 7 and 28 days under 60% stress. The decrease was 6.9 and 9.5%, respectively. The flexural moduli was basically unchanged. Specimens stressed in water at 180°F for 2 days displayed test results very similar to the 28-day specimens.

Metal-Clad P-1700/A-S

The metal-clad specimens stressed at 60% for 7 and 28 days exhibited very little change in flexural stress at 7 days (1.9% increase) but a 12.1% decrease after 28 days. A small decrease in flexural moduli was noted after 7 and 28 days of stress aging. Respectively, a decrease of 5.6 and 9.9% in flexural stress and modulus was noted for specimens stressed at 60% in water at 180°F for 2 days.

Flexural specimens stressed under moisture environment displayed very little change in flexural stresses and moduli. The flexural modulus was increased by the environmental aging. These data are reported in Figures 9A and 9B. The increase in moduli was possibly due to the additional heat (120°F) in the environmental chamber which increased the modulus of the polymer.

2.2.2.3 Salt Water Environment

To assess the effects of salt-water environment upon stressed P-1700/A-S composites properties, flexure specimens were exposed in a salt-spray chamber for a period of 200 hours. The salt-spray chamber was maintained per ASTM B-117-64. The chamber had an environment of 100% relative humidity, 100 ± 5°F temperature with a 5 ± .5% sodium chloride solution. The exposed specimens were weighed before and after exposure to determine the moisture pick-up of the laminates. Specimens were placed into the stress fixtures under stresses of 10, 25, 50, 60, and 75% of the ultimate stress of the laminate. The results of these tests are shown in Figure 10.

No visible deterioration of the P-1700/A-S laminate was noticed. Test results show a slight loss of flexural stress after the 200-hour exposure with the loss ranging from 2.4% at 10% stress level to 10.6% at 75% stress level. The flexural moduli data showed an increase of 6.0% at 75% level to 15% at the 10% stress level. A large decrease in flexural strength (31.7%) was noted for the epoxy-clad specimens with a 10.6% decrease in flexural moduli. The decrease in flexural strength and moduli of the metal-clad specimens was 9.4 and 20.9%, respectively. The large decreases in strength and moduli were the result of the salt corrosion on the aluminum skins.

2.2.2.4 Fluid Resistance

Because of the complexity of manufacturing aircraft, missiles and other aerospace hardware, composite materials must be able to withstand a large number of solvents, fuels, lubricants and fluids. Earlier studies (Reference 4) indicated that polysulfones were resistant or compatible with jet fuel JP-4, MIL-H-5606 hydraulic fluid, MIL-L-7808 aircraft lubricant, and MIL-H-83306

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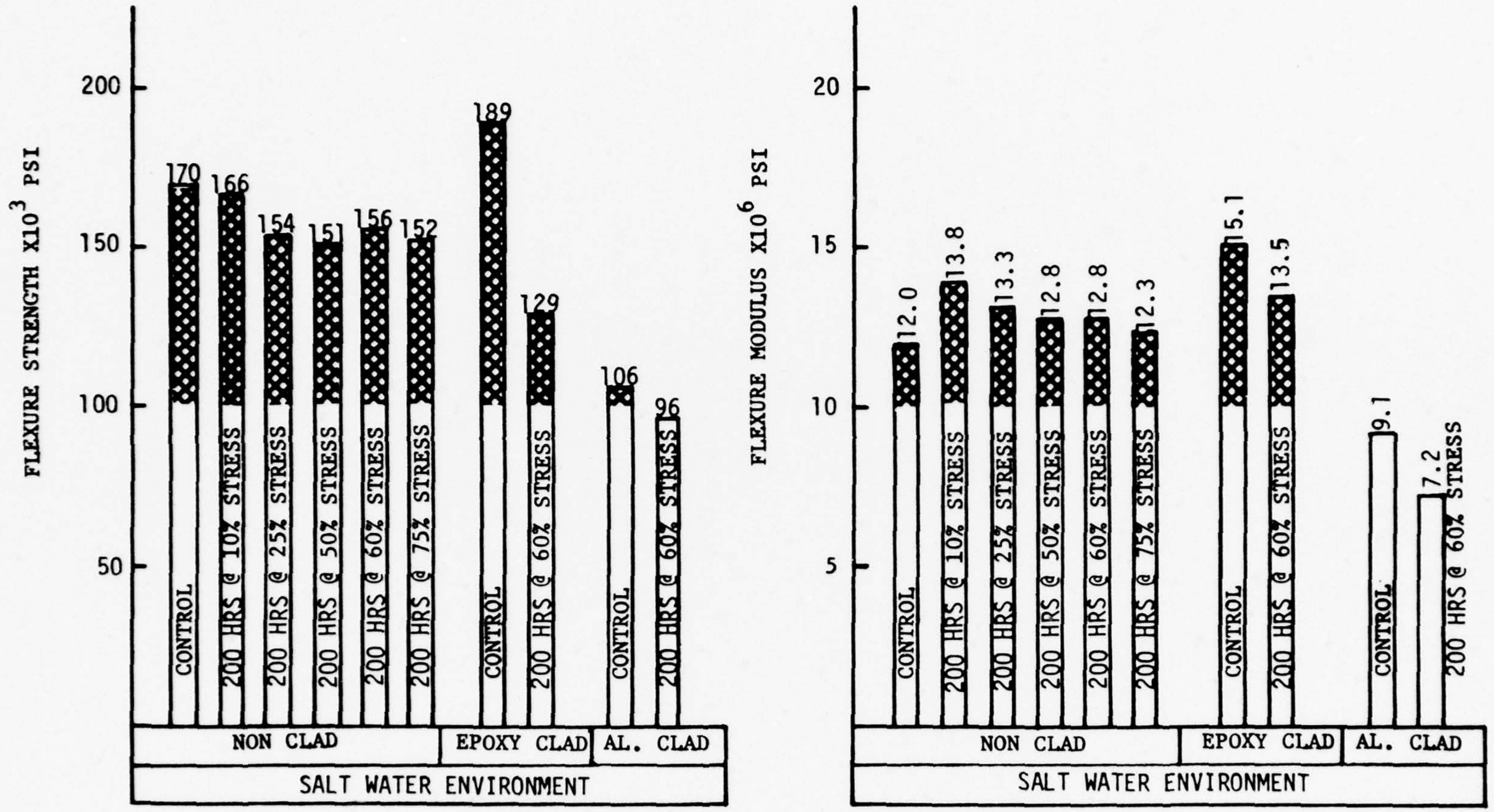

FIGURE 10 P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES IN SALT WATER ENVIRONMENT

hydraulic fluid for short periods of time. However, these composites were exposed to fluid environments unstressed. This study was therefore designed to assess the fluid resistance or incompatibility of the polysulfone composites in fluid environments under stress loads of 10, 25, and 50% of ultimate.

The initial specimens submerged in JP-4 jet fuel, MIL-L-7808 aircraft lubricant, and MIL-H-5606 hydraulic fluid all at room temperature and were stressed at 50%. After testing these specimens and analyzing the data, it was noted that the 50% stress data was basically the same as the control data. The 10% and 25% stress levels were replaced with 60% and 75%. The epoxy clad and the aluminum clad specimens were stressed to 60% of ultimate.

JP-4 Fuel

Specimens stressed to 50, 60 and 75% and submerged in JP-4 fuel for 28 days exhibited 5.9, 8.2 and 10% decreases in flexural stresses (see Figure 11) with flexural moduli increases of 9.2, 5.0, and 1.7%, respectively. The epoxy-clad specimens decreased in flexure stress but increased 2% in flexural modulus. An increase in flexural stress was noted for the aluminum clad specimens.

MIL-L-7808 Lubricant

While the 50% stress level had only a 5% effect on the flexural stress (see Figure 12), specimens stressed to 60 and 75% decreased in flexural stress by 21.2 and 24.1%. The flexural moduli of specimens were decreased by 2.5 - 6.7%. The epoxy-clad specimens displayed a 12.7% decrease in flexural strength with no significant change in modulus. The stressed metal-clad specimens increased in flexural stress.

MIL-H-5606 Hydraulic Fluid

Non-clad specimens submerged in hydraulic fluid under 50, 60, and 75% stress loads displayed a loss in flexural stresses of 5.3, 4.1 and 7.1%. A slight increase in flexural moduli was noted for these same specimens (see Figure 13). The epoxy-clad specimens decreased in flexural strength (12.2%) but modulus was basically unchanged. The metal-clad specimens increased in flexural strength (11.2%) but a small decrease in flexural modulus was noted.

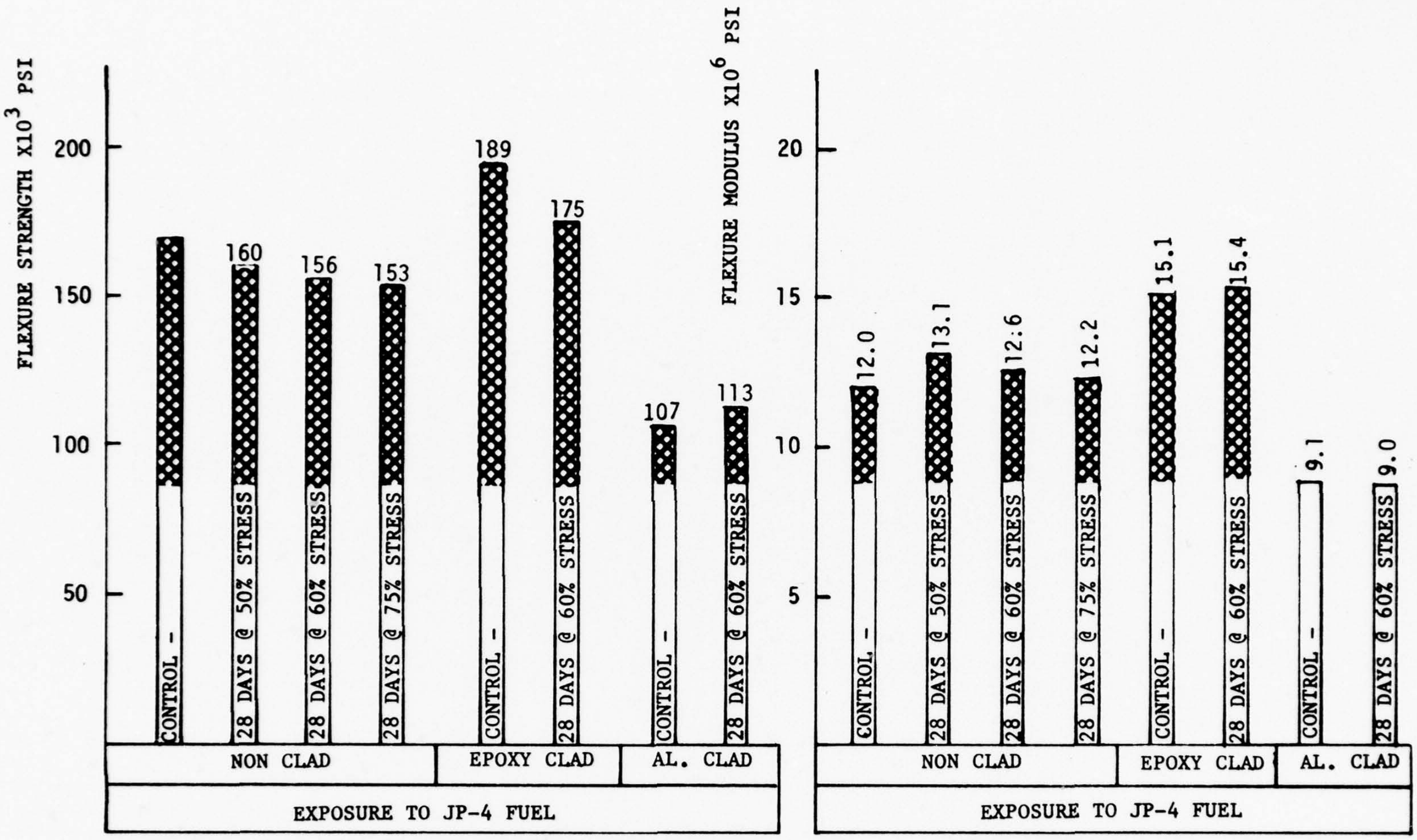

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FIGURE 11 FLUID RESISTANCE OF P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES

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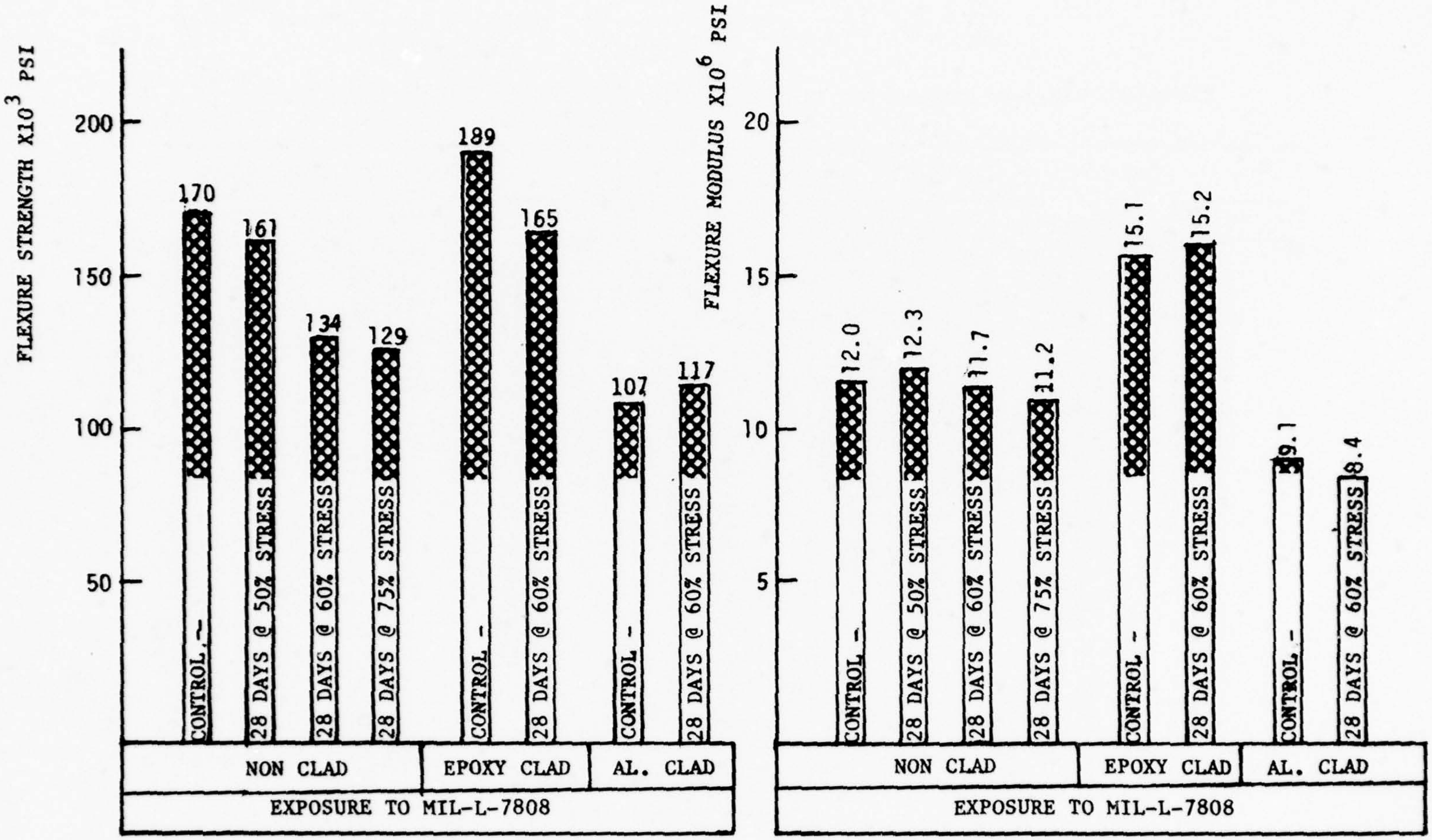

