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REPORT NO. HA-2559

MONOLAYER BORON-ALUMINUM
COMPACTED SHEET MATERIAL

By

E. V. Sumner

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Prepared under Contract No. NAS8-27626 by

MARTIN MARIETTA ALUMINUM INC.
Research and Development Division
Torrance, California 90509

For

National Aeronautics and Space Administration
GEORGE C. MARSHALL SPACE FLIGHT CENTER

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Martin Marietta Aluminum
Torrance, California

ABSTRACT

This report describes the manufacturing techniques, basic materials used, and equipment required to produce monolayer boron-aluminum composites. Tentative materials and process specifications are included. The report discusses improvements in bonding and filament spacing obtained through use of brazing powder in the fugitive binder.

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Huntsville, Alabama

January 1973

FOREWORD

This is the final report of a program conducted to increase reliability through investigation of the problem areas of boron-aluminum monolayer metal matrix composites. The work was performed by Martin Marietta Aluminum Inc. for the National Aeronautics and Space Administration, George C. Marshall Space Flight Center, Huntsville, Alabama. Mr. Ron Nichols was the NASA contracting officer's representative for the program.

The project was conducted in the Research and Development Department of Martin Marietta Aluminum Inc. and was managed by R&D Project Engineer Mr. E. V. Sumner. This report carried the contractor's identification of HA-2559.

The author wishes to acknowledge the significant assistance offered throughout the program by the following Martin Marietta Aluminum personnel: Messrs. C. N. Doyle and L. Huss, composite fabrication; Mr. David Farnham, sample testing; Mr. D. Q. Cole, quality control, and Mr. A. J. Goulding, reporting.

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SECTION I. SUMMARY

The effect of raw material and processing factors on the production of monolayer boron-aluminum composites were investigated. Thirty-two experimental pieces were produced and tested. Twenty-six additional pieces were produced for evaluation by McDonnell Douglas Corporation, East (MDC) and Marshall Space Flight Center (MSFC), Huntsville.

A simple system was developed for measuring efficiency of the compositing operation. A good correlation was found between the strength of filament samples obtained from the end of each run of filament and bundle strength of filament from within the run.

Solvent cleaning of the foil and filament was the most satisfactory method for pretreating the raw materials. The eddy current method of measuring foil thickness was found to be satisfactory. The use of brazing powder in the styrene fugitive binder was found to be helpful in improving bonding and maintaining filament alignment.

The customary system of units was used for the principal measurements and calculations in this report.

SECTION II. INTRODUCTION

The boron-aluminum metal matrix composite system has received the most attention in the area of metal matrix composites and has been selected as one of the composite systems by "Project Composite Recast" to be developed for system application. The major barriers confronting increased usage of composite material are reliability and cost. This reported program is concerned primarily with increasing reliability through investigation of the problem areas of boron-aluminum monolayer metal matrix composites.

The primary problem areas which have become evident are concerned with those factors which effect voids and poor bonding, filament spacing and scatter in mechanical properties. These areas are inter-related with the raw material used, processing procedures, bonding techniques and quality assurance procedures.

Discussion of the work performed under the contract reported herein is divided into five areas: (1) the approach used; (2) raw material; (3) procedure; (4) results, and (5) conclusions and recommendations.

Appendixes A, B, and C contain additional information on measurement of foil thickness by use of eddy currents; examination of the factors affecting transverse properties of boron-aluminum composite, and preliminary specifications covering the processing of boron-aluminum composites.

SECTION III. DESCRIPTION

A. TECHNICAL DISCUSSION OF APPROACH

1. General. The difficulties that have been encountered in the fabrication of monolayer boron-aluminum composite material may be divided into four areas: poor bonding; non-uniform filament spacing; occurrence of voids, and scatter in the physical property data. These problems are affected by the fabrication sequence and environment, raw material qualification and pre-treatment, composite bonding technique, and testing and quality assurance procedures.

The factors which affect the problem areas are given in outline form in subparagraph 2, and the program to improve the quality and consistency is presented in subparagraph 3.

2. Factors Affecting Problem Areas:

a. Scatter in Data

- (1) Bundle strength of filament
- (2) Transverse strength of filament
- (3) Filament pre-treatment
- (4) Bonding aluminum to aluminum

b. Voids

- (1) Uneven filament spacing
- (2) Foil thickness variation
- (3) Uneven temperature
- (4) Uneven pressure

c. Filament Spacing

- (1) Filament twist
- (2) Breaks in filament during drum winding
- (3) Application of plastic

d. Poor Bonding Aluminum to Aluminum

- (1) Cleaning of aluminum and filament
- (2) Filament spacing
- (3) Uneven foil thickness
- (4) Uneven bonding temperature and pressure
- (5) Facilities in clean area and in sequence

3. Program to Improve Quality and Consistency

a. Environmental Control

- (1) Cover and put positive air pressure in composite room
- (2) Move big winding drum lathe into composite room
- (3) Move foil cleaning line to end of composite room
- (4) Install vacuum exhaust on wire brush machine
- (5) Set up bonding jig cleaning procedure

b. Quality Control

- (1) Install resistance measuring device
- (2) Develop Quality Control monitoring procedures

c. Procedure Evaluation

(1) Test Raw Material

(a) Foil - 1100 aluminum

Chemical analysis

(b) Filament - .42 and .102-mm (.0046 and .004-in.)

- Bundle test for each wrap
- Manufacturer's test data

(2) Evaluation of Processing Factors

- (a) All pieces 304.8 x 609.6 mm (12 x 24 in.) monolayer, 1100 aluminum, .142 mm (.0056-in.) diameter boron
- (b) Evaluation of cleaning procedures
- (c) Evaluation of factors affecting spacing
- (d) Evaluation of processing parameters

d. Test Program for 32 Pieces

- (1) Resistance of foil prior to assembly
- (2) X-ray
- (3) C-scan
- (4) Short transverse UTS
- (5) Longitudinal UTS
- (6) Transverse UTS
- (7) Microscopic examination

e. Correlate Test Results

B. MATERIAL

1. General. The material used in this program consisted of 1100 aluminum foil furnished by Martin Marietta Aluminum and .908 kg (two pounds) of .10 mm (.004-in.) and 3.62 kg (8 pounds) of .14 mm (.0056-in.) diameter boron furnished by Avco. A small amount of 6061 foil and 718 brazing foil furnished by the American Lamotite Corp. (a distributor for Alcoa) was used. Table 1 lists an analysis of the foil.

TABLE 1. Analysis of Foil Material

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Ti	Ph	Zr
1100	.08	.43	.16	.0	.01	.0	.0	.01	.002	.0	.0
6061	.72	.40	.28	.09	1.06	.19	.0	.09	.06	.0	.0
718	9.35	.31	.02	.03	.0	.0	.0	.01	.002	.0	.0

C. WORK PERFORMED

1. Procedure. The procedure followed will be discussed in five areas: qualification of raw material; preparation of raw material; diffusion bonding; quality control, and testing.

2. Qualification of Raw Material. The raw material used in the experimental work was 1100 aluminum foil and .14-mm (.0056-in.) boron filament. The aluminum foil was measured for thickness and found to be uniformly .05-mm (.002-in.) thick. The 3.62 kg (8 lb) of boron filament was segregated into lots by average strength with each lot consisting of four spools. The coefficient of variation (cv) was calculated for each lot by three methods: (1) using the average strength of each spool element for the lot; (2) averaging the individual spool cv in each lot, and (3) averaging the filament test cv for each lot. The results, by lot, are listed in Table 2.

3. Preparation of Raw Material. Raw material preparation is discussed under the following subsections: cleaning; filament winding; application of styrene, and layup for bonding.

a. Cleaning Aluminum Foil and Boron Filament. Two methods were used with the boron filament and these were either as-received or cleaned in methanol. The cleaning of filament with methanol was accomplished by passing the filament through an S-shaped glass tube (containing

TABLE 2. Summary of .14mm (.0056-in.) Boron Filament
Coefficient of Variation

Lot	(1) Lot by Spool Element Strength	(2) Spool Average	(3) Filament Strength
1	11.3	12.2	11.0
2	6.0	7.0	9.8
3	3.0	3.8	9.7
4	5.9	6.4	11.0
5	6.5	7.4	10.5
6	10.6	11.7	10.7
7	7.6	8.0	10.2
8	3.1	2.1	11.3
9	1.5	---	11.8
Total	55.5	58.6	96.0
Average	6.2	7.3	10.7

Note: Columns 1 and 2 show a lower cv because of averaging effects.

circulating methanol) during the winding operation. The methanol was supplied from a reservoir by a Monostal Tube Pump (No. 54846-205) using 6.35mm (1/4-in.) ID Tygon tubing.

The aluminum foil was cleaned by the following procedures:

- MEK
- MEK plus wire brushing
- Degrease and deoxidize
- Degrease, deoxidize and wire brush
- Degrease, deoxidize and zincate
- Degrease, etch, deoxidize and wire brush
- As-received
- Degrease, etch, deoxidize
- Methanol

The chemical cleaning was accomplished in 1219.2 x 1524 x 25.4mm (4 x 5 x 1-ft) cleaning tanks as shown in Figure 1; solvent cleaning was performed in a shallow aluminum pan, and wire brushing was carried out using a stainless steel custom rotary wire brush 711.2mm (28 in.) long by 177.8 mm (7 in.) in diameter.

b. Filament Winding. The sequence of steps used to produce the boron filament mats for composite fabrication is as follows:

(1) Attach cleaned aluminum foil to 1447.8 mm (57 in.) dia. drum in filament winding lathe using scotch tape to hold the foil in place. See Figure 2.

(2) Set feed on lathe at .1676 mm (.0066-in.)

(3) Attach filament to drum at cut line with scotch tape.