FIGURE 12 FLUID RESISTANCE OF P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES

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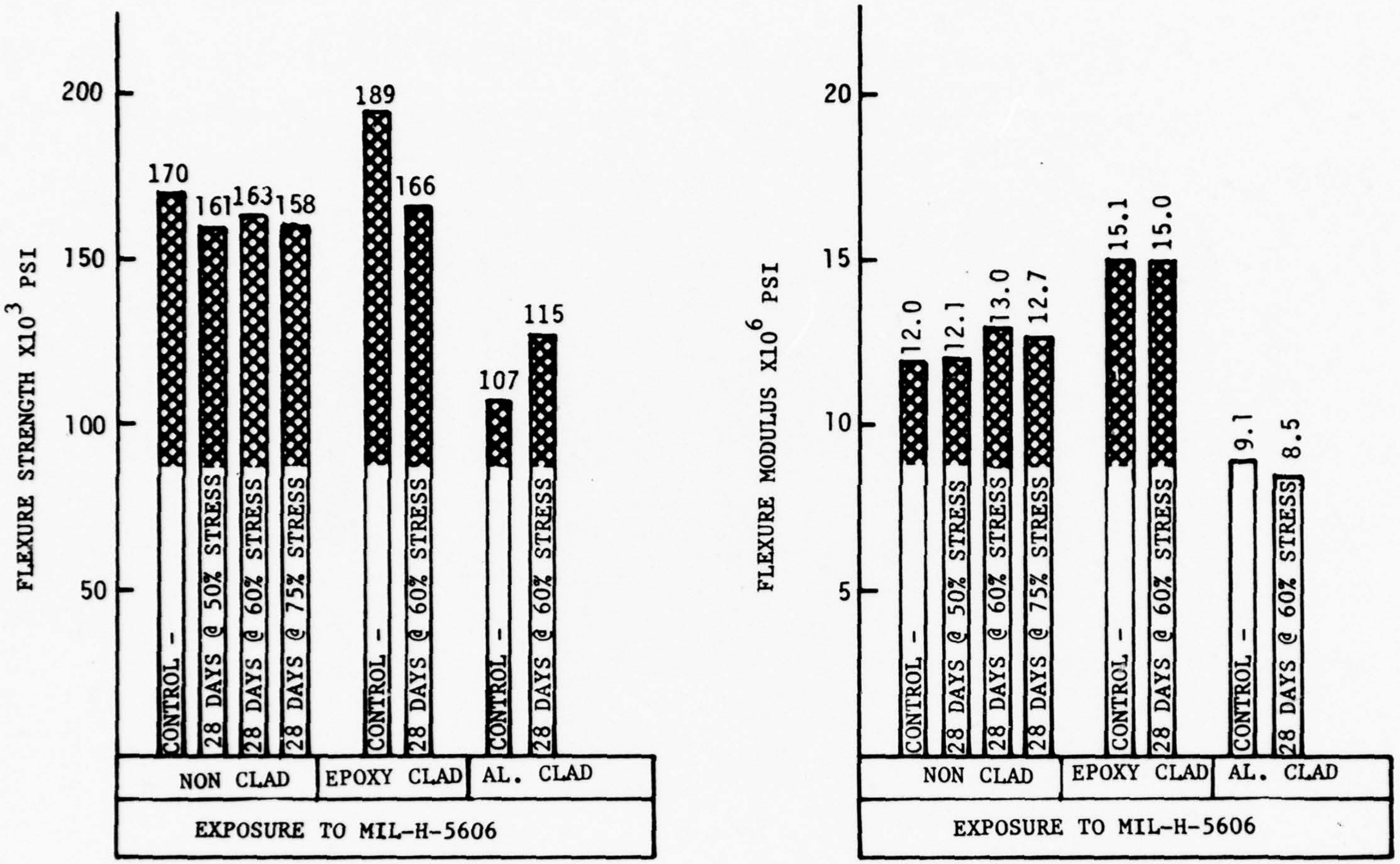

FIGURE 13 FLUID RESISTANCE OF P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES

MIL-H-83306 Hydraulic Fluid

MIL-H-83306 fluid had a significant effect on the stressed flexural specimens. Non-clad specimens exposed for 7, 28 and 50 days decreased in flexure strength from a range of 56 - 75%. The epoxy and metal cladding did provide some protection for the components except in the elevated temperature environment. These specimens had taken permanent set and/or delaminated. These data are reported in Figure 14. MIL-H-83306 is a phosphate ester type hydraulic fluid. Since phosphate esters are strong plasticizers, it is suspected that the permanent set and/or delamination in the laminates are the result of an increase in plasticity of the epoxy and polysulfone polymers at elevated temperature prior to a chemical attack on the polymer chain.

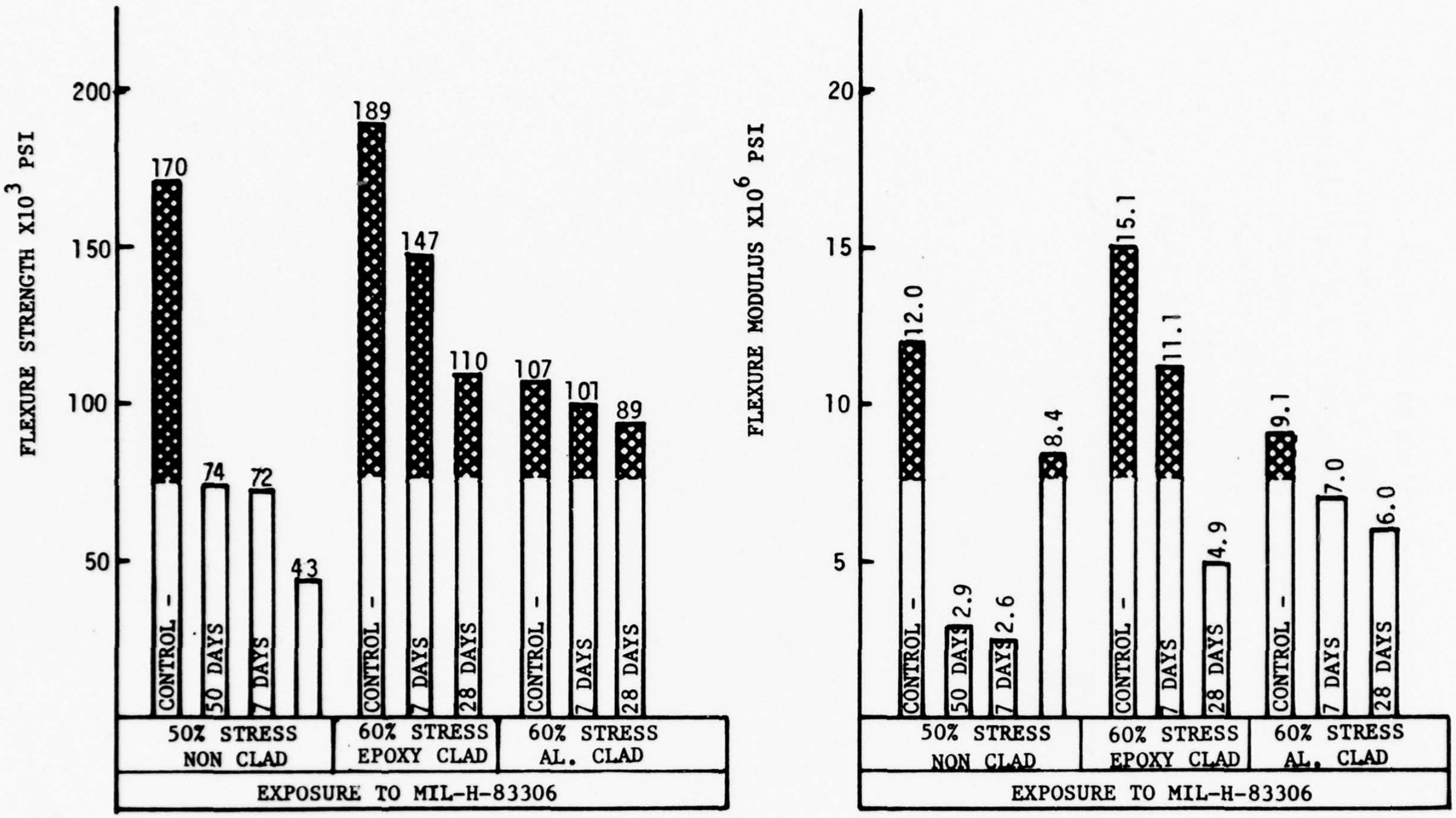

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FIGURE 14 FLUID RESISTANCE OF P-1700 CLAD AND NONCLAD GRAPHITE LAMINATES

2.3 PHASE III - HYBRID THERMOPLASTIC COMPOSITES

A promising means of protecting thermoplastic composites is through hybridizing. Hybridizing composites with various types of reinforcements improves performance and makes the best utilization of the properties of the different reinforcements.

2.3.1 Fiber Hybridizing

Process development and material evaluation studies were performed on three hybrid laminates. These fiber hybrid laminates were fabricated from glass/graphite, glass/Kevlar-49, and Kevlar-49/graphite fabrics with P-1700 polysulfone matrix.

Prepregs for this phase of work were made in the laboratory. The fabric materials were impregnated to a resin content of 34% by weight with P-1700 polysulfone resin. All prepregs were dried until they reached constant weight in an air circulating oven at 225 - 250°F.

Each laminate was fabricated from two different reinforcing fiber materials. The ply arrangement for the laminates is shown in Table 3.

The laminates were molded in a press using the same molding cycle used in earlier laminate fabrication (Section 2.1.2).

Tensile, flexural, interlaminar shear (ASTM D2345) and izod impact specimens were fabricated from the laminates. The laminates for the izod impact specimens were fabricated using 30 plies of prepreg, rather than 10 plies as outlined in Table 3, using the same alternate layers.

Table 3: HYBRID FABRIC COMPOSITE LAYOUT [1]

PLY	COMPOSITE		
	Glass/Graphite	Kevlar/Glass	Kevlar/Graphite
1	Glass	Glass	Graphite
2	Graphite	Kevlar	Kevlar
3	Glass	Glass	Graphite
4	Graphite	Kevlar	Kevlar
5	Glass	Glass	Graphite
6	Glass	Glass	Graphite
7	Graphite	Kevlar	Kevlar
8	Glass	Glass	Graphite
9	Graphite	Kevlar	Kevlar
10	Glass	Glass	Graphite

[1] NOTE: Glass = 181 Style S-glass/9 mil
Graphite = 181 Style Graphite/11.2
Kevlar = 181 Style Kevlar-49/9 mil

Tensile, flexure and interlaminar specimens were tested at -65°F, RT and 300°F. Izod impact specimens were tested at room temperature only. Test results are reported in Table 4 and Figure 15.

Photographs of the tested specimens are shown in Figures 16 through 27.

TABLE 4 HYBRID POLYSULFONE/FABRIC COMPOSITE DATA

COMPOSITE	TEST TEMP. °F	TENSILE Stress ($x10^3$ psi)	TENSILE Mod. ($x10^6$ psi)	FLEXURAL Stress ($x10^3$ psi)	FLEXURAL Mod. ($x10^6$ psi)	INTERLAMINAR Shear Stress psi
Glass/Graphite	-65	61.4	5.6	83.7	5.5	6450
	RT	58.9	5.4	78.8	5.6	5130
	300	56.7	5.6	45.5	4.7	2995
Kevlar/Glass	-65	56.3	4.2	73.9	4.2	4195
	RT	47.4	3.5	64.6	3.3	3975
	300	46.0	3.3	45.4	3.6	2845
Kevlar/Graphite	-65	56.2	7.0	73.1	5.4	5300
	RT	53.4	6.9	69.0	5.8	4935
	300	53.1	7.2	49.9	4.8	3315

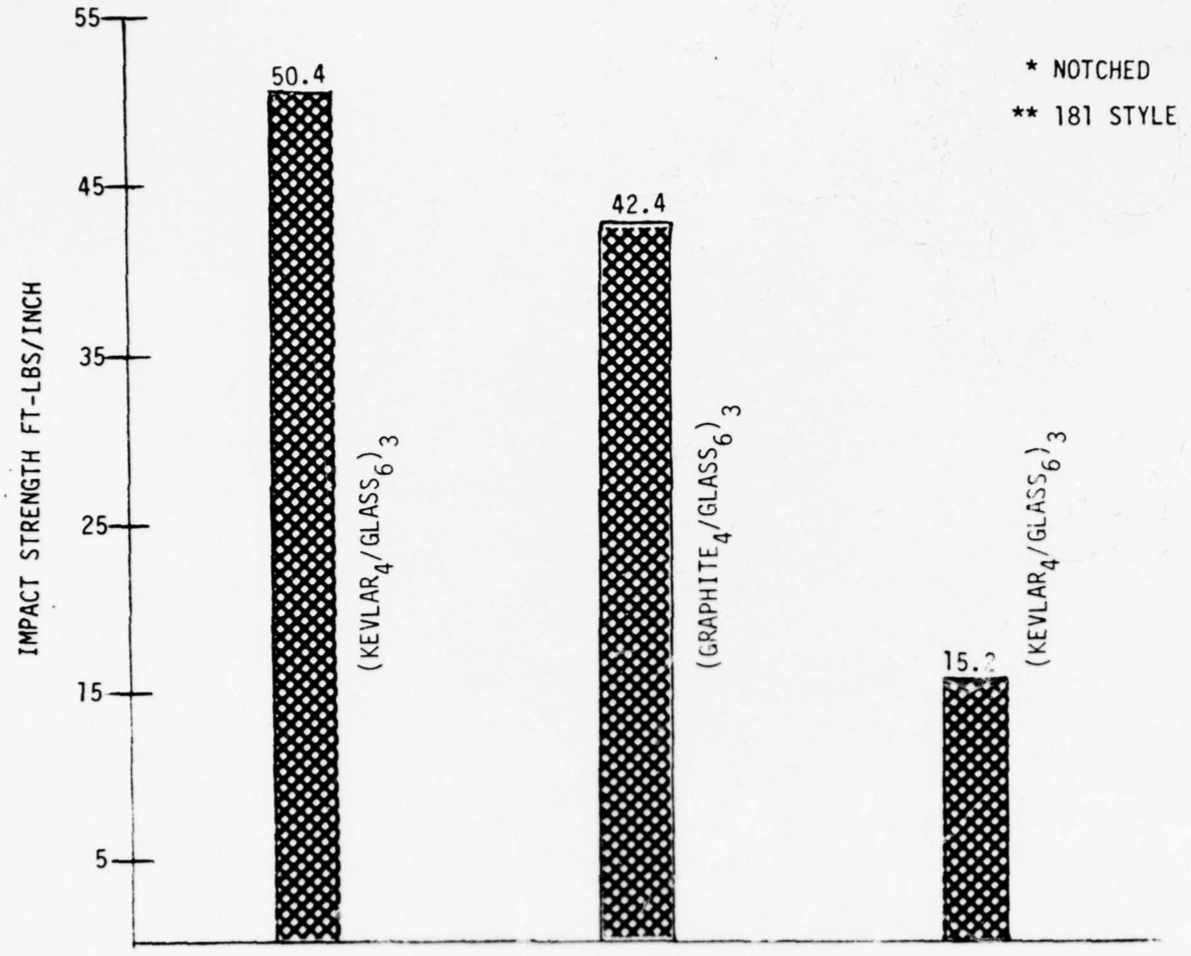
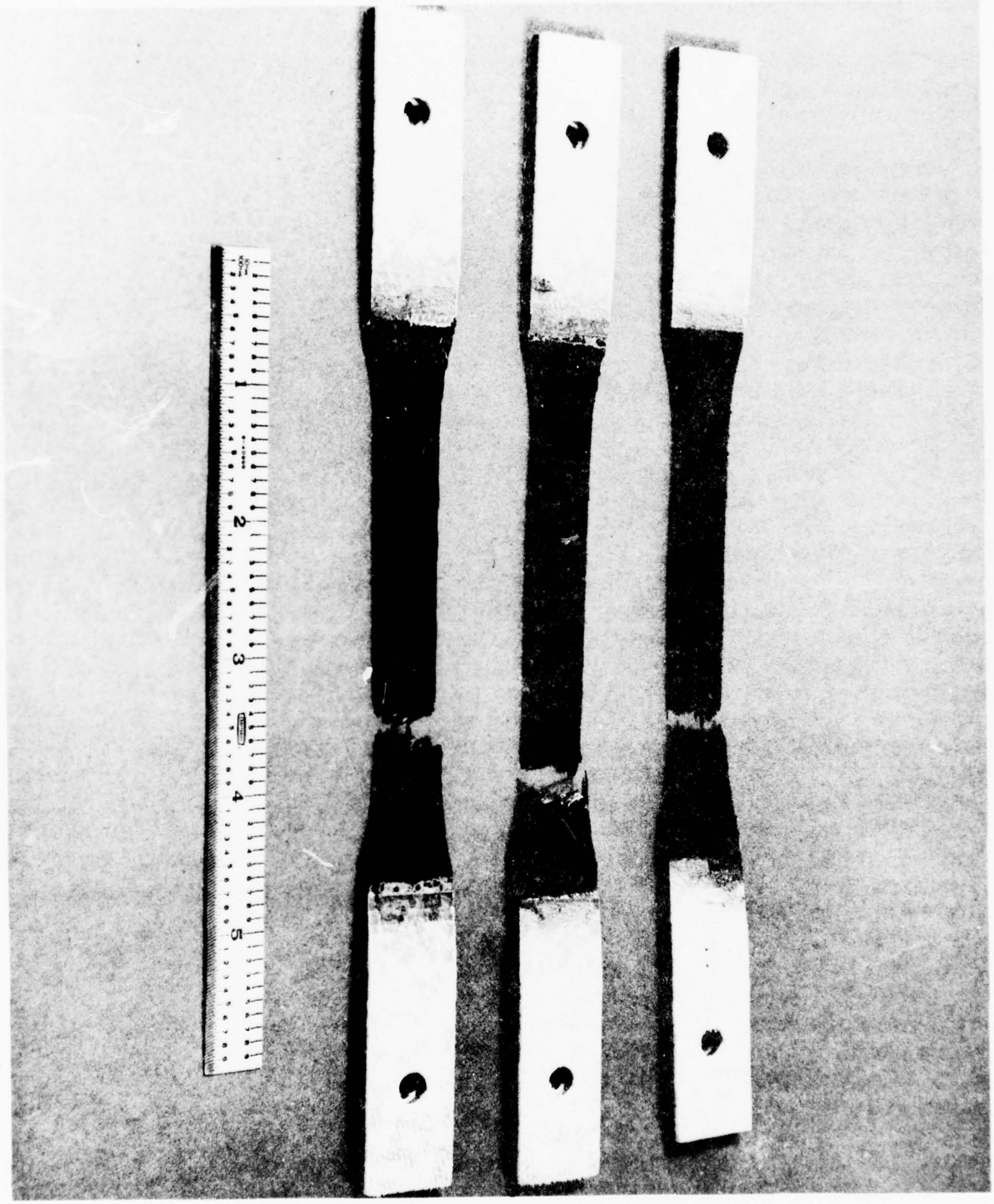
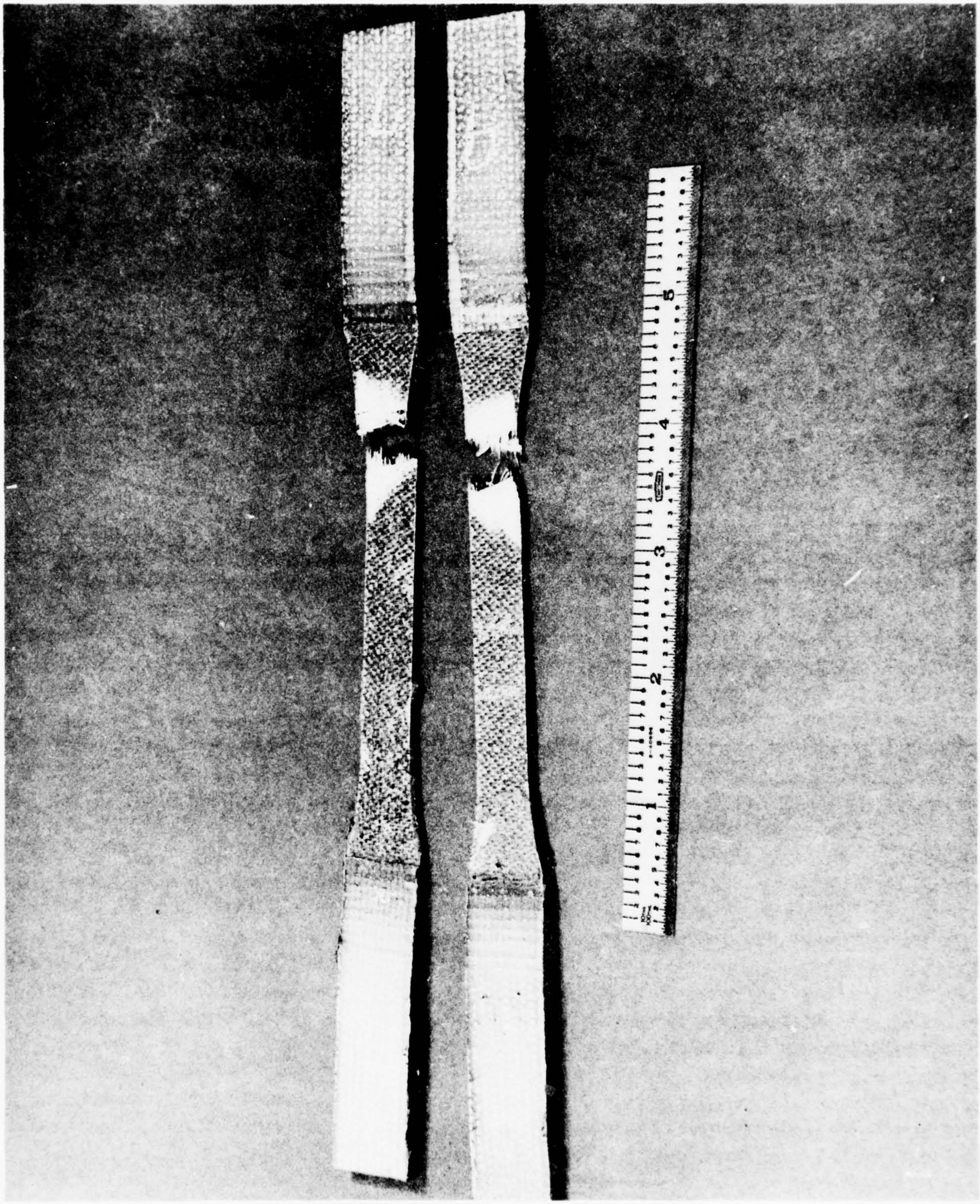