(4) Wind to width called for on print.

c. Application of Styrene. Upon completion of winding the boron filament, the end is fixed to the cut line with scotch tape. With the drum turning at 36 surface meters per minute (120 surface feet per minute), a solution of Dow Chemical 685 Styrene diluted in MEK is sprayed on the wound filaments to hold them in position on the aluminum foil. After the styrene has dried for 20 minutes, the mat is cut from the drum at the cut line and placed between two pieces of clean paper for storage prior to assembly in the bonding package.

d. Lay-up for Bonding. The elements contained in the diffusion bonding container consist of: the mild steel container; stainless steel glide sheets; aluminum bumper sheets; aluminum foil, and boron filament mats held together with styrene binder. All of the previously prepared elements are positioned in the lay-up area and assembled in the following sequence.

(1) Spray-coat inside surface of package covers with graphite (Aquadag No. 8)

(2) Pre-place spacers in bottom cover

(3) Lay first outside bumper sheet

(4) Lay first outside graphite-coated glide sheet

(5) Lay first sheet of prepared aluminum foil (cut to size)

(6) Lay sheet of filament mat (cut to size)



FIGURE 1. ALUMINUM FOIL AND BORON FILAMENT CHEMICAL CLEANING TANKS

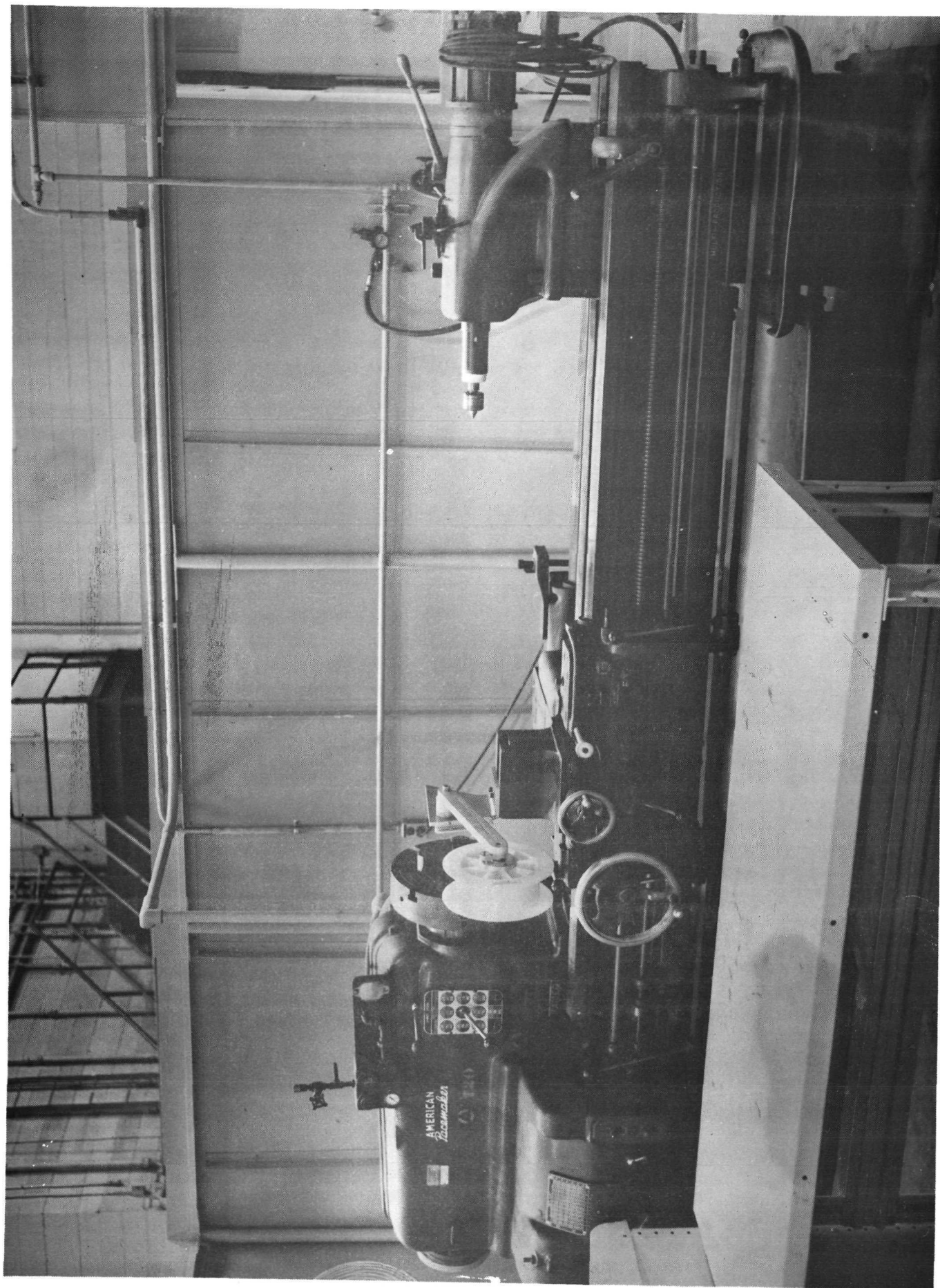


FIGURE 2. FILAMENT LATHE WITH WINDING DRUM

- (7) Lay second sheet of prepared aluminum foil (cut to size)
- (8) Repeat steps (6) and (7) for required number of layers of composite.
- (9) Lay second sheet of coated glide sheet
- (10) Lay second sheet of bumper plate
- (11) Repeat steps (4) through (9) for required number of composite sheets per package
- (12) Lay second outside bumper sheet
- (13) Position top cover
- (14) Clamp package for welding
- (15) Weld and leak-test package

4. Diffusion Bonding. The diffusion bonding process was carried out on a 1500-ton hydraulic press. The hot dies were electrically heated with 15.9 mm (5/8-in.) dia. cartridge heaters controlled with Alnor high-low controllers. The die heaters were calculated to furnish 12,903.25 watts/surface sq. cm (200 watts/surface sq. in.) at full output. Vacuum was maintained with two mechanical pumps and one diffusion pump. Two stainless steel dry-ice and acetone traps were used to condense the styrene evacuated from the package. Time was varied from 10 minutes to 1-1/2 hours; temperature from 481°C to 504°C (900°F to 940°F), and pressure was held constant at 420 kg/sq. cm (6000 psi). The sequence of steps in the process are as follows:

- a. Assemble package in press and turn on vacuum pumps
- b. Set controls for 425°C (800°F) and turn on heat
- c. Out-gas package for minimum of 4 hours or when vacuum drops (which indicates styrene is removed from package)
- d. Raise temperature to 10°C (50°F) below print callout
- e. Gradually apply pressure to 420 kg/sq. cm (6000 psi)
- f. Adjust temperature to print requirement
- g. Press for required time
- h. Release pressure, and cool

5. Quality Control. Each piece to be produced was issued to the shop on a composite engineering drawing specifying the requirements for the piece. See Figure 3. Each operation in the fabrication of the piece was recorded on a process control record which followed each piece through processing. See Figure 4. Preliminary quality control specifications for each major component or process are contained in Appendix C.

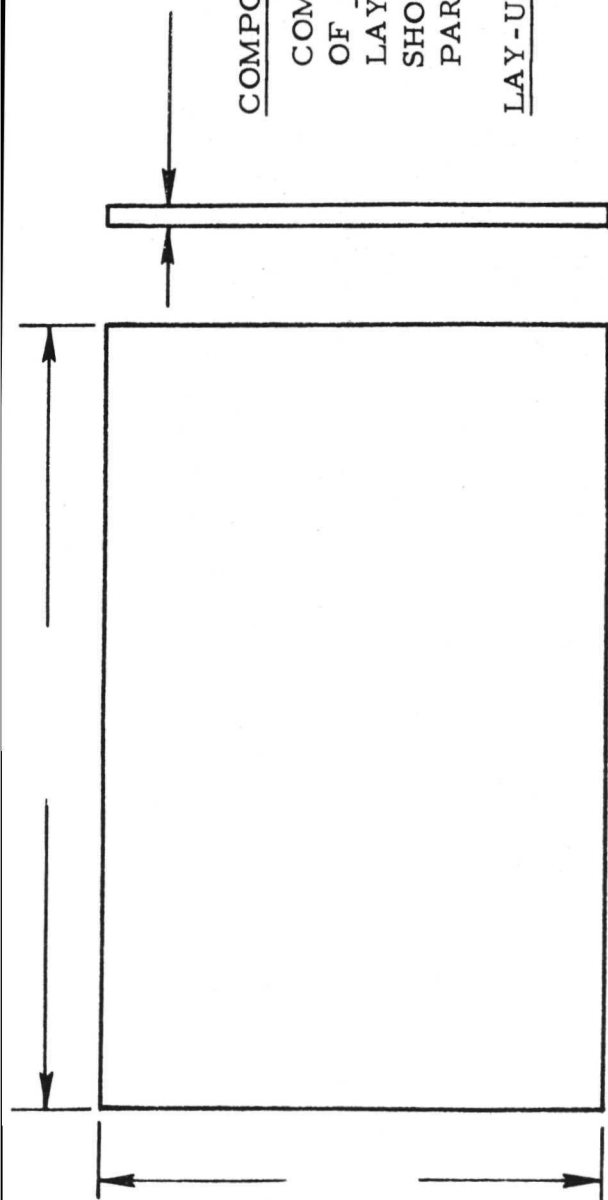
6. Testing. The following tests were conducted on the material produced: (1) foil surface resistance; (2) filament bundle test; (3) composite ultimate tensile strength; (4) composite transverse tensile strength; (5) composite short transverse strength; (6) X-ray; (7) C-scan, and (8) metallographic examination. The test specimen is shown in Figure 5.

a. Foil Surface Resistance Test. The electrical contact resistance of the surface films was measured using the procedure reported by Dr. W. G. Zelley.⁽¹⁾ The procedure (obtained from private communication with Dr. Zelley) is: (1) placing the piece of foil between two gold-plated half-inch round flat faced copper contact points; (2) applying a 454 kg (1000-pound) load to the contacts, and (3) measuring the resistance with a Leeds & Northrup 4287 Kelvin Bridge. See Figure 6. In conducting the test, a reading was taken with no sample between the contacts to obtain the resistance of the circuit. The sample was then inserted between the contacts, the load applied, and the resistance read. The difference between the two readings was taken as the sample resistance. The average of three readings was reported as the sample resistance.

b. Filament Bundle Test. Filament bundle tests were conducted on specimens 12.7 mm (1/2-in.) wide by 177.8 mm (7 in.) long taken from the end of each wrap. Rubber tabs 63.5 to 76.2 mm (2 1/2 to 3 in.) long were epoxied to the ends of the sample for gripping and to give a testing gage length of 25.4 mm (1-inch). During the filament wrapping operation, a record was kept of the length of filament used from each spool and its position in the wrap. Consequently, the bundle strength obtained could be directly compared with the manufacturer's test data. The number of filaments in each sample and their average diameter were recorded, and the product was divided into the load obtained in testing to determine the bundle strength.

Calculated bundle strengths were obtained by determining the average strength and cv from ten individual filament tests representing a spliced length of filament on the spool. Deriving the Weibull m parameter from the relation $cv = \frac{1.2}{m}$ when cv is the coefficient of variation and m is the Weibull parameter.⁽²⁾ The bundle strength was calculated

from the following relation:
$$\frac{\sigma_b}{\sigma} = \frac{(me)^{-1/m}}{T \left(\frac{m+1}{m} \right)}$$
⁽³⁾



COMPOSITE DESIGN:

COMPOSITE TO CONTAIN _____ LAYERS
OF _____ THICK _____ SHEET AND _____
LAYERS OF FILAMENT SPACED AS _____
SHOWN IN VIEW A WITH WIRES
PARALLEL TO THE _____ DIMENSION

LAY-UP, PROCESSING AND INSPECTION

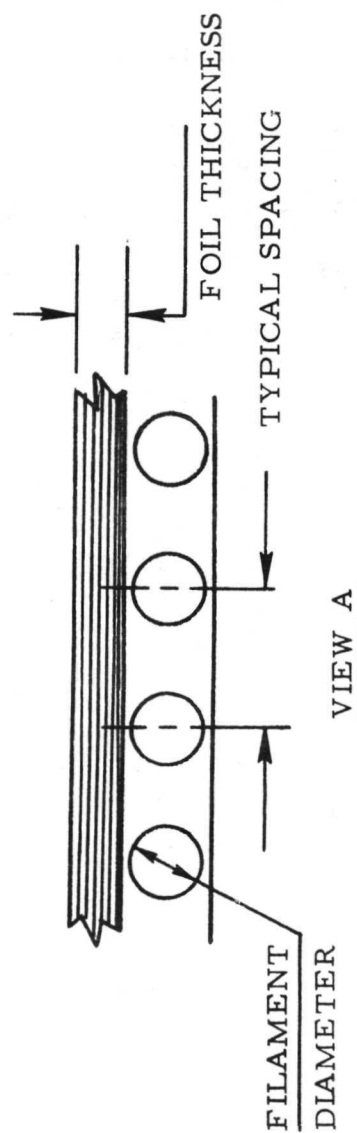


FIGURE 3. COMPOSITE ENGINEERING SPECIFICATION DRAWING

Customer _____		Job No. _____		Sheet No. _____	
Aluminum _____		Filament _____		Type _____	
				Size _____	
Operation	Date	Time			
Cut Foil			For Thickness _____		
Clean			Method _____		
Wire Brush					
Test Resistance					
Wrap			Drum Size _____ Spool No. _____ Spool Portion _____		
Lay Up			Package Size _____ Cushion Thickness _____ Glide Sheet Thickness _____		
Foil Resistance					
Weld					
Test			Vacuum Readings _____		
Press			Pressure _____ Vacuum _____ Temperatures _____ Method _____		
Open					
Trim					
Cut Samples			Sample Size _____		
X-ray					
C-scan					

FIGURE 4. PROCESS CONTROL RECORD FORM

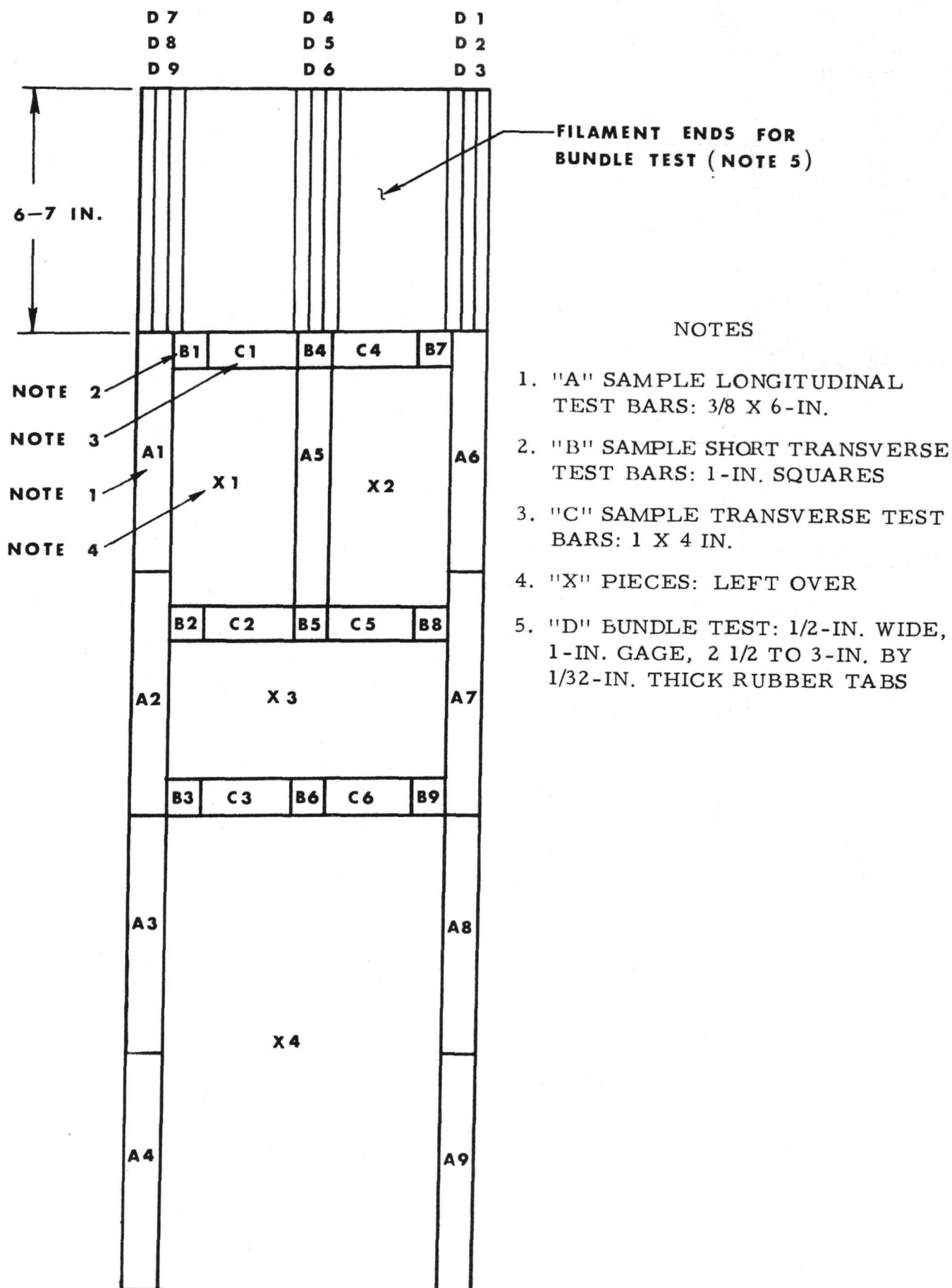


FIGURE 5. TEST SPECIMEN LAYOUT

c. Composite Ultimate Tensile Strength. Samples for ultimate tensile strength were straight sided, 9.5 mm (3/8-in.) wide by 152.4 mm (6 in.) long, with 63.5 mm (2.5 in.) long, .79-mm (1/32-in.) thick rubber tabs epoxied to the specimens with Chemlock 305 adhesive. Originally, the specimens were sheared to size; however, it was found that more consistent results were obtained if samples were sheared oversize (12.7 mm wide) (1/2-in. wide) and then eloxed to finished size.

	<u>Sheared Edge</u>	<u>Eloxed Edge</u>
Av ₂₄	11.46 kg/sq. cm (163 psi)	12.30 kg/sq. cm (175 psi)
Coefficient of Variation	9.7	8.1

d. Composite Transverse Strength. Samples for transverse tensile strength were 12.7 mm (1/2-in.) wide by 101.6 mm (4 in.) long, straight sided with eloxed edges, and .79 mm (1/32-in.) thick rubber tabs glued to give a 25.4 mm (1-in.) gage length. Initially, samples were monolayer; however, because erratic results were frequent, it was felt more meaningful data would be obtained from six-layer samples. In only one case out of twelve was the average strength of six individual samples greater than a six-layer sample. The six-layer samples were prepared by taking six oversized monolayer pieces and diffusion bonding them together at 481°C (900°F) for one hour at 420 kg/sq. cm (3 tons per sq. in.).