FIGURE 15 IZOD IMPACT STRENGTH* OF POLYSULFONE/FABRIC** COMPOSITES



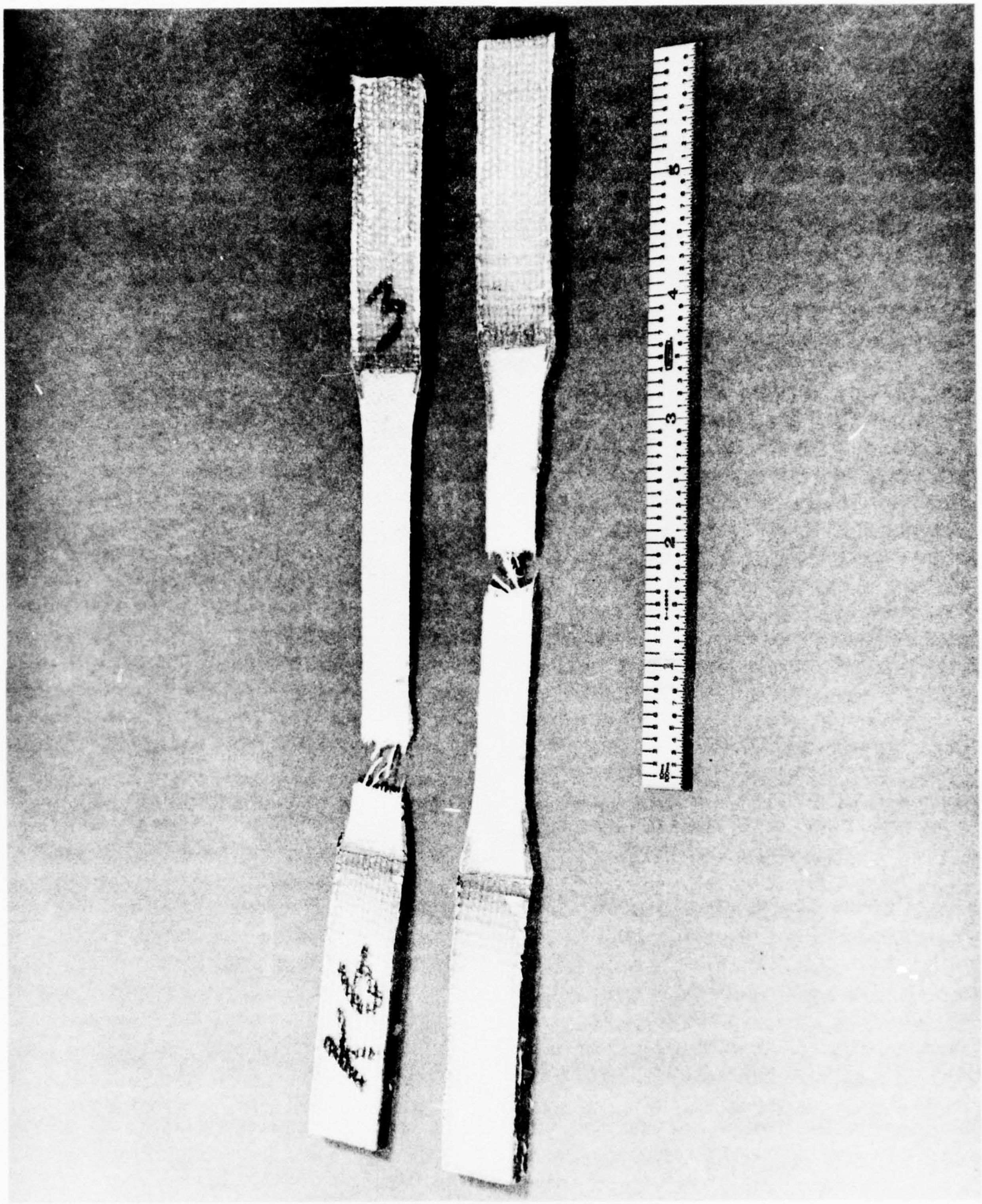
P-1700/GRAPHITE/S-GLASS FABRIC

Figure 16: Tensile Specimens – Typical Fractures



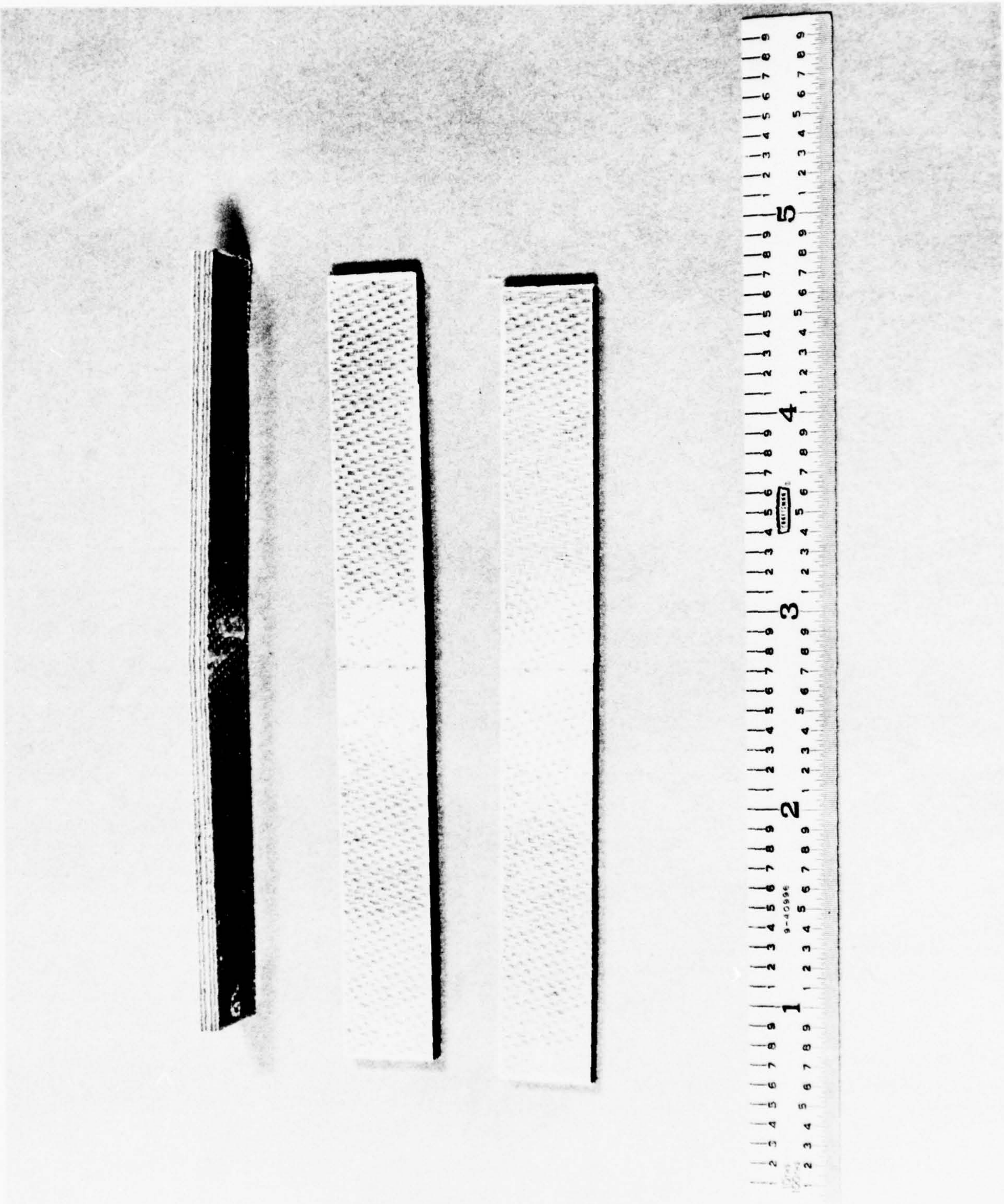
P-1700/KEVLAR/GRAPHITE FABRIC

Figure 17: Tensile Specimens – Typical Fractures



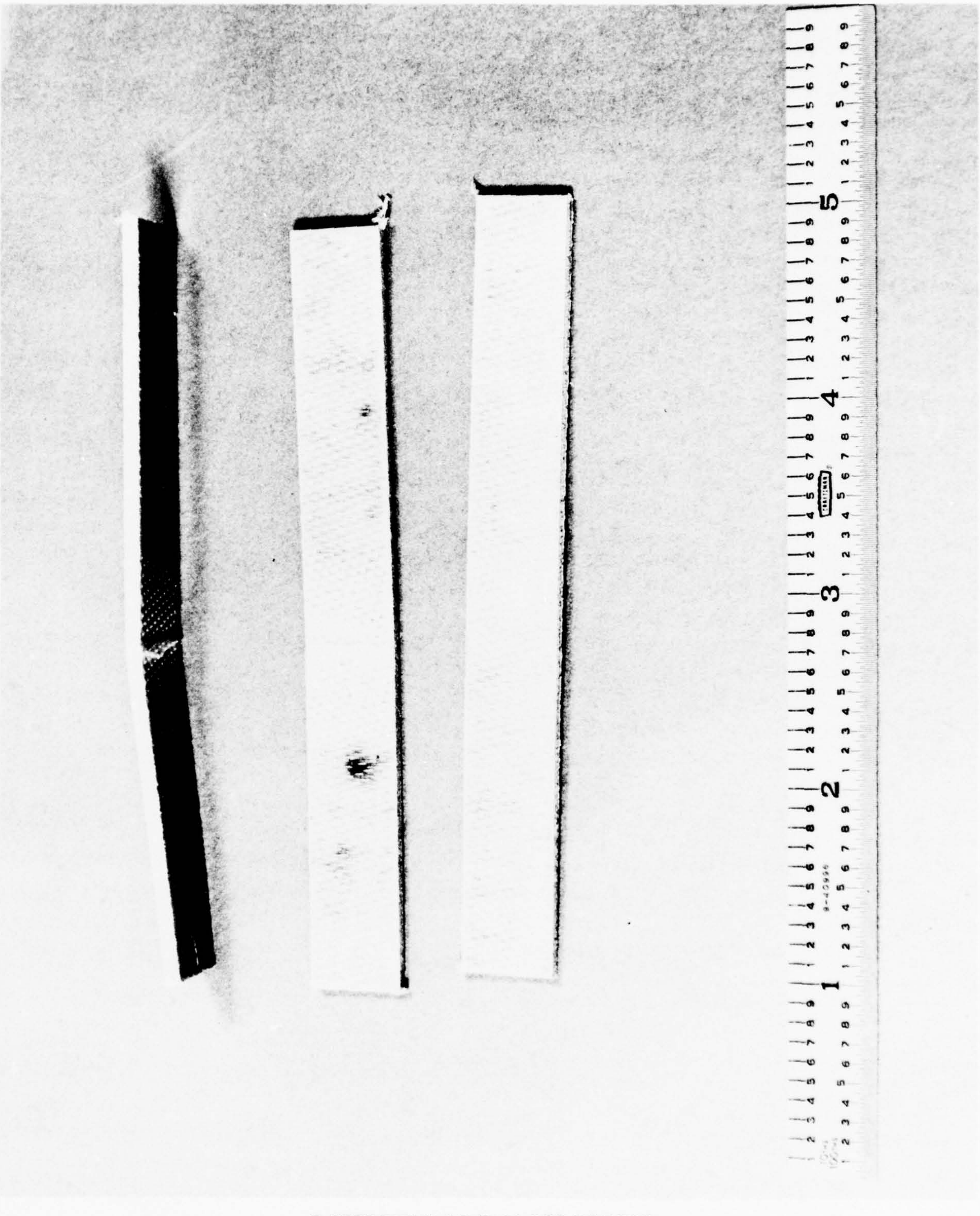
P-1700/KEVLAR/S-GLASS FABRIC

Figure 18: Tensile Specimens – Typical Fractures



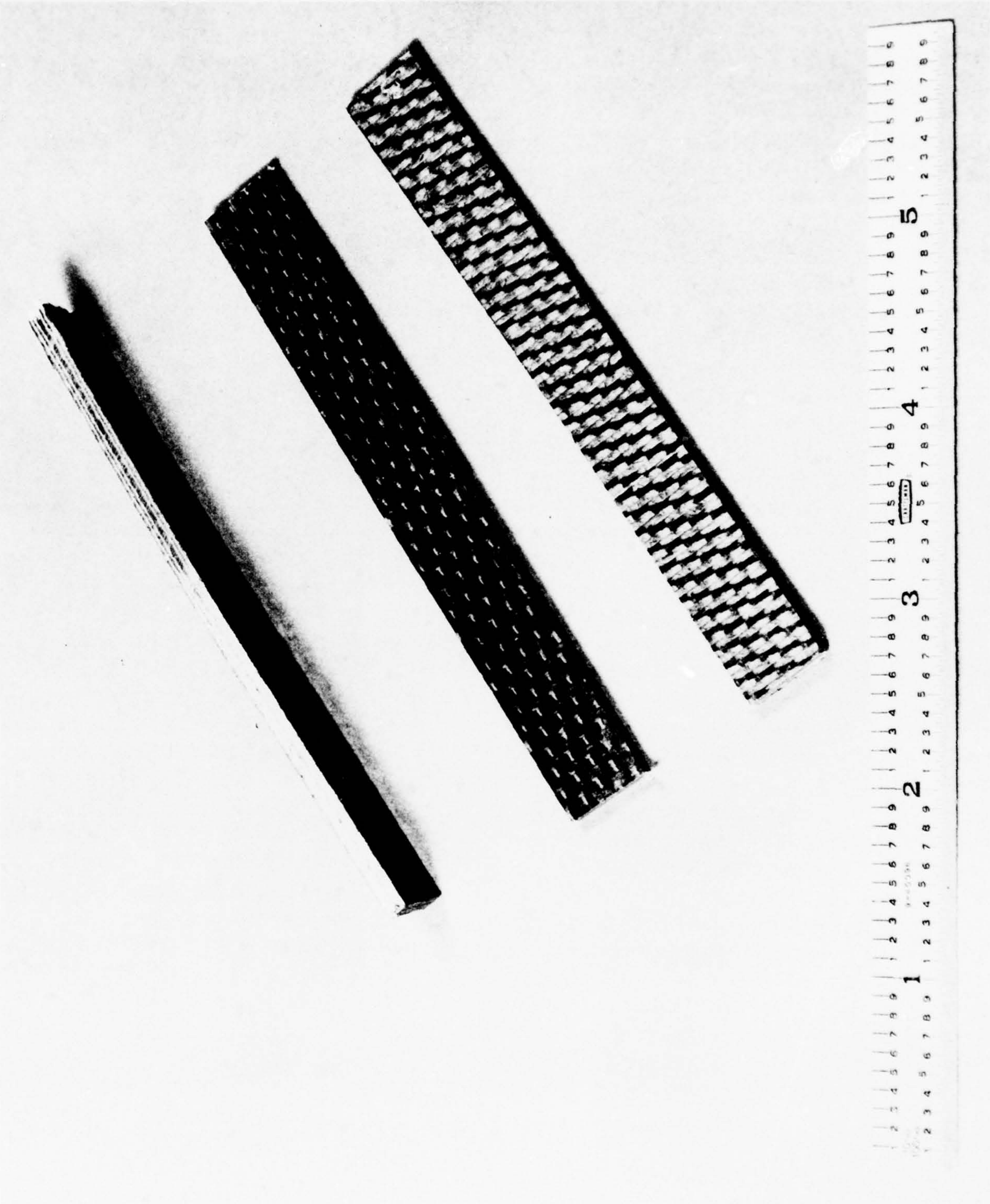
P-1700/KEVLAR/GRAPHITE FABRIC

Figure 19: Flexure Specimens – Typical Fractures



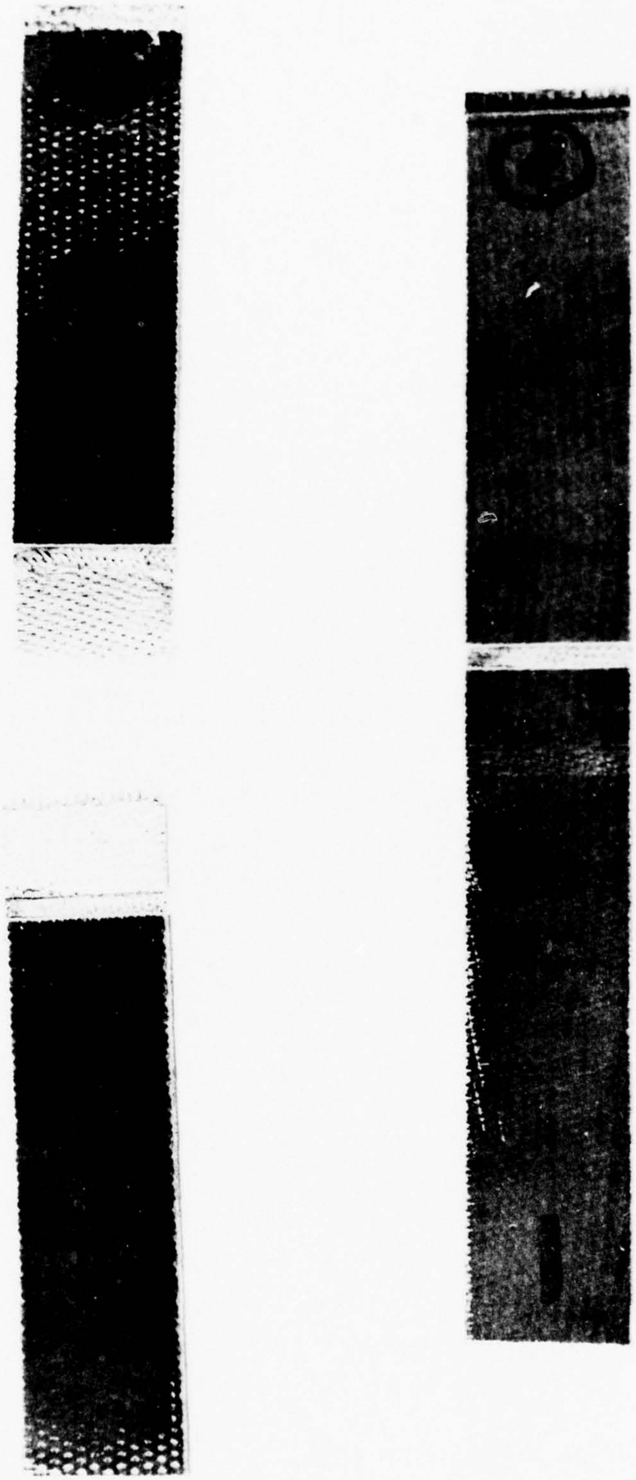
P-1700/KEVLAR/S-GLASS FABRIC

Figure 20: Flexure Specimens – Typical Fractures



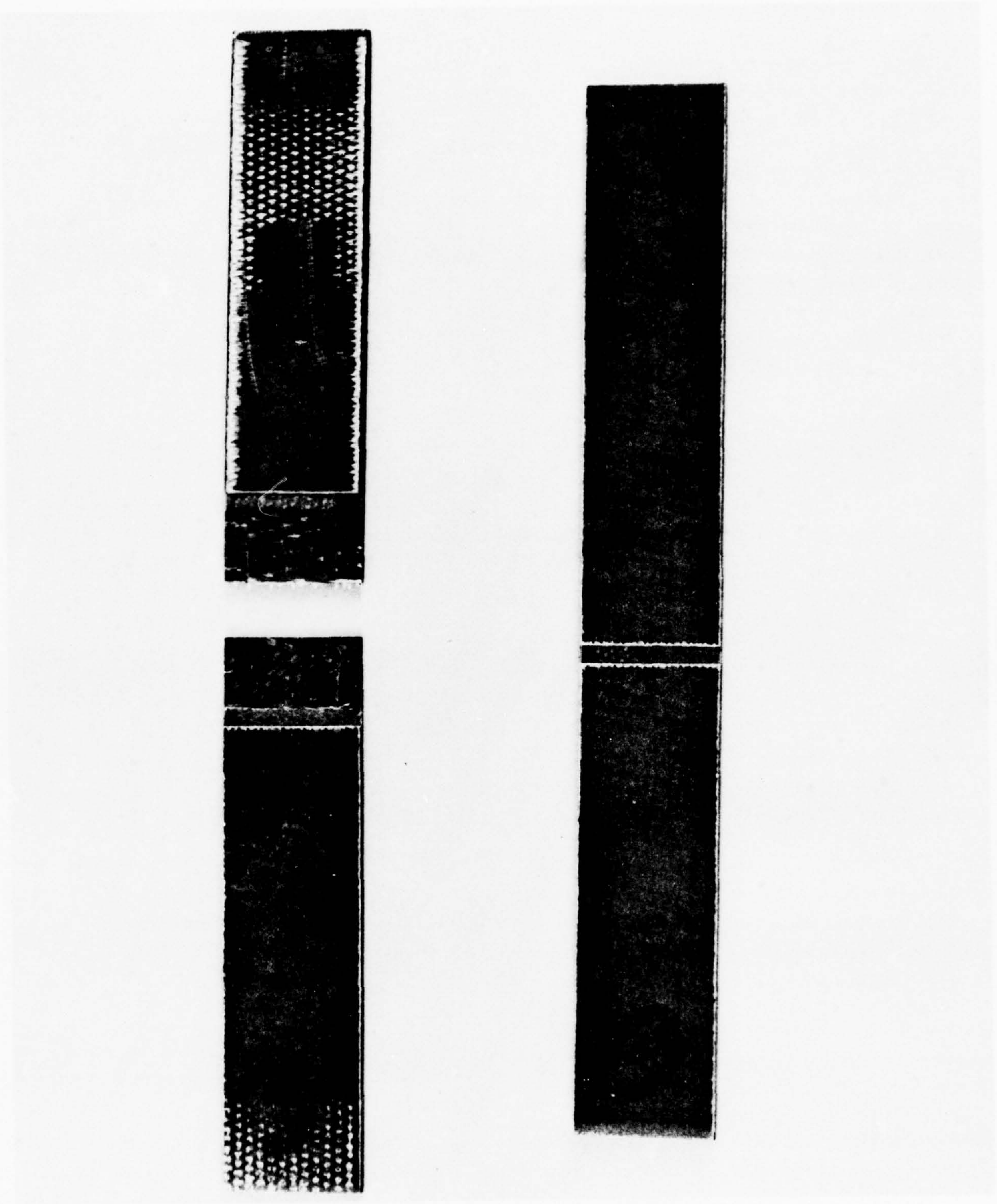
P-1700/GRAPHITE/S-GLASS FABRIC

Figure 21: Flexure Specimens – Typical Fractures



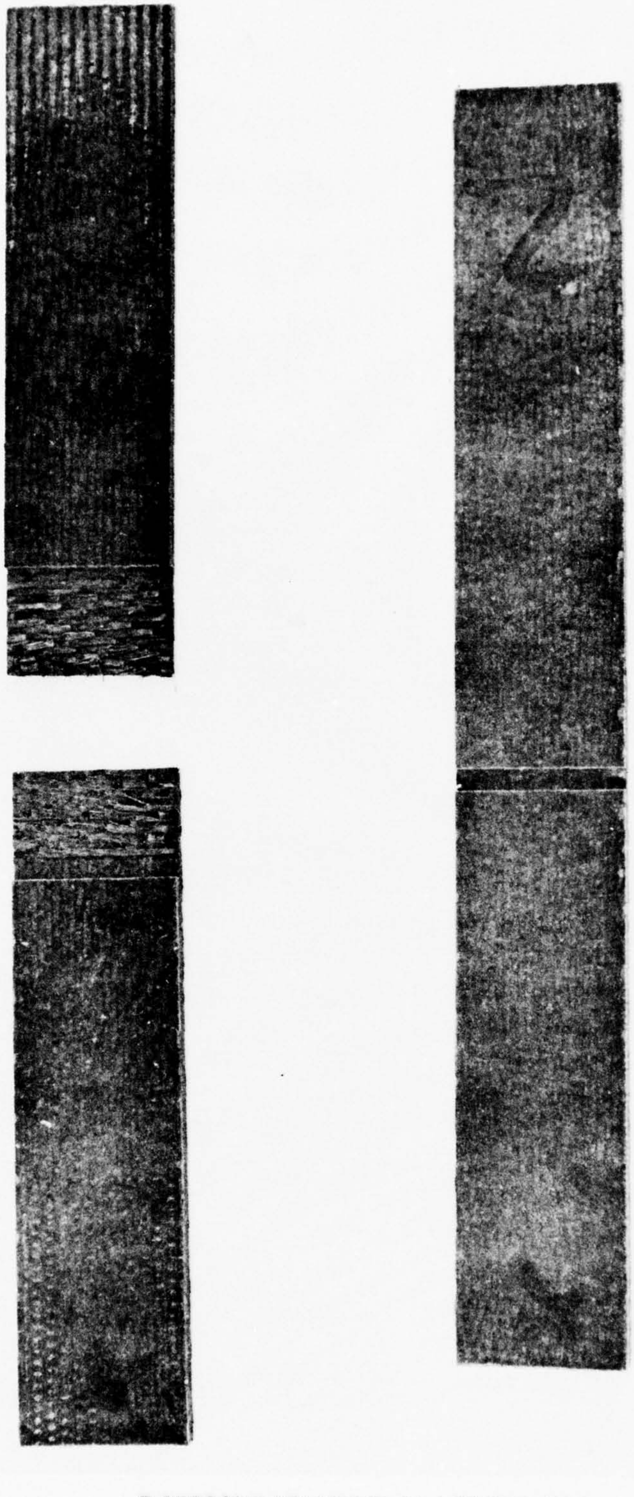
P-1700/KEVLAR/S-GLASS FABRIC

Figure 22: Interlaminar Shear Specimens – Typical Fracture



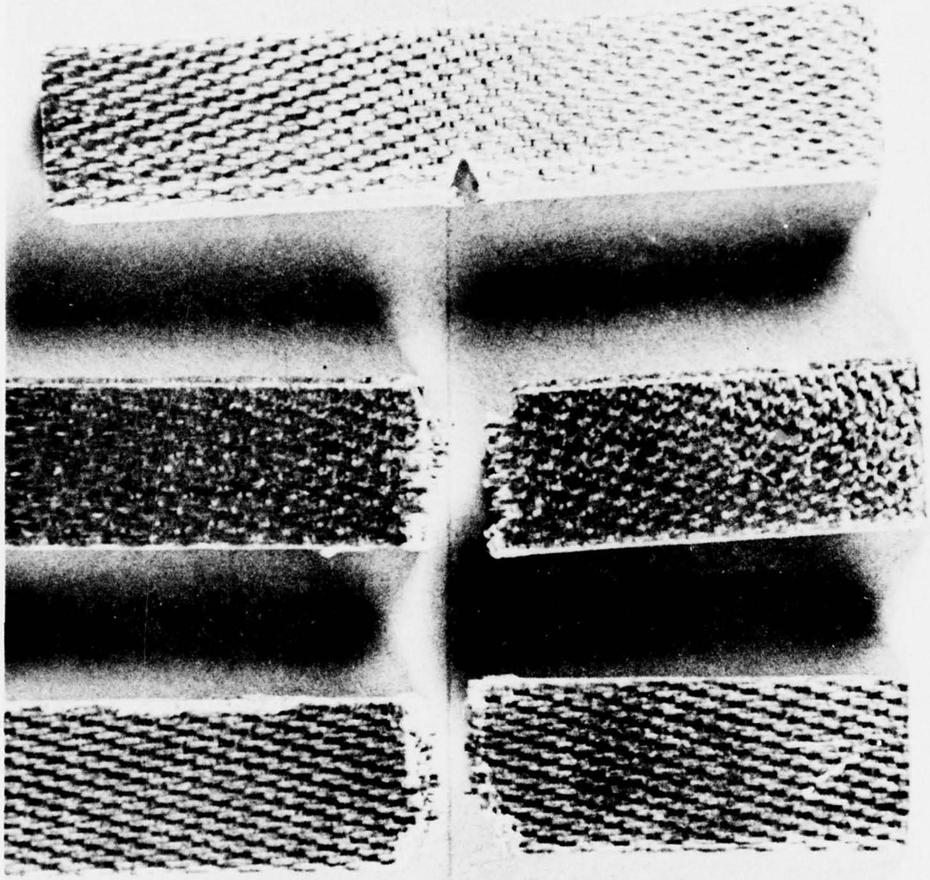
P-1700/KEVLAR/GRAPHITE FABRIC

Figure 23: Interlaminar Shear Specimens – Typical Fracture



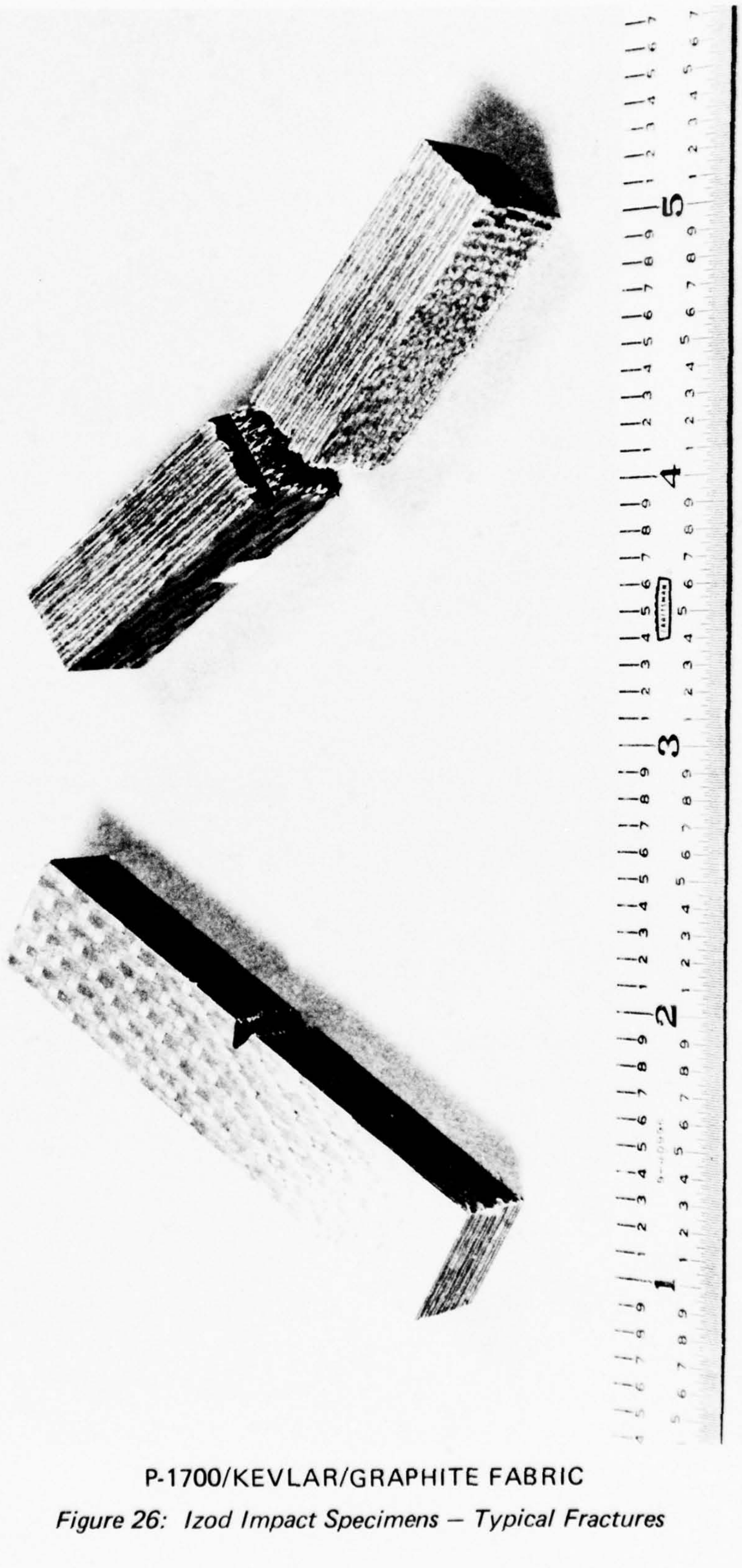
P-1700/GRAPHITE/S-GLASS FABRIC

Figure 24: Interlaminar Shear Specimens – Typical Fracture



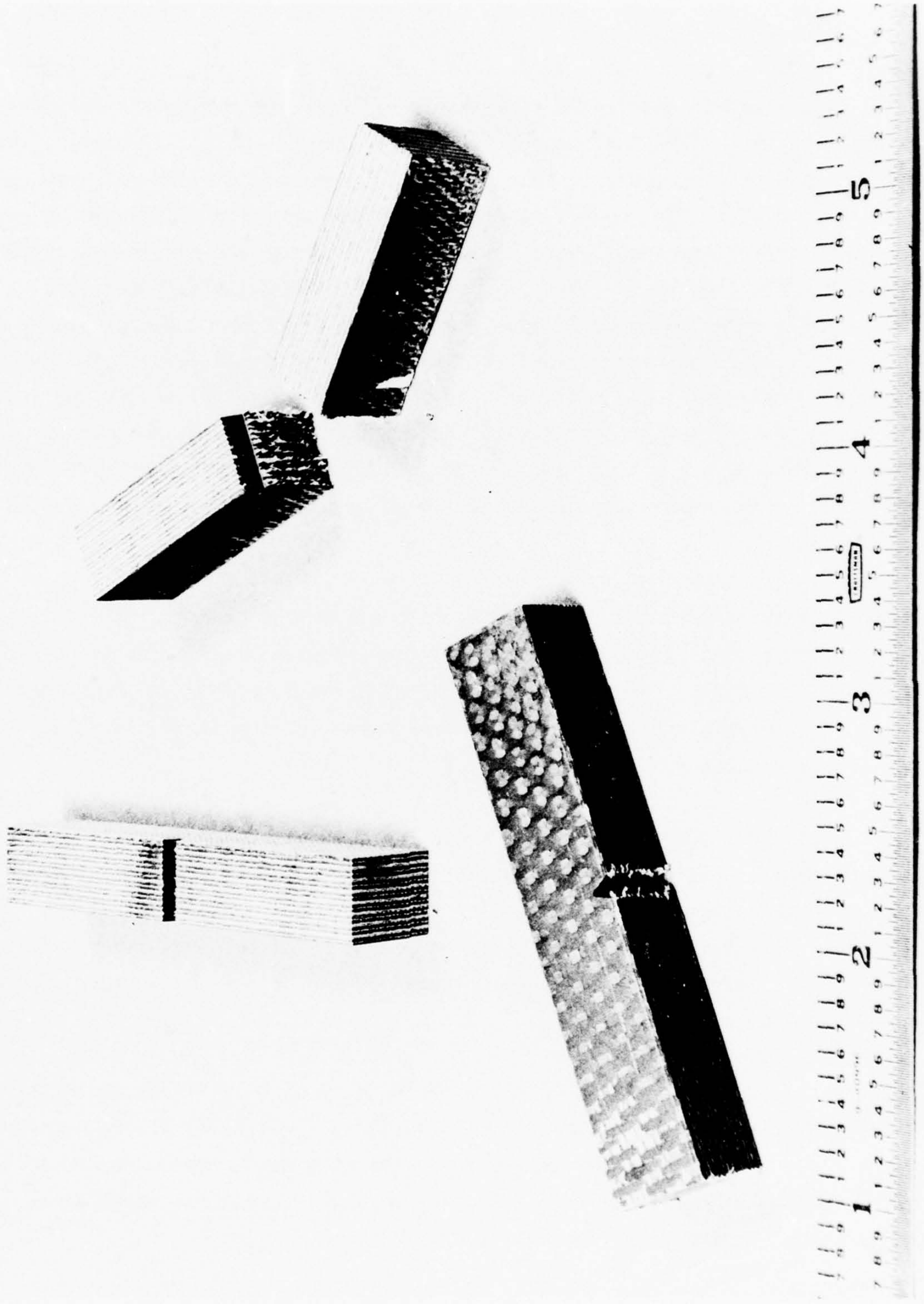
P-1700/KEVLAR/S-GLASS FABRIC

Figure 25: Izod Impact Specimens – Typical Fractures



P-1700/KEVLAR/GRAPHITE FABRIC

Figure 26: Izod Impact Specimens – Typical Fractures



P-1700/GRAPHITE/S-GLASS FABRIC

Figure 27: IZOD Impact Specimens – Typical Fractures

2.3.2 Metal-Clad Hybridizing

2024T3 clad aluminum and 17-7PH stainless steel were the two metals selected for cladding the P-1700/A-S composites. Two different surface treatments were used for each metal selected. Phosphoric acid anodize and sodium dichromate-sulfuric acid etch were the two surface treatments used for cleaning aluminum. The steel surface treatments were sulfuric acid anodize and nitric-hydrofluoric acid etch. The treated surfaces of .005" steel, .011" aluminum, and .016" aluminum were primed with BR127 epoxy primer from American Cyanamid Company and bonded to P-1700/A-S unidirectional laminates per Section 2.2.1.1. The number of plies of P-1700/A-S and configuration of the laminate varied with the test being conducted and are reported with the test data. Tension, compression, bearing strength, and metal clad to metal clad lap shear specimens were fabricated from these assemblies and tested. Test results are reported in Tables 5, 6, and 7.