During studies for his master's degree at the University of Arizona, Mr. Robert C. Kietzman examined the factors effecting the transverse properties of boron aluminum composite. His thesis is included as Appendix B.

e. Short Transverse Test. The short transverse test was conducted by adhesive-bonding a flat-faced, 25.4-mm (1-in.) round dumbbell tab to each side of a 25.4-mm (1-in.) round sample. The sample was obtained by shearing the piece of monolayer composite foil in a punch and die set. The sample was then pulled in tension, and the load and type of failure was recorded, i.e., bond failure or adhesive failure. Three types of adhesive were used: Eastman 910, Chemlock 305, and Whittaker 3725/7148. The Whittaker adhesive afforded the best results although a large percentage of failures were in the adhesives.

f. X-ray. The procedure used for X-ray of the composite samples is as follows: the composite sample is taped to a piece of .79-mm (1/32-in.) thick aluminum sheet slightly larger than the sample. The piece is then

X-rayed by the X-ray Products Co. placing the composite side next to the film. The equipment and parameters used are listed below:

- Ceifert 30-150 KVCP Be window equipment
- 50 kv
- 8 ma
- 60 sec
- 914.4mm (36-in.) focal distance
- .7mm (.027-in.) focal spot
- Eastman R, single emulsion film

g. C-scan. C-scans were conducted by placing the monolayer composite sample in a picture frame-type fixture. The outer 12.7 mm (half-inch) of the sample was held by the fixture. The fixture was used to hold the composite foil rigid during the C-scan operation. Equipment and parameters used for C-scan testing are presented in the following list:

- Immerscope 725 Through Transmission
- Rej. - 2
- Display - Filt
- PFR - 2500
- Mode - Delay Sinc.
- Attenuator - 4 db
- STC - 0
- Freq. - 10 mhz
- Sens. - 2.5
- Damp. - 10
- Recording level - 160
- Transmitter - 5 mc
- Receiver - 2.25 mc

SECTION IV. RESULTS

A. INTRODUCTION

A total of 58 pieces of monolayer boron-aluminum composites were produced. Of the 58 pieces, 32 were experimental and 26 were demonstration pieces shipped to NASA, Huntsville, and MDC East. All pieces were 25.4 x 50.8 mm (1 x 2 ft) in size except two demonstration pieces which were nominally 50.8 x 152.4 mm (2 x 6 ft). Samples for testing were taken from each piece as previously shown in Figure 5; except, where poor bonding precluded taking samples.

The results have been divided into the following three groups for discussion: raw material; monolayer composites, and quality control.

B. RAW MATERIAL

The results discussed in this section concern: foil resistance, filament strength, and filament bundle strength.

1. Foil Resistance. Foil resistance measurements were taken on orientation samples listed in Table 3 and production samples shown in Figure 7.

In general, Table 3 indicates that those systems in which water is not involved are better than those containing water and that the Zincote coating provided the lowest foil contact resistance. Figure 7 shows a summarization of the foil resistance of 32 test samples plotted against the UTS efficiency (defined in subsection III. C. 6). It is not difficult to visualize a regression line running down and to the right which is what would be expected; however, the correlation would not be high. The indications are that it is desirable to keep the surface contact resistance below 200 micro ohms.

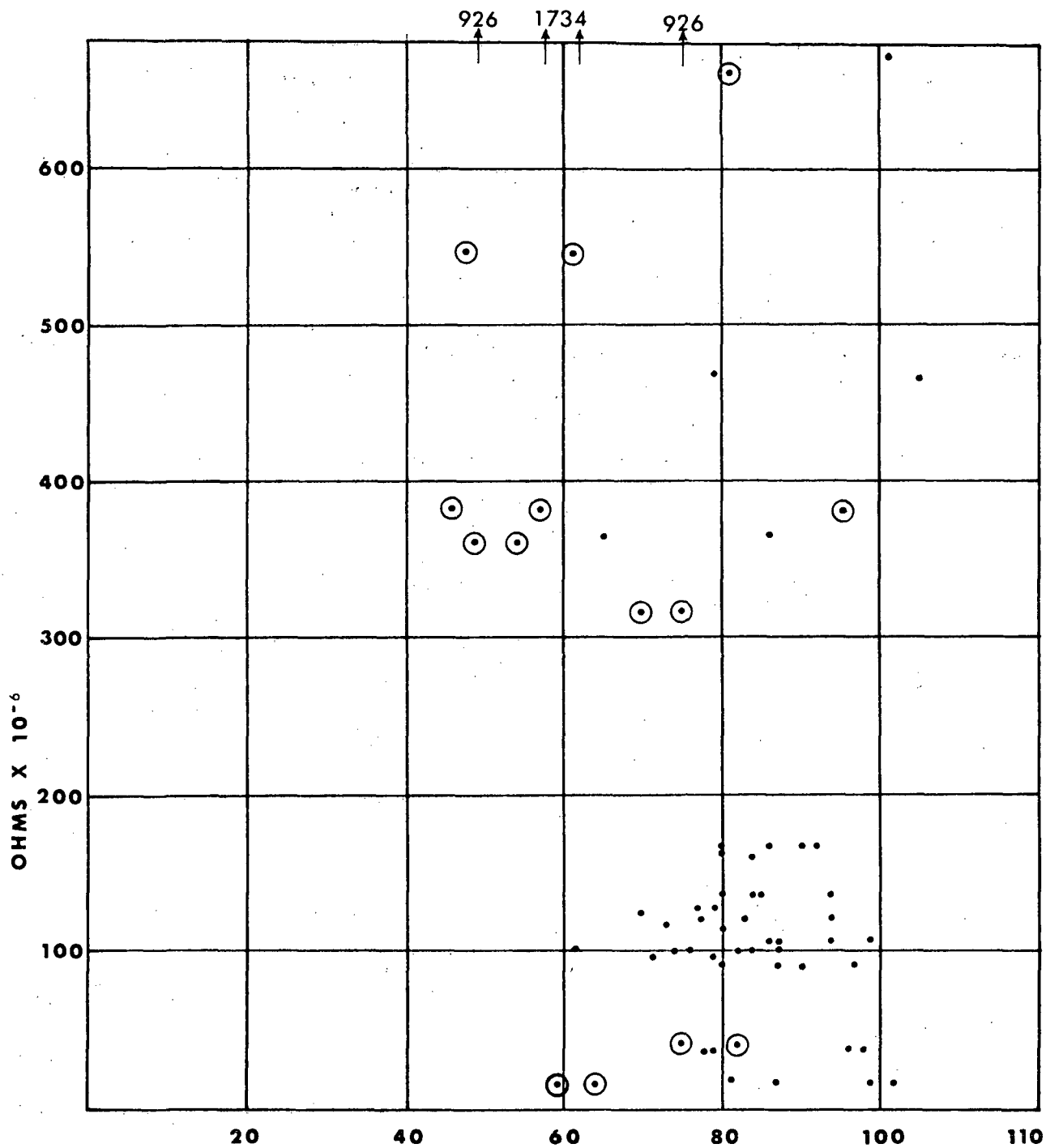
2. Filament Test Data. The filament manufacturer tests 10 filament samples from the end of each run and records the load and diameter. The average load is divided by the area to obtain the average strength of the filament for that run. The runs in a spool are then averaged to obtain the spool average UTS. The strength distribution for the 132 runs is shown in Figure 8.

3. Filament Bundle Test and Calculated Bundle Strength. Samples for the filament bundle test were obtained from the end of each wrap and consequently represented a sample of filament taken every 1447.8 mm

TABLE 3. Foil Resistance (two sides)
Average of Three Readings

Sample	Condition	Resistance (ohms x 10 ⁻⁷)		
		Initial	124 hrs.	1 week
A	Degreased (MEK)	49	141	266
B	Degreased + Wire-brushed (MEK)	29	105	143
C	Etch + Rinse (NaOH)	23,118	41,282	145,985
D	Etch + Deoxidize (NaOH) (HNO ₃ CrO ₃)	235	375	538
E	Acid Etch (Avcil)	68	687	1528
F	Etch + Deoxidize + Wire-brush + Lacquer*	37	94	153
G	10% Hexionic Acid in H ₂ O	1927	2839	4811
H	Zincate + Water Rinse	20	20	31
I	10% DMSO in MEK	45,977	44,032	85,288

*Lacquer removed with solvent prior to test.



LEGEND

• NORMAL PACKAGE

⊙ CONTINUATION PACKAGE

FIGURE 7. FOIL RESISTANCE-VS-PERCENT

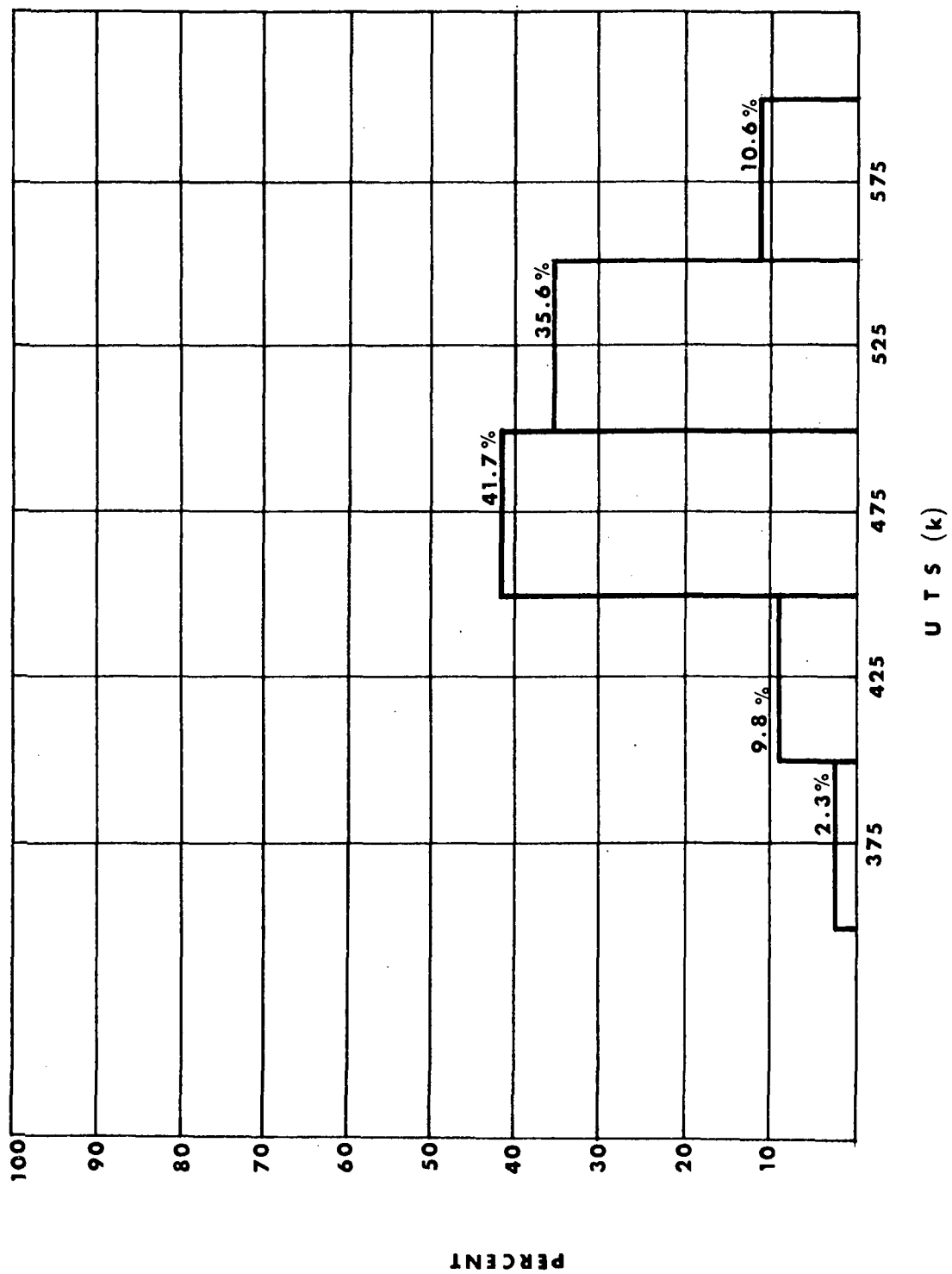


FIGURE 8. FILAMENT STRENGTH DISTRIBUTION

(57 inches) for approximately 106 meters (350 ft); whereas, the calculated bundle strengths were obtained from ten individual filament tests from the end of each run. Comparisons between actual and calculated bundle strengths are listed in Table 4.

TABLE 4. Bundle Test Comparison

	Bundle Strength (@ .41 ksi)	Calculated Bundle Strength (@ .41 ksi)
Number of Tests	90	900
Average UTS	147	153
High	165	199
Low	122	103
CV	8.3	12.8
	12.2	19.6

The calculated bundle strengths are approximately 4% higher than the actual tests which indicates that on an average, the test from the end of each run gives a good indication of average strength of the filament in the run. A plot of bundle strength tests-vs-calculated bundle strength is shown in Figure 9.

C. COMPOSITE TEST

The results of testing on the 32 experimental test pieces are summarized in Table 5. Foil resistance, transverse strength; short transverse strength; longitudinal strength; UTS efficiency, and fabricating parameters are listed. Some data is missing for samples N-13 through N-22. These samples were processed in two packages, and in each case, very poor bonding resulted from failure of impure styrene to distill from the package. The origin of the impure styrene was two-fold: coated filament and sheet styrene.

Both the filament coated with styrene and the styrene film failed to accomplish their objective of obtaining more uniform filament spacing; in addition, they interfered with the bonding operation.

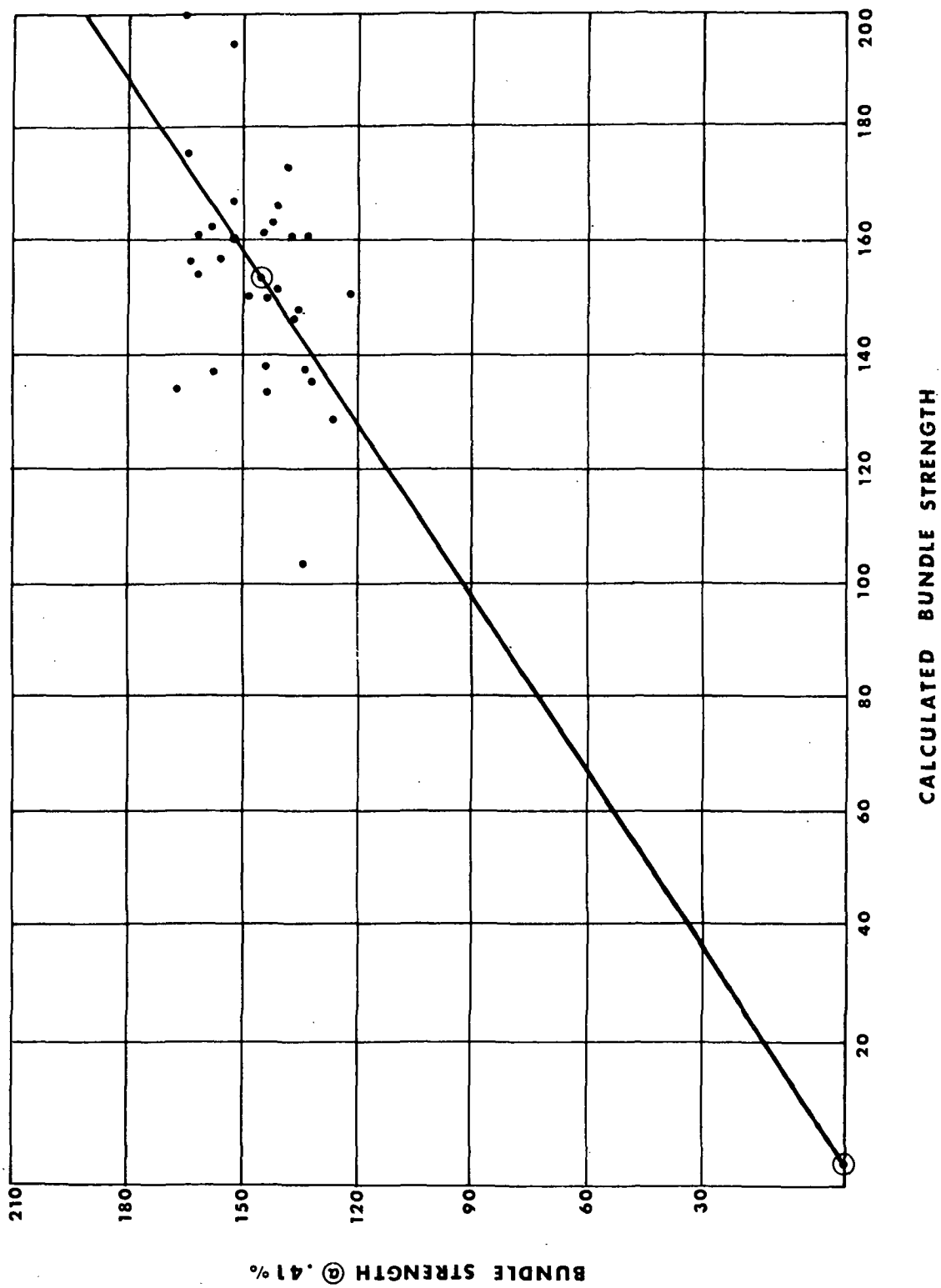


FIGURE 9. BUNDLE STRENGTH-VS-CALCULATED BUNDLE STRENGTH

TABLE 5. Summary of Test Results (contd.)

Sample No.	Foil Resistance $\times 10^{-6} \Omega$	Transverse UTS (psi)		Short Transverse Strength (lb)		Longitudinal Strength (ksi)	UTS Efficiency (percent)	Pressing Parameters		Cleaning		Separator Sheets	
		Avg. of 6	6-layer	Epoxy	Bond			Deg. F.	Time	Foil	Filament	Thick	Thin
N 1	166	(6200)	11,100	990	925	158 178	80 92	925	1 hr	degr-MEK	as-recd	X	
2	166	(6200)	9,800	2360	480	157 167	80 86	↑	↑	degr-MEK	↑		X
3	34	(4700)	4,400	2120	280	172 162	96 79	↑	↑	degr-wire br	↑	X	
4	34	(4100)	7,040	960	210	175 161	98 78	↑	↑	degr-wire br	↑		X
5	101	(7700)	8,100	1660		185 178	96 99	↑	↑	degr-deox	↑	X	
6	101	(7000)	14,500	430	1650	165 165	86 87	↑	↑	degr-deox	↑	X	
7	89	(6100)	11,700	1280	1775	177 151	97 87	↑	↑	degr-deox-wire br	↑	X	
8	89	(6300)	10,300	670	1710	145 157	80 90	↑	↑	degr-deox-wire br	↑	X	
9	18	(7100)	12,500	305	945	141	81	↑	↑	degr-deox-zincate	↑	X	
10	18	(6700)	12,100	760	1160	178 157	102 87	↑	↑	degr-deox-zincate	↑	X	
11	122	(700)	9,500	450	1970	125 195	70 94	↑	↑	deox-etch-deox-wire br	↑	X	
12	122	(5600)	9,800	1450	2860	140 159	79 77	↑	↑	deox-etch-deox-wire br	↑	X	
13	361	---	---	---	---	110 97	49 54	↑	↑	as-recd styr-coat fil	↑	X	
14	384	---	---	---	---	125 83	55 46	↑	↑	degr-deox styr-coat fil	↑	X	
15	926	---	---	---	---	94 132	48 71	↑	↑	as-recd uncoated fil	↑	X	
16	1734	---	---	---	---	114 114	58 62	↑	↑	degr-deox	↑	X	
17	543	---	---	---	---	96 102	48 61	925	1 hr	as-recd	as-recd	X	