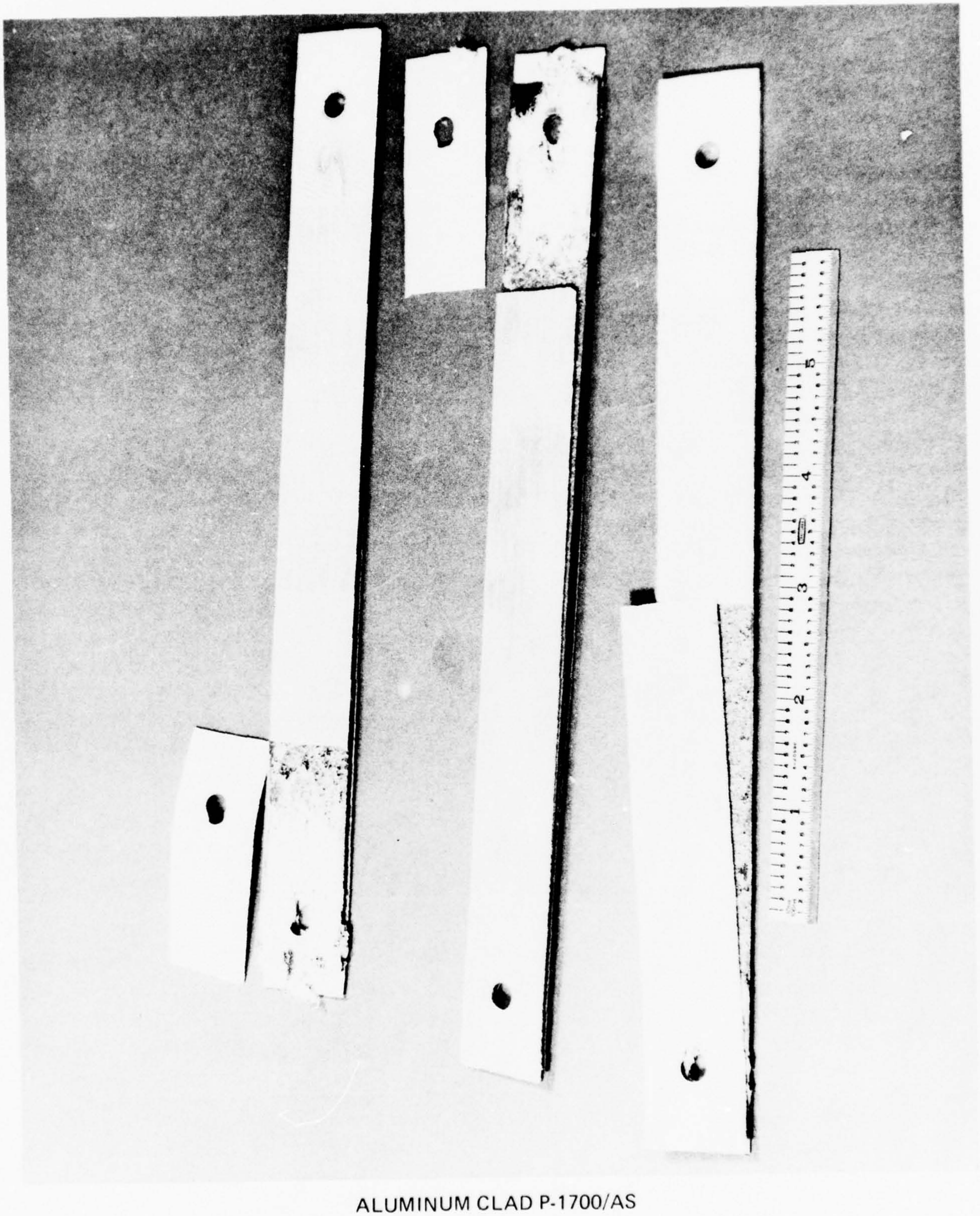
The initial failure of the tension specimens was in the cladding materials. The steel stretched and buckled during testing, causing a delamination of the clad under the buckled area. The aluminum cladding stretched and broke prior to complete delamination of the clad. Photographs of the failed tension specimens are shown in Figures 28 and 29.

The bearing specimens failed in the metal and composite materials uniformly with the metal cladding carrying most of the load. The tension load on the pin caused the unidirectional fibers to slide in shear. The addition of cross fibers in the bearing area would force the unidirectional fibers to carry higher stress loads before failing. Figures 30 and 31 are photographs of the failed bearing specimens.

It was noted by Boeing that cold post-forming is possible in some thicknesses of aluminum cladding on thermoplastic composites. Twelve-mil aluminum-clad composites with $(0, +45, -45, 90)_S$ ply arrangements were fabricated and cold post-formed by hand. The 0° and 45° plies were unidirectional graphite tape. The 90° plies were S-glass fabric (181 style).

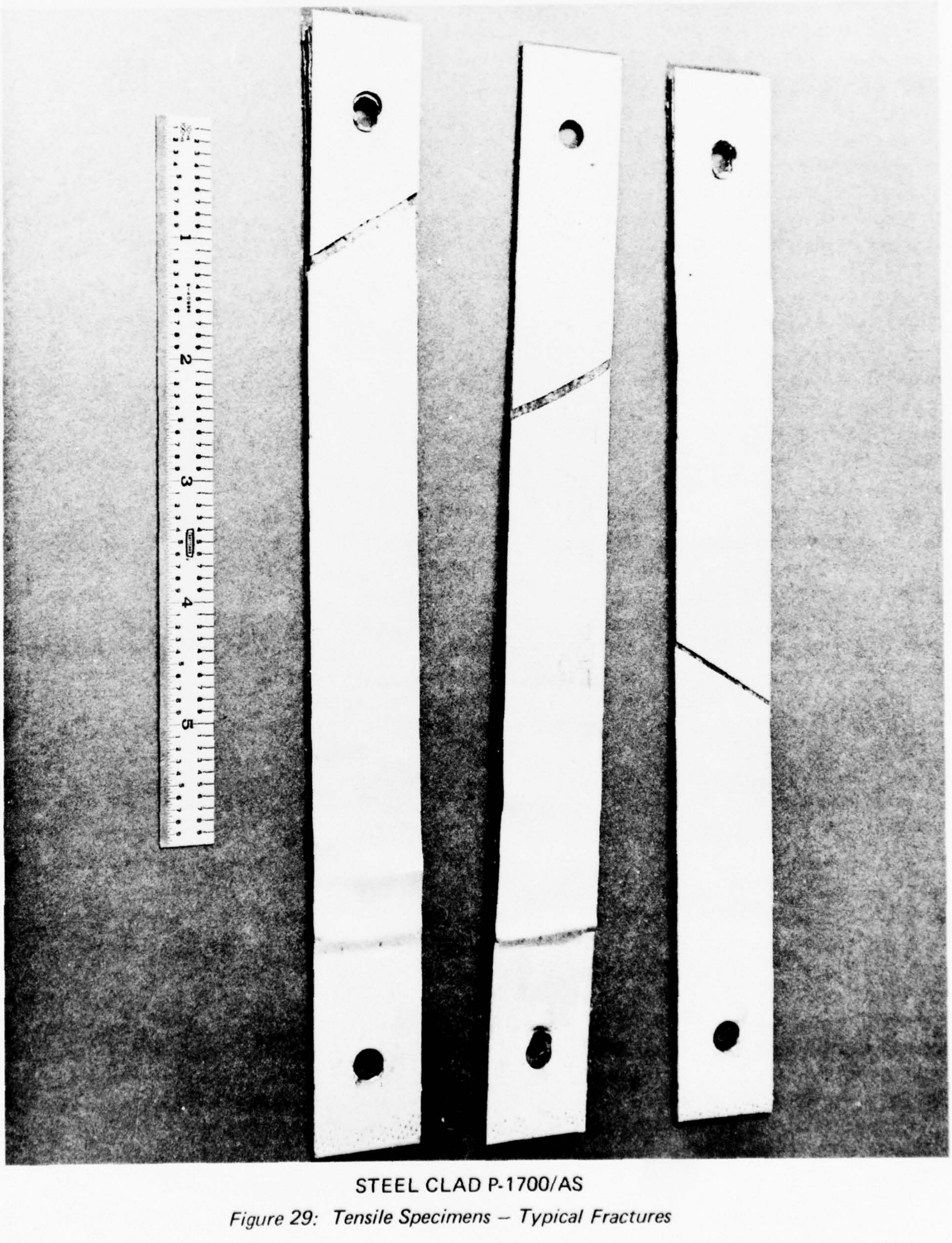
TABLE 5 METAL CLAD LAMINATE PROPERTY DATA

Cladding on 12-Ply P-1700/AS Laminate	Test Temp. °F	Compression		Tensile		Bearing
		Strength x 10^3 psi	Modulus x 10^6 psi	Strength x 10^3 psi	Modulus x 10^6 psi	Strength x 10^3 psi
Steel Clad	RT	55.0	31.3	118.4	36.4	100.8
.005"	300	22.5	30.1	55.8	36.1	70.1
Aluminum Clad	RT	46.3	24.9	95.5	25.7	69.5
.011"	300	17.4	12.2	22.9	13.3	48.1
.016"	RT	-	-	-	-	61.3
	300	-	-	-	-	38.2