TABLE 5 (concluded)

Sample No.	Foil Resistance x 10 ⁻⁶ Ω	Transverse UTS (psi)		Short Transverse Strength (lb)		Longitudinal Strength (ksi)	UTS Efficiency (percent)	Pressing Parameters		Cleaning		Separator Sheets		
		Avg. of 6	6-layer	Epoxy	Bond			Deg.F	Time	Pressure	Foil	Filament	Thick	Thin
N 18	14	---	---	---	---	128 99	64	925	1 hr	3 T	degr-zincate	degr-zincate	X	
19	316	---	---	---	---	135 122	75 70	↑						X
20	41	---	---	---	---	136 142	75 82	↑						X
21	684	---	---	---	---	152 172	81	↑						X
22	382	---	---	---	---	179 155	96	↑						X
23	135	---	15,600	---	---	172 155	94 85	↑						X
24	135	---	16,900	---	---	155 146	84 80	↑						X
25	469	---	13,500	---	---	157 154	79 105	↑						X
26	366	---	19,200	---	---	130 126	65 86	925	1 hr	3 T	as-recd	as-recd	X	
27	117	---	12,350	1540	None	142 148	80 73	940	1/2 hr	3 T	etch-deox	etch-deox	X	
28	120	---	12,300	1860	None	146 156	83 77	940	1/2 hr	3 T	as-received	1100/P	X	
29	98	---	15,900	1500	None	148 137	71 79	940	10 min	3 T	etch-deox	6061/P	X	
30	100	---	15,200	1620	None	127 143	61 82	940	10 min	3 T	as-recd	1100/P	X	
31	100	---	12,850	1720	1150	168 170	84 76	900	1 1/2 hr	3 T	as-recd	1100/P	X	
32	100	---	13,300	1540		147	74	900	1 1/2 hr	3 T	Methanol	Methanol 1100/P	X	

Table 5 lists the foil resistance data summarized in Figure 7. Columns 2 and 3 of the table list a summary of the transverse strength data. Column 2 is the average of six individual tests, and Column 3 is one test of a six-layer sample. In all areas except one, the six layered sample gave higher results than the average of six monolayer samples.

The average of the first 12 samples is a little over 10,000 psi; whereas, the average of the last 10 samples is approximately 14,700 psi. The principal cause of the improvement in transverse strength is attributed to the use of braze powder in the styrene which helps maintain the spacing between filaments and assists in assuring a good bond. A summary of transverse strength-vs-UTS efficiency is shown in Figure 10.

Column 4 lists the results for the short transverse tests. The data are given in two columns: epoxy and bond, indicating that the failure was in the epoxy or in the composite bond. The numbers are the load at failure.

Because of the large numbers of failures in the epoxy, the test was not very satisfactory; however, later samples did indicate fewer failures in the bond when compared with earlier samples. A summation of this data is shown in Figure 11.

Column 5 is the longitudinal strength with each entry representing the average results of four end-to-end samples. The two entries for each piece represent material from the right and left side of the sample.

Column 6 gives a composite efficiency index which is obtained by dividing composite strength by bundle strength and then dividing by the bundle strength factor which is obtained from the cv of the filament strengths. In essence, the efficiency index says: "If the composite behaves like a bundle of fibers, a low efficiency will result; if because of compositing, the composite strength approaches the rule of mixtures, a high efficiency rating will be attained."

Figure 12 summarizes the composite efficiency and UTS data. In general, the data indicate that higher efficiencies are associated with better strengths which was expected.

Columns 7, 8, 9 and 10 of Table 5 list the pertinent conditions of cleaning and processing for each of the samples. Because of the large number of variables and the limited number of samples, only tentative conclusions can be reached on the variations investigated, which are as follows:

a. Contaminated styrene definitely affected the bonding adversely with consequent reduction in UTS and UTS efficiency.

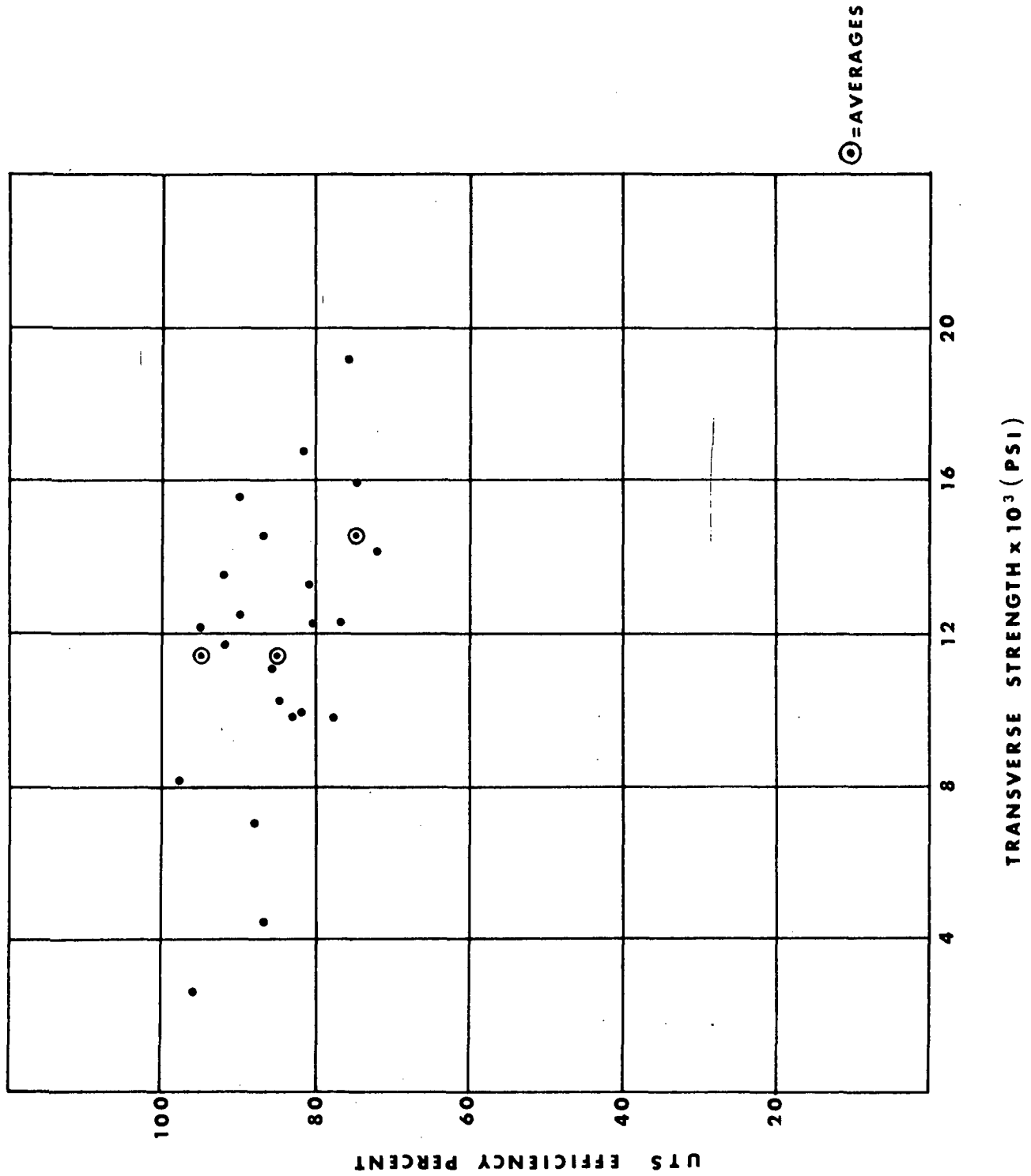
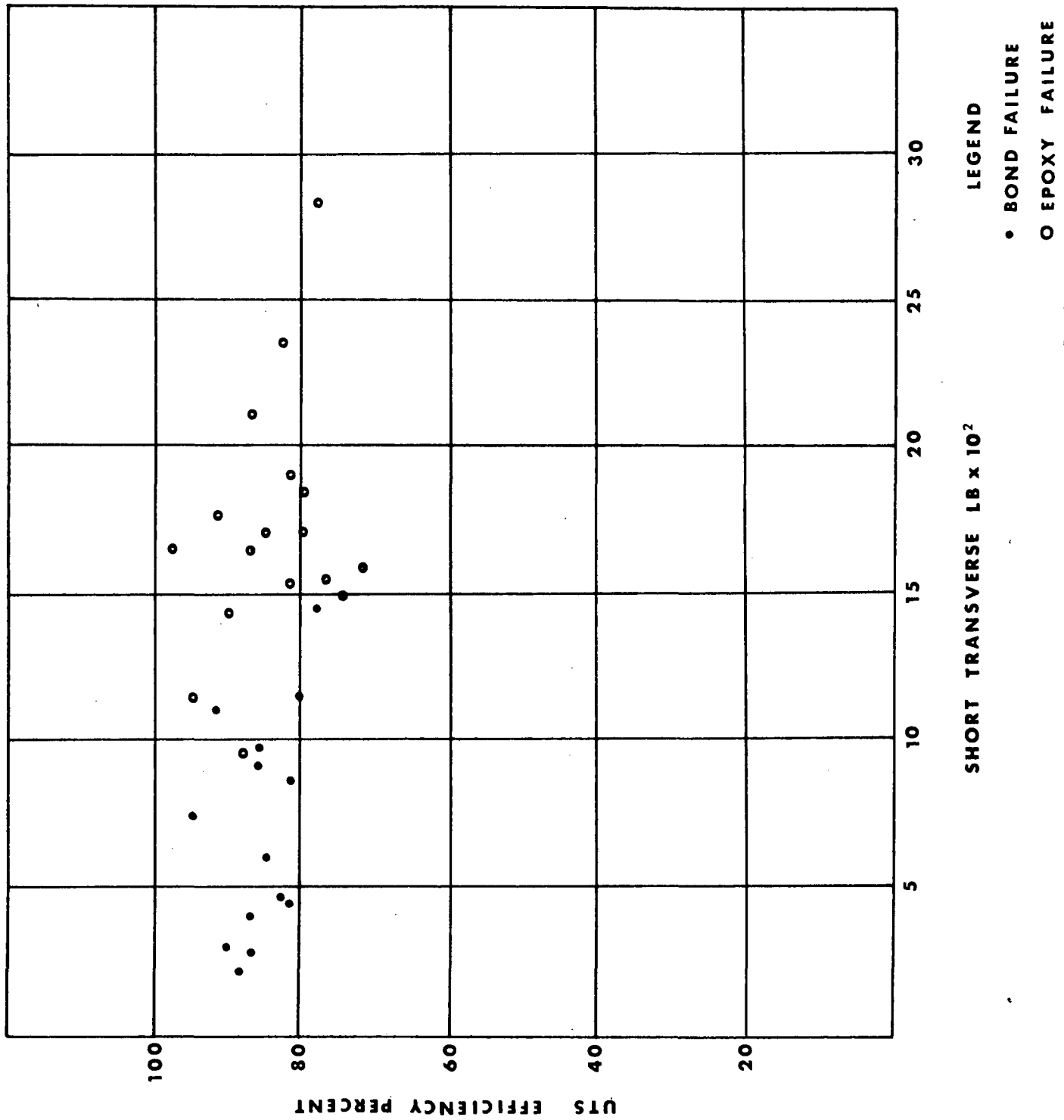


FIGURE 10. TRANSVERSE STRENGTH-VS-UTS EFFICIENCY



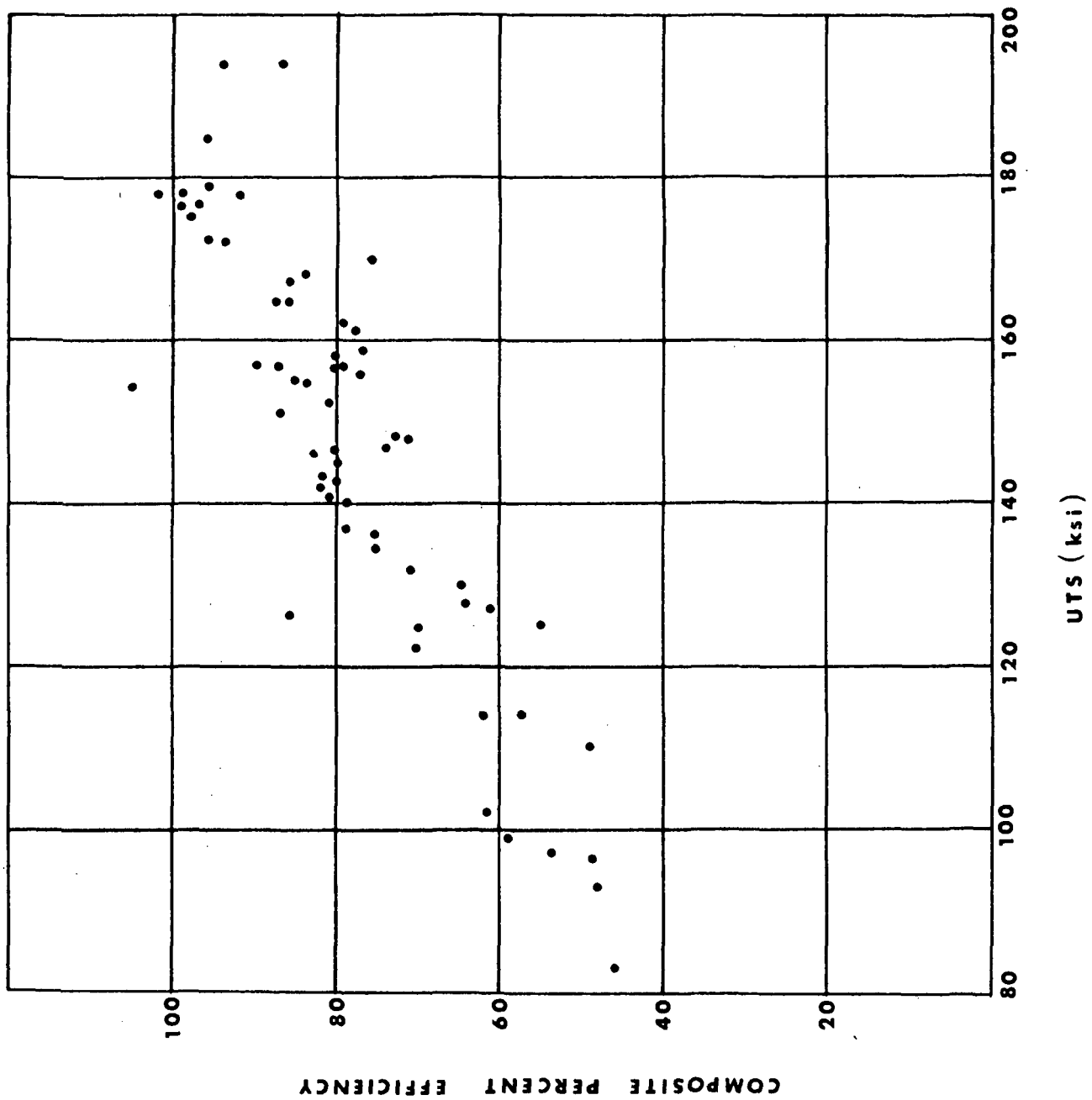


FIGURE 12. UTS-vs-COMPOSITE EFFICIENCY

- b. The thickness of shim material produced little noticeable effect.
- c. No significant difference was noted from wire brushing or omission of this operation.
- d. Cleaning the aluminum and filament in Methanol was the best compromise between cost and obtaining a uniformly clean surface. Etch and deoxidizing or zincate were also satisfactory.
- e. The best combination of time, temperature and pressure appeared to be: 900°F, 3 T/sq. in. for one-hour when brazing powder was used in the styrene, and 925°F when no brazing powder was used.
- f. The use of brazing powder in the styrene materially assisted in obtaining more consistent bonding between the two pieces of aluminum foil.

D. QUALITY CONTROL

The discussion of work performed in the area of quality control will be covered in the following five subparagraphs: Specifications, Foil Thickness, X-Ray, C-Scan and Metallographic Examination.

1. Specifications. Twelve specifications were written covering the major material used and process operation for fabrication of boron-aluminum monolayer filament. These specifications are enclosed as Appendix C.
2. Foil Thickness Gaging. Using mechanical methods for measuring the thickness of small samples of foil is very satisfactory; however, on large pieces, this method is not very practical. In view of the need for accurately measuring foil thickness in large pieces, NDT Instruments, Inc. was contacted to determine if their eddy current techniques could meet the requirements. The results of their effort is contained in Appendix A. Thickness gaging accuracies on aluminum foil of $\pm .00001$ -in. were achieved.
3. X-Ray Examination. The advantages of X-rays are that they provide a permanent record of the following items: filament spacing, filament breaks, filament crossovers and filament alignment. On monolayer composite material, all of the above items, except small filament breaks, are reflected in the surface of the composite and can be detected upon visual examination.
4. C-Scan. Through transmissions, C-scans were performed on all pieces. Some difficulty was experienced initially because of the flexibility of the composite foil. This problem was overcome by placing the pieces

in a rigid aluminum picture frame which securely held the outer .5-inch edges of the piece. Three types of C-scans were obtained: uniform, a few small areas exhibiting attenuation and large areas of attenuation. The results were in general agreement with visual observation of the piece. The latter pieces, with good bond, had uniform C-scans. The pieces with poor bonding and misaligned filaments showed large areas of attenuation. The in-between C-scans were not too helpful; test bars taken from these samples exhibited either high or low results but were not necessarily correlated with the attenuation areas.

5. Metallographic Examination. Test bars exhibiting both high and low results were examined metallographically and with the electron microscope. No significant differences were noted between satisfactory and unsatisfactory specimens.

SECTION 5. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations are made:

- Measuring the contact resistance of the aluminum gave a good indication of the surface condition of the foil.
- The best pretreatment for aluminum foil was solvent degreasing.
- The eddy current method for determining foil thickness was satisfactory.
- Use of styrene sheet to maintain filament alignment was unsatisfactory and interfered with bonding.
- Use of styrene-coated filament to maintain alignment was unsatisfactory.
- Correlation between filament test from the end of run, and bundle test covering filament within the run, was good.
- Low ultimate tensile strength results were not explained by metallographic examination.
- Straight-sided ultimate tensile strength specimens with eloxed edges produced higher strength and more consistent results than sheared specimens.
- Short transverse strengths obtained by bonding tabs on the sample were unsatisfactory.
- Transverse strengths on monolayer samples produced low and erratic results.
- The use of brazing powder in the styrene fugitive binder improved bonding and filament spacing.
- It is recommended that further work be done on the use of brazing powder in the fugitive binder.

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APPENDIX A

THICKNESS GAGING OF ALUMINUM FOIL BY
LINEARIZED EDDY CURRENT TECHNIQUES

Applications Report No. 504

Covering

Program Conducted by

NDT Instruments, Inc.
705 Coastline Drive
Seal Beach, California

For

Martin Marietta Aluminum Inc.

INTRODUCTION

Initial work by NDT Instruments, Inc. verified that eddy current techniques can sense small thickness changes in aluminum foil. However, as is typically the case with eddy currents, the response was nonlinear and adequately sensitive only across a small thickness range (for a given calibration point).