ALUMINUM CLAD P-1700/AS

Figure 28: Tensile Specimens – Typical Fractures



STEEL CLAD P-1700/AS

Figure 29: Tensile Specimens – Typical Fractures

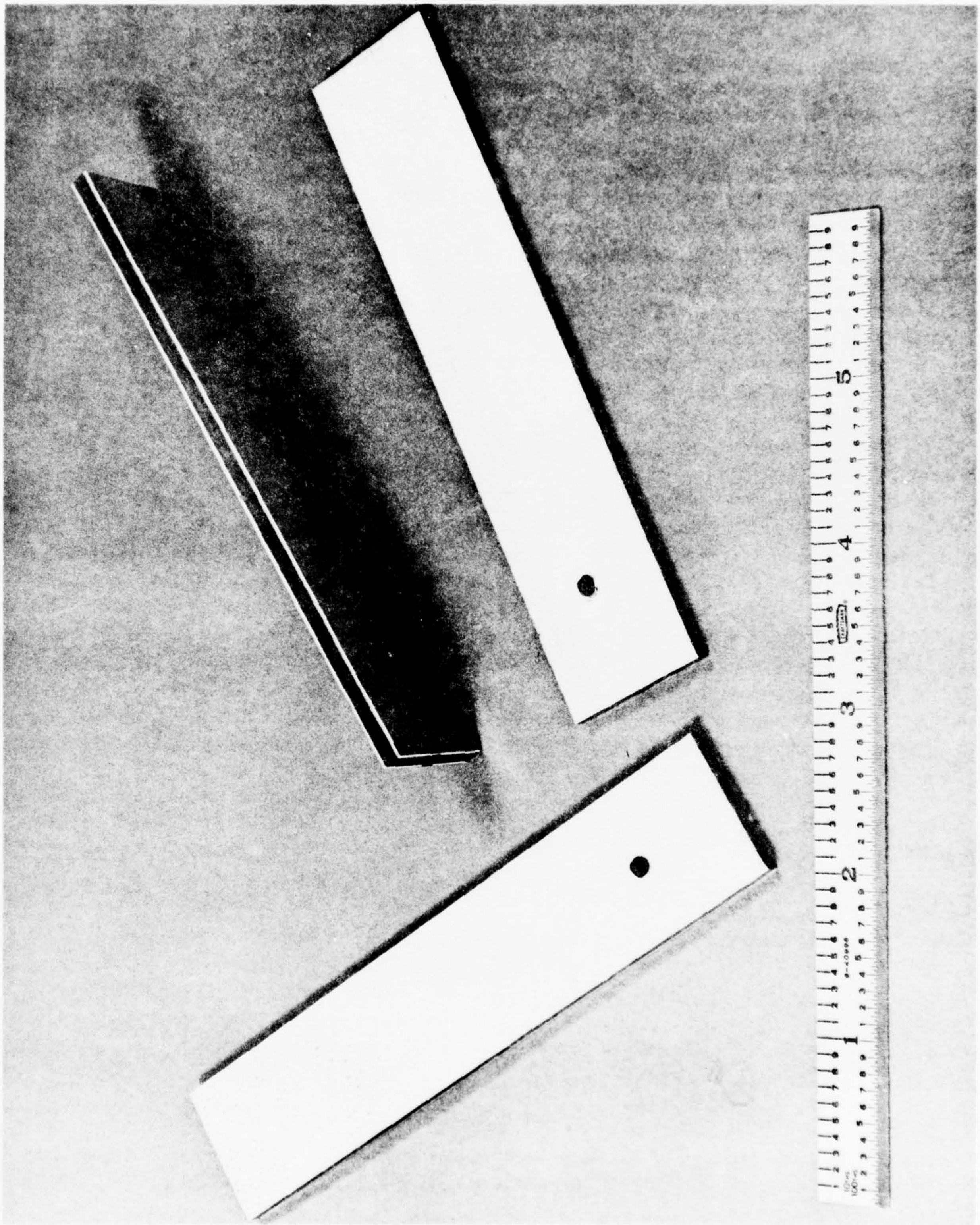


Figure 30: Steel-Clad P-1700/A-S Bearing Specimens — Typical Failure

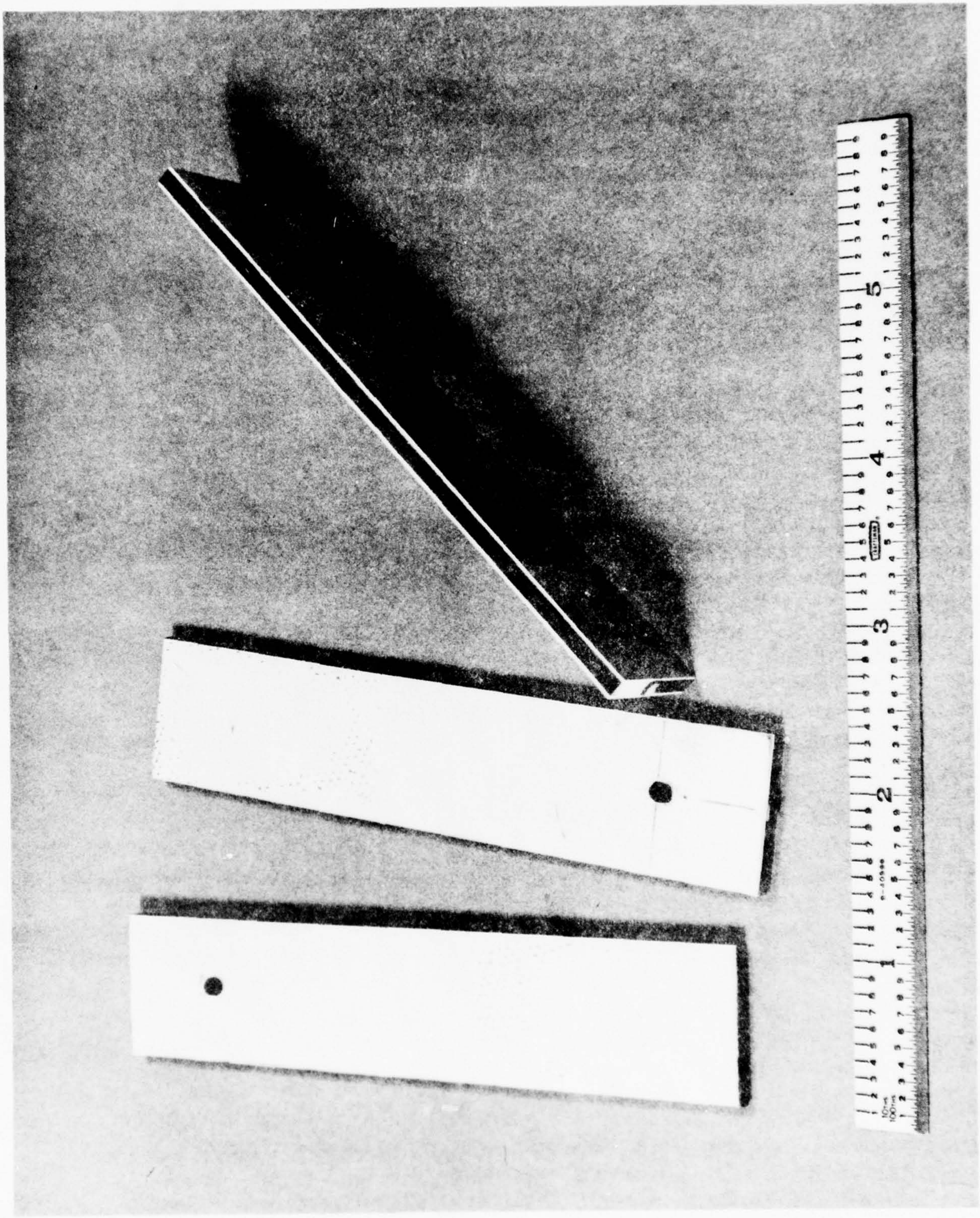


Figure 31. Aluminum Clad P-1700/A-S Bearing Specimens — Typical Failure

Under this contract, .012" and .016" aluminum-clad P-1700/A-S composites were fabricated for cold post-forming. The composites were fabricated from P-1700/A-S tape per Section 2.2.1.1 using (0, +45, -45, +45, 0) and $(0, +45, -45, 90)_S$ as the ply arrangements. These laminates could not be cold post-formed by hand. It appears that cold post-forming of metal-clad P-1700/A-S composites can only be accomplished when a glass fabric is present in the center of the composite.

Metal clad flexure specimens were environmentally exposed to various fluids while under stress loads of 60% of ultimate. These data are reported in Table 6 and Figures 8 through 14.

Of all the fluid tests, the most severe appeared to be MIL-H-83306. At 150°F in two days, the clad laminate delaminated with the fluid attacking the laminate rather than the metal/composite interface. This result was expected as reported earlier in Phase II (Figure 14) wherein the fluid attacked the composite at the edges where there was no clad protection. These metal-clad specimens are shown in Figure 32. However, these specimens were not tested because of delamination and/or permanent set in the specimens from the 150°F MIL-H-83306 hydraulic fluid.

Some metal clad specimens were epoxy sealed on edges prior to exposure in MIL-H-83306 at 150°F. These specimens delaminated in the environment also. Photographs of these specimens are shown in Figure 33. Specimens delaminated within the P-1700/A-S composite.

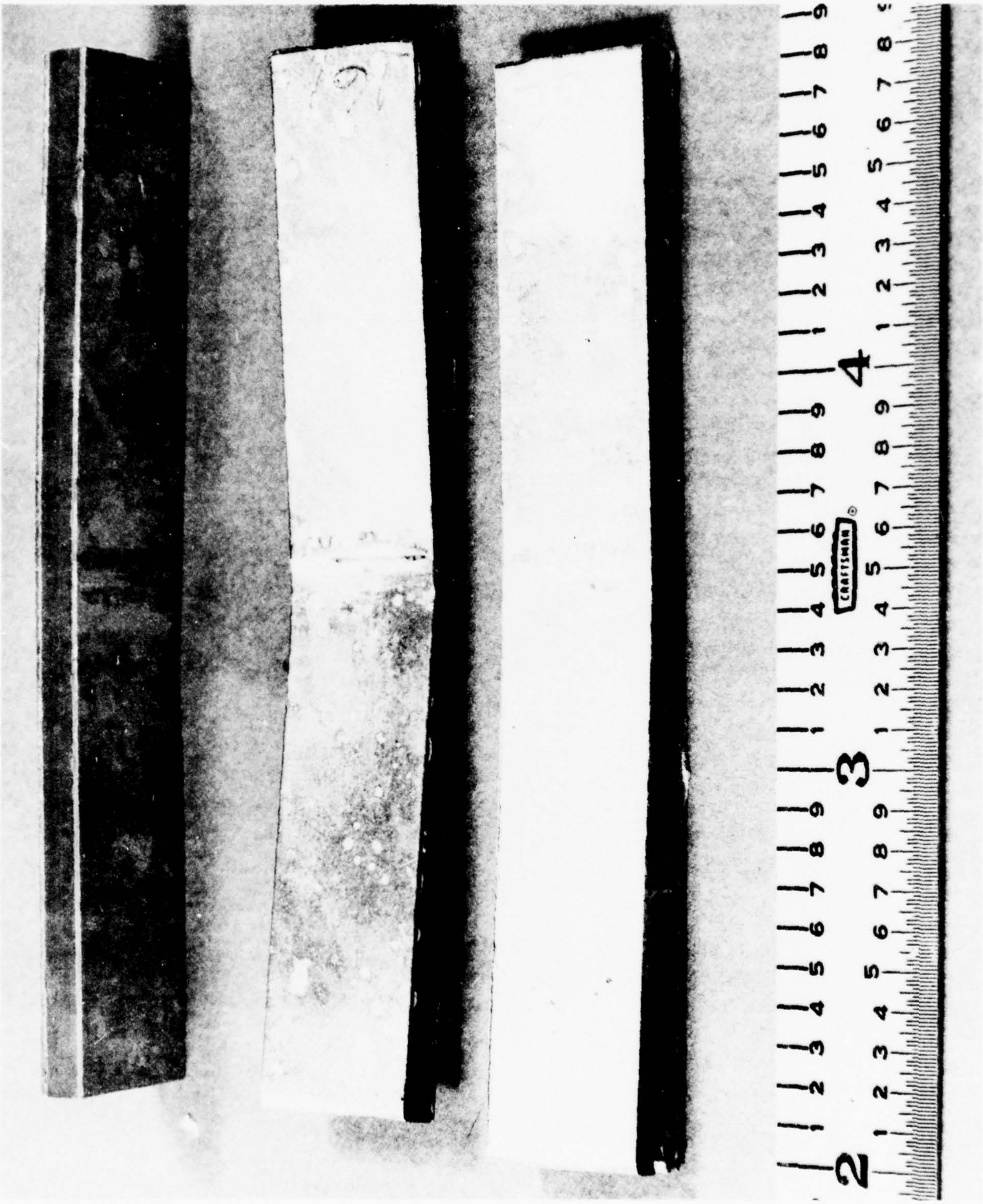
Standard 4" x 6" lap shear panels were fabricated from the metal-clad laminates and bonded with 10-mil P-1700/112 E-glass film in a laboratory press. The lap length was 0.5". The construction and surface preparation of these specimens are shown in Table 7. Specimens were tested at -65°F, room temperature and 300°F. The failures were 85 - 95% cohesive.

Metal-clad laminates were exposed to 200 cycles of thermal shock (-65°F to +250°F) and thermal aging (500 hours at 250°F), to determine if differences in thermal expansion of the materials would cause delamination. The tests appeared to have no adverse effect on the composites. Figures 34 through 41 are photographs of the laminates before and after exposure.

TABLE 6 METAL-CLAD STRESSED/ENVIRONMENTAL EXPOSURE DATA

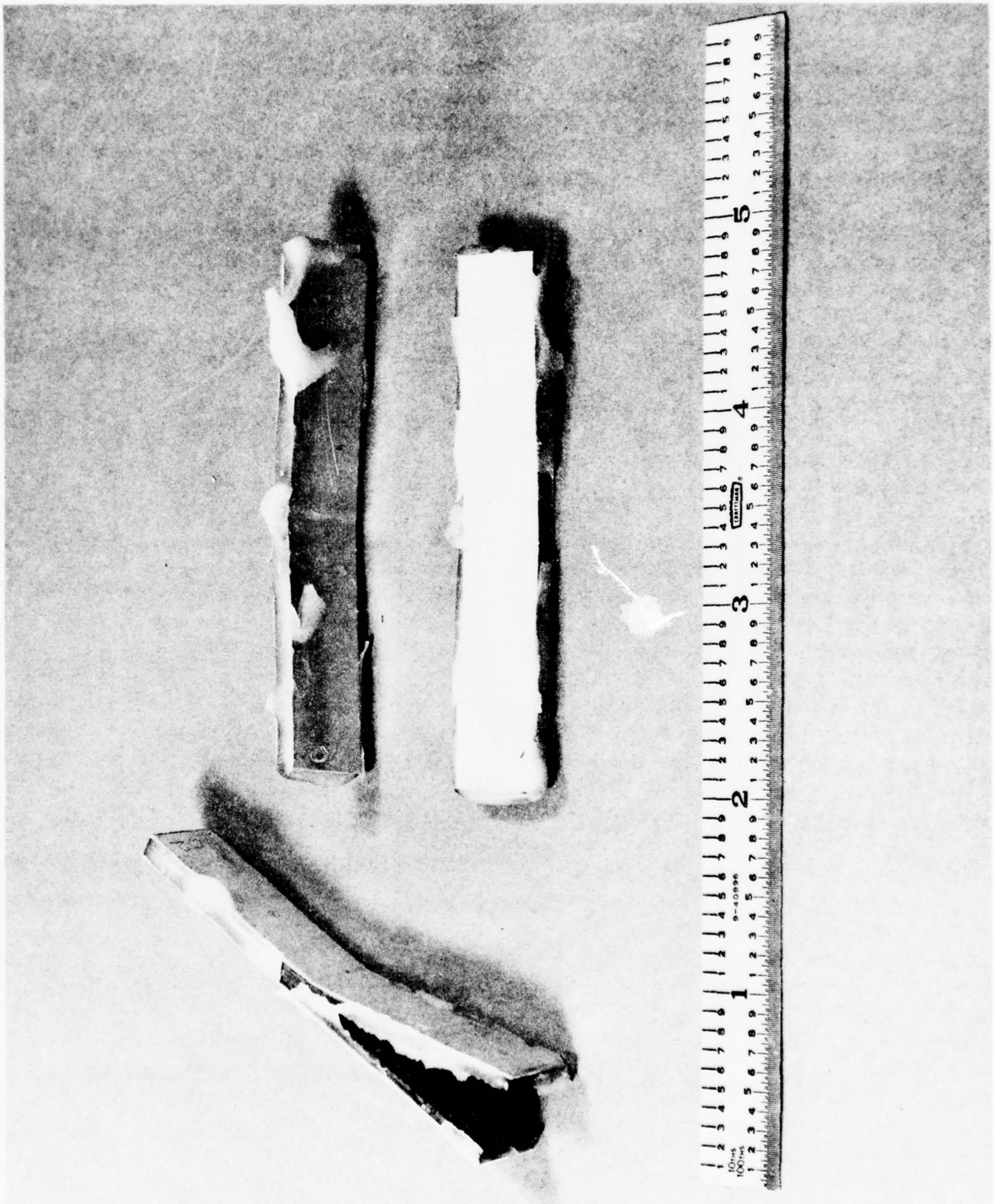
Exposure	Stress Level % of Ultimate	Environmental Flexural Data	
		Stress x 10^3 psi	Modulus x 10^6 psi
Control (No exposure)	0	106	9.1
JP-4 28 days	60	113	9.0
MIL-H-5606 28 days	60	114	8.5
MIL-L-7808 28 days	60	116	8.4
MIL-H-83306 7 days	60	101	6.9
MIL-H-83306 28 days	60	89	6.0
MIL-H-83306 2 days @ 150°F	60	Delaminated	Delaminated
Weathering 600 - 20 min. Sunlight/Rain Cycles	60	106	7.5
Salt Water Environment 200 hours	60	96	7.2
Humidity 7 days	60	109	9.2
28 days	60	84	8.8
2 days @ 180°F	60	101	8.2

Laminate: 20 plies of P-1700/A-S with .016" clad aluminum skins



ALUMINUM-CLAD P-1700/A-S

Figure 32: Flexure Specimens – Typical Fractures



ALUMINUM CLAD P-1700/A-S

Figure 33: Flexure Specimens – Typical Fractures

Table 7: METAL CLAD TO METAL CLAD LAP SHEAR TEST DATA

Cladding On 20 Ply P-1700/AS V_f = 61%	Surface Treatment	Lap Shear Strength (psi)		
		-65^{0}F	RT	300^{0}F
Steel, 5-mil	HNO_3 . HF/epoxy *	5460	4800	3200
Steel, 5-mil	Anodize/epoxy *	5700	5320	3400
Aluminum .011	$NA_2CR_2O_7$. H_2SO_4/epoxy *	5100	4470	3120
.011	Phosphoric acid anodize/ epoxy *	5300	4620	3190

* (BR-127) American Cyanamid Company

Figure 34: Aluminum Clad Laminate Before Thermal Shock (50X)

Figure 35: Aluminum Clad Laminate After Thermal Shock (50X)

Figure 36: Aluminum Clad Laminate Before Heat Aging (50X)

Figure 37: Aluminum Clad Laminate After Heat Aging (50X)

Figure 38: Steel Clad Laminate Before Heat Aging (50X)

Figure 39: Steel Clad Laminate After Heat Aging (50X)

Figure 40: Steel Clad Laminate Before Thermal Shock (50X)

Figure 41: Steel Clad Laminate After Thermal Shock (50X)

2.3.3 Thermoset Clad Hybridizing

Epoxy cladding was evaluated as a means to provide exposure protection from various fluids to P-1700 polysulfone thermoplastic laminates. Epoxy polymers are not significantly degraded by exposure to ketones and chlorinated solvents, and it was felt that cladding would offer protection from these and other solvents and fluids.

Fiberite's 1034 prepreg material was selected as the graphite/epoxy system for cladding the P-1700 polysulfone thermoplastic laminates. The 1034 material is Fiberite's 934 epoxy resin on Thornel T-300 unidirectional fibers. This system was selected because of its wide use in advanced composite materials and its capability to withstand the short high temperatures used in thermoforming the thermoplastic composites.

Fabrication of the clad composites was accomplished by laying up and molding a selected number of plies of P-1700/A-S graphite prepreg in a small laboratory press and autoclave curing the 1034 epoxy prepreg on the molded thermoplastic laminate. Different configurations of the epoxy-clad laminates were fabricated for different tests. Some laminates were clad one side only while others were clad on both sides.

A laminate consisting of 12 plies P-1700/A-S and 12 plies 1034 prepregs was tested in interlaminar shear per FTM-STD-406, Method 1042. This laminate was clad on one side to place the shear plane at the interface of the two materials.

The failures were at the interface of the two composite materials with some of the fibers being broken in the epoxy side. Figure 42 is a typical failure of the interlaminar shear specimens. Shear test results are reported in Table 8. The thick specimen configuration eliminated most of the bending which usually leads to peel rather than shear.

An epoxy-clad laminate was fabricated with four plies of epoxy on each side of a 12-ply polysulfone laminate. The laminate was evaluated in tension and flexure. The latter tests were conducted in a stressed and unstressed condition.

Tension specimens were fabricated using the straight-sided specimens with end

TABLE 8 EPOXY CLAD LAMINATE PROPERTY DATA

Laminate Configuration (epoxy, P-1700, epoxy)	Test Temperature °F	Flexure Strength 10^3 psi	Flexure Mod 10^6 psi	Tensile Strength 10^3 psi	Tensile Mod 10^6 psi	ILS Strength psi
12, 12, 0	RT	--	--	--	--	4185
	300	--	--	--	--	2045
6, 14, 0	RT	181	13.9	--	--	--
	* RT	140	14.1			
	300	129	11.4	--	--	--
	300	198	11.1			
4, 16, 0	RT	175	13.4	--	--	--
	* RT	133	13.1			
	300	130	11.1	--	--	--
	* 300	103	11.2			
4, 12, 4	RT	189	14.8	122.7	12.4	--
	300	133	12.3	88.7	11.9	--
14, 8, 0	RT	145	8.6	--	--	--
	* RT	120	8.7	--	--	--

* Specimens tested with epoxy side up - delamination of the interface during testing.

-- Not tested

tabs. Tests were run at room temperature and 300°F. Test data are presented in Table 8. The specimens failed with a sort of brooming effect, with the fibers breaking throughout the laminate. Figure 43 is a typical failed epoxy clad tensile specimen.

The flexure specimens were loaded to 60% of their ultimate strength and exposed to different environments. Test results were reported earlier under Section 2.2, Stress Cracking Study. Table 9 is a summary of these results.

Three epoxy-clad laminate configurations were fabricated and tested in flexure. Two of the configurations were clad on one side. One laminate consisted of 14 plies thermoplastic/6 plies epoxy and the other, 16 plies thermoplastic and 4 plies of epoxy. Specimens were tested with the epoxy side up and epoxy side down. The test results were dependent upon whether the epoxy side of the laminate was in tension or compression.

Test results are also reported in Table 8. Specimens tested with the epoxy side in compression caused delamination at the interface of the two resins without a tension or compression failure. Specimens tested with the thermoplastic side in compression did not delaminate during testing with the epoxy layers failing in tension. This phenomenon indicates that the laminate configuration is governed by strain in the epoxy layers rather than total laminate stress. The thermoplastic composites in compression have a greater strain to failure than the epoxy composites in tension, causing the fracture to initiate in the epoxy plies.

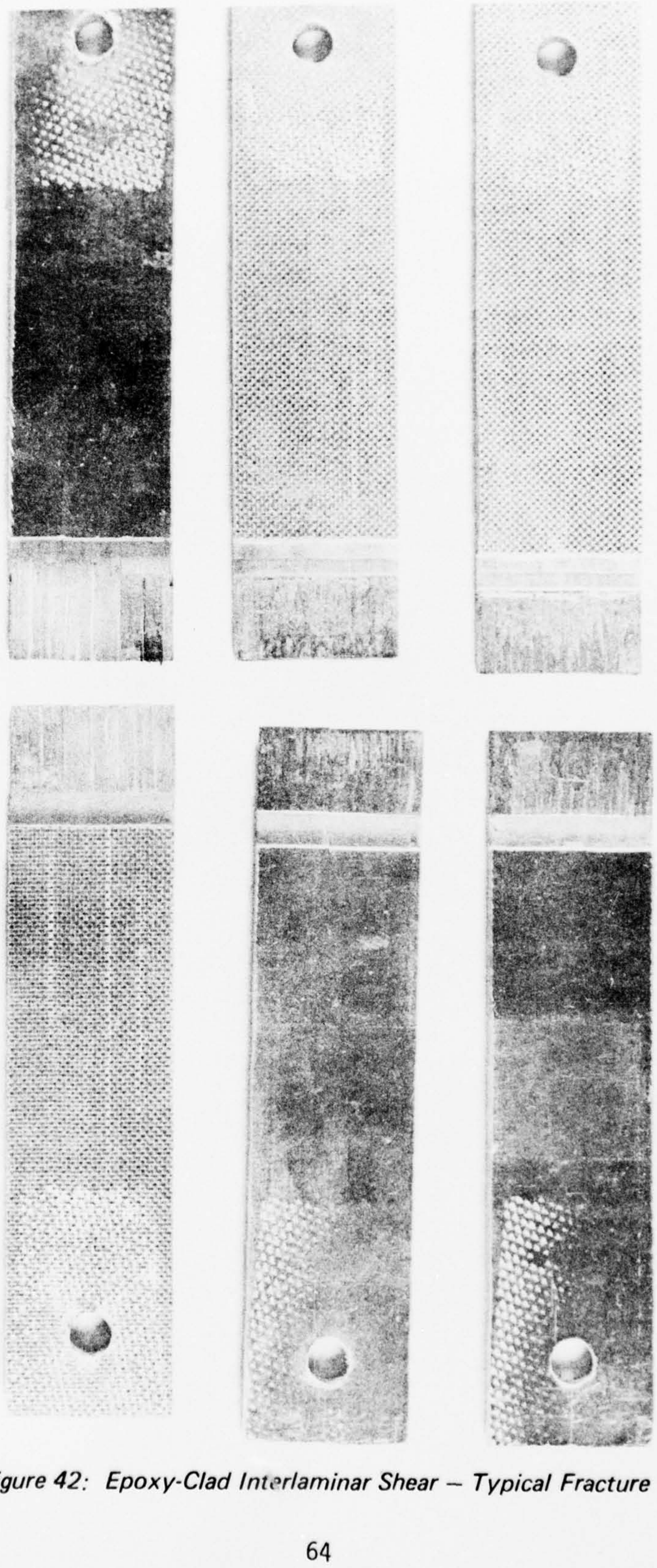


Figure 42: Epoxy-Clad Interlaminar Shear – Typical Fracture

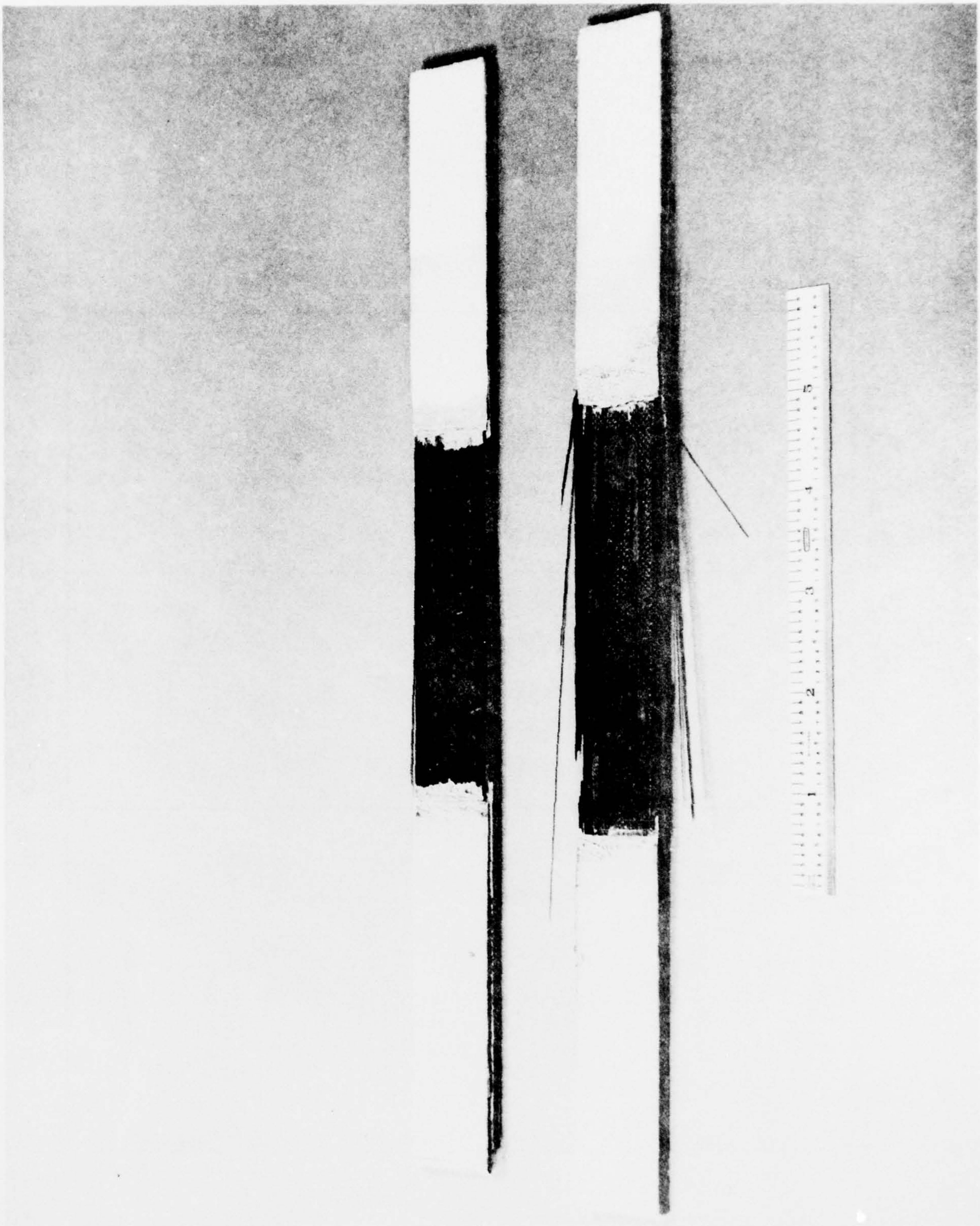


Figure 43: Epoxy-Clad Tensile Specimen – Typical Fractures

TABLE 9 EPOXY CLAD STRESSED ENVIRONMENTAL EXPOSURE DATA

Exposure	Stress Level % of Ultimate	Environmental Flexural Data	
		Stress x 10^3 psi	Modulus x 10^6 psi
Control	0	189	15.1
JP-4 28 days	60	175	15.4
MIL-H-5606 28 days	60	166	15.0
MIL-L-7808 28 days	60	165	15.2
MIL-H-83306 7 days	60	147	11.1
MIL-H-83306 28 days	60	110	4.9
MIL-H-83306 2 days at 150^0F	60	--	--
Weathering 600 - 20 min. Sunlight/Rain Cycles	60	160	14.8
Salt Water Environment 200 hours	60	129	13.5
Humidity 7 days	60	176	15.4
28 days		171	15.0
2 days @ 180^0F		172	14.9

* Laminate configuration: 4 plies 1034, 12 plies P-1700/A-S, 4 plies 1034

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 CONCLUSIONS

No structural advantages or improved adhesion of polysulfone resins are achieved by placing an epoxy sizing of Fiberite's 934 resin on Hercules Type A-S graphite fibers.

The adhesion of the new thermoplastic polysulfone resins (PKXA .24 and PKXA .41) to the normal surface finishes on Hercules Type A-S and HM-S graphite fibers is equivalent to the adhesion of P-1700 polysulfone or better.

The mechanical properties of PKXA composites are lower than P-1700 properties but PKXA composites are more solvent resistant in methylene chloride, ketones and other chlorinated solvents that readily attack P-1700 composites. With added experience in both prepreg and composite fabrication, the properties of PKXA composites should improve significantly.

P-1700/A-S composites exhibited minor decreases in flexural strength with definite increases in flexural moduli when exposed to weathering under stress conditions of 50% or less. Epoxy-clad P-1700/A-S specimens show a loss (< 7%) in flexural strength and no appreciable loss in moduli after exposure to 600 sunlight/rain cycles. The flexural strength of aluminum-clad P-1700/A-S laminates is not affected by the weathering environment. A minor decrease in modulus was noted.

A decrease in flexural strength of 2.9 - 18.8% was noted for specimens that were stressed and exposed to humidity environments.

Specimens soaked in water at 150°F for 2 days showed decreases of 2.3 - 21.8% in flexural strength with 10.8 - 27% increases in flexural moduli.

Flexural properties of P-1700/A-S clad and non-clad laminates were reduced 2.4 - 31.7% when exposed to salt water environment under stress conditions.

No significant flexural property loss was noted for clad and non-clad specimens exposed to JP-4 (jet fuel) under stress conditions.

Flexural stresses are reduced (5%) when P-1700/A-S laminates are exposed to MIL-L-7808 (aircraft lubricant) under 50% stress loads.

Exposure of stressed flexural specimens to MIL-H-5606 hydraulic fluid causes no significant property losses.

MIL-H-83306 hydraulic fluid has a detrimental effect upon P-1700/A-S, clad, and non-clad composites under stress conditions. Metal-clad P-1700/A-S composites need edge protection when exposed to MIL-H-83306 fluids. Metal-clad composites will delaminate in hot (150°F) MIL-H-83306 hydraulic fluid under stress conditions.

No stress cracking was detected in any of the polysulfone or PKXA composites which were exposed to the various environments. Even the specimens which showed some property degradations did not have any evidence of stress cracking.

This study revealed no structural disadvantages in hybridizing the resins or carriers in thermoplastic composites. Hybridizing appears to be a viable way of tailoring composites to meet the needs and desired of designers.

3.2 RECOMMENDATIONS

It is recommended that further processing and evaluation and forming studies of the PKXA resins be conducted on glass, Kevlar and graphite carriers.

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

This program was a three-phase effort. Phase I was devoted to evaluating compatible fiber surface finishes for thermoplastic matrices. Three polysulfone resins (P-1700, PKXA.41 and PKXA.24) from Union Carbide were evaluated. Under Phase II graphite reinforced thermoplastic composites were evaluated for stress cracking in various service environments. Methods were also studied for providing a protective metallic or polymeric clad surface on the thermoplastic laminates. Specimens were exposed to various

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aircraft fluids and then tested for residual properties. Phase III of the program was devoted to hybridizing graphite reinforced thermoplastic laminates with Kevlar and glass reinforcements. The structural potential of the hybrid systems was assessed before and after exposure to critical service environments. Three tests showed no deficiencies that would preclude the use of hybrids on structural components.

Graphite reinforced polysulfone composites showed no tendency to stress crack or craze in a stressed or unstressed condition when exposed to jet fuel, weathering, salt water, hydraulic fluid or synthetic lubricant. However, some environmental exposures did reduce the mechanical properties of the laminate.