The eddy current technique offered the potential advantage of rapid thickness measurements with probe access to only one surface of the foil. Therefore, a program was initiated to determine if an eddy current gage could be designed which gave a linear, direct readout over an adequately wide range of foil thickness.

SPECIFIC OBJECTIVES

The specific objectives of this program were to obtain, for 1100 and 6061 aluminum foil, a linear eddy current response exhibiting a minimum gaging accuracy of $\pm 10\%$, over a thickness range of about 0.0025" to 0.0032".

CONCLUSIONS

Based on the experimental results of this program, the following conclusions appear justified.

1. With the proper circuit and probe design, eddy current methods can surpass all of the performance specifications set forth in the above SPECIFIC OBJECTIVES.

2. Thickness gaging accuracies of ± 0.0001 " can be expected over a range of, at least, 0.0017" to 0.0041", for both

1100 and 6061 aluminum foil.

3. The linear response can be shifted to either higher or lower foil thickness ranges by simple calibration techniques. For example, a calibration change will permit accurate gaging of foil thicknesses either greater or less than the range of 0.0017" to 0.0041".

RECOMMENDATIONS

It is recommended that linearized eddy current techniques be used as a rapid means of gaging aluminum foil thicknesses from a single surface.

SPECIMENS

A total of 12 type 1100 aluminum and 13 type 6061 aluminum samples (each several inches square) were initially submitted. Later in the program, 8 additional "unknown thickness" samples of both foil types were provided. Details concerning these samples appear later in the report.

INSTRUMENTATION

A laboratory-level eddy current circuit was adapted/oriented for the foil thickness measurements. Several probe configurations were constructed, which had diameters in the vicinity of 1/4".

EXPERIMENTAL PROCEDURE

A series of preliminary tests were conducted for the purpose of optimizing the eddy current response (thru circuit/probe design changes). Once the equipment appeared satisfactory, calibration

curves were generated for both the type 1100 and 6061 aluminum foil samples.

The "unknown thickness" samples were then gaged with the eddy current setup and, through the use of the respective calibration curve, the thickness of each sample was determined. The actual thickness of the "unknown samples" was then measured with a mechanical micrometer and compared with the eddy current values.

A Model 106-102 Mitutoyo micrometer, designed especially for measuring foils, was purchased to standardize all mechanical thickness gaging reference data. The micrometer's accuracy was specified as ± 0.0001 ". Experience verified that its repeatability was within this range.

Following the eddy current tests, all the samples (known and "unknown") were gaged with the micrometer, by a single operator. This procedure assured that all mechanical thickness values were standardized to a single micrometer and operator. A slight micrometer thickness variation (about 0.0001" maximum) was noticed across the surface of some of the foil samples. When such a variation was observed, the most prevalent value in the region of the eddy current measurement was accepted as being the actual value.

RESULTS

The final results of the experimentation are given in Table I, Figure 1 and Table II for the type 6061 aluminum foil are given in Table III, Figure 2 and Table IV.

Table I

Eddy Current Readings On 1100 Aluminum Foil Reference Samples

Sample Identity	Micrometer Thickness-mils	Replicate Meter Readings	Average Reading
1100-A-1	2.1	34, 34, 34	34
1100-A-2	2.1	34, 34, 34	34
1100-A-3	2.1	34, 34, 34	34
1100-I-II	2.1	34, 34, 34	34
1100-II-II	2.1	34, 34, 34	34
1100-P-1	2.6	51, 51, 51	51
1100-P-2	2.6	50, 50, 50	50
1100-P-3	2.6	50, 50, 50	50
1100-I-III	2.6	51, 51, 51	51
1100-II-III	2.6	50, 50, 50	50
1100x	0.7	-- -- --	--
1100y	1.0	-- -- --	--
1100z	3.1	66, 66, 65.5	65.8
1100x + 1100y	1.7	22	22
1100-II-II + 1100x	2.8	57	57
1100x + 1100-I-III	3.3	73	73
1100y + 1100-I-III	3.6	80	80
1100x + 1100z	3.8	86	86
1100y + 1100z	4.1	94	94

(Reference samples used to generate calibration curve)

Figure 1 Eddy Current Response On 1100 Alum. Foil
(calibration curve)

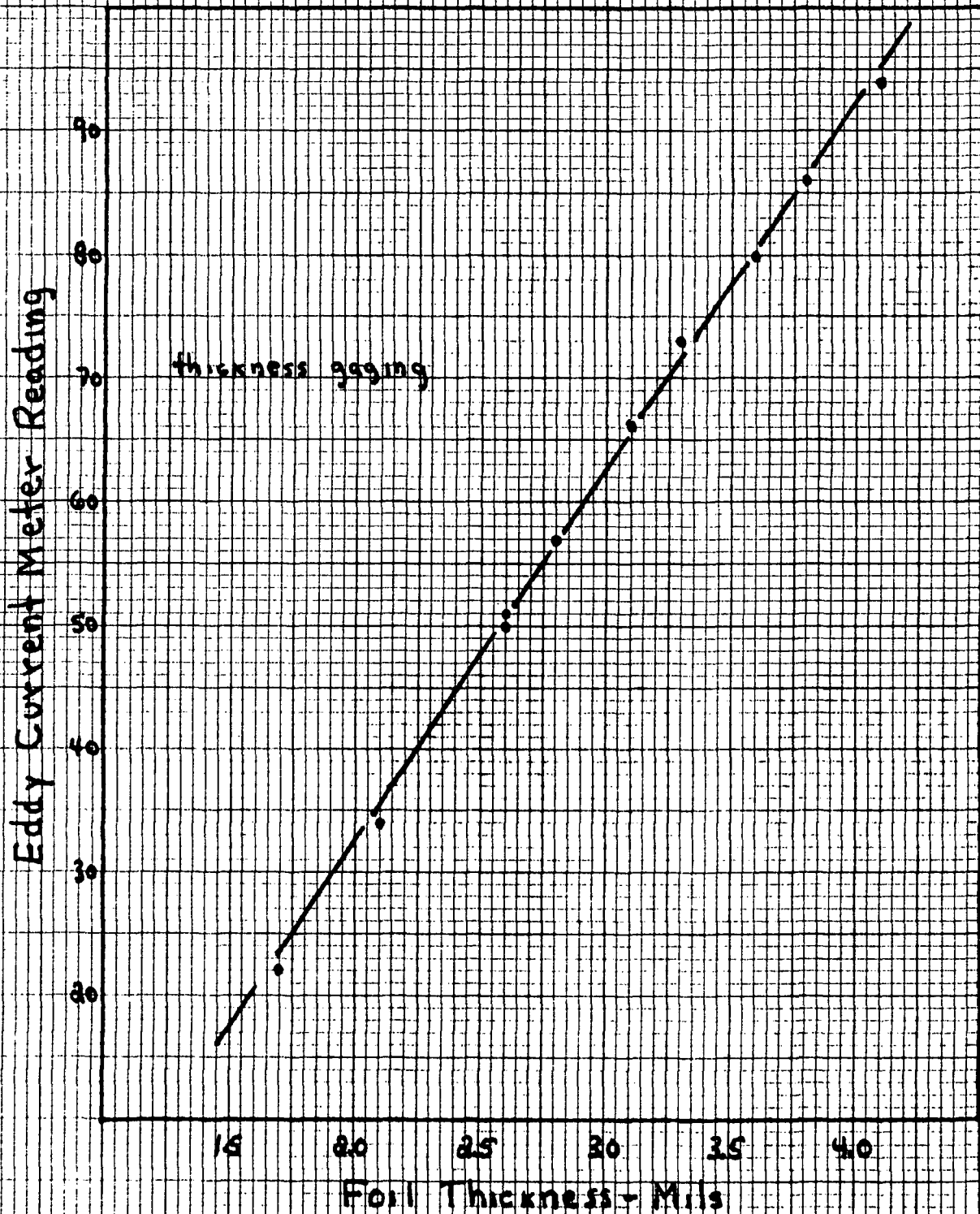


Table II

Eddy Current Gaging Of "Unknown"

1100 Aluminum Foil Samples

Sample Identity	Eddy Current Meter Reading	Thickness Per Calib. Graph-mils	Micrometer Thick.-mils	Deviation $\pm$ mils
1100-B	35, 35, 35	2.1	2.1	0.00
1100-C	35, 35, 35	2.1	2.1	0.00
1100-D	36, 35.5, 35.5	2.11	2.1	0.01
1100-E	36, 36, 36	2.12	2.1	0.02
1100-G	50, 50, 50	2.6	2.6	0.00
1100-H	50, 50, 50	2.6	2.6	0.00
1100-I	49.5, 49.5, 49.5	2.6	2.6	0.00
1100-J	49, 49, 49	2.6	2.6	0.00

* See Figure 1

Table III

Eddy Current Readings On 6061 Aluminum

Foil Reference Samples

Sample Identity	Micrometer Thickness-mils	Replicate Meter Readings	Average Reading
6061-A1	2.1	23, 23, 23	23
6061-A2	2.1	22, 22, 22.5	22.3
6061-A3	2.1	23, 23, 23	23
6061-I-IV	2.1	24, 23.5, 23.5	23.7
6061-II-IV	2.1	23.5, 23.5, 23.5	23.5
6061-F	2.6	37, 37, 37	37
6061-I-V	2.6	35, 35, 35	35
6061-II-V	2.6	34, 34.5, 34	34.25
6061-F-3	3.6	66, 66, 66	66
6061-I-II	3.6	67, 67, 67	67
6061-II-II	3.6	65.5, 66, 65.5	65.7
6061-F-1	3.6	66, 66, 66	66
6061-F-2	3.6	65, 65, 65	65

(Reference samples used to generate calibration curve)

Figure 2 Eddy Current Response On 6061 Alum. Foil
(calibration curve)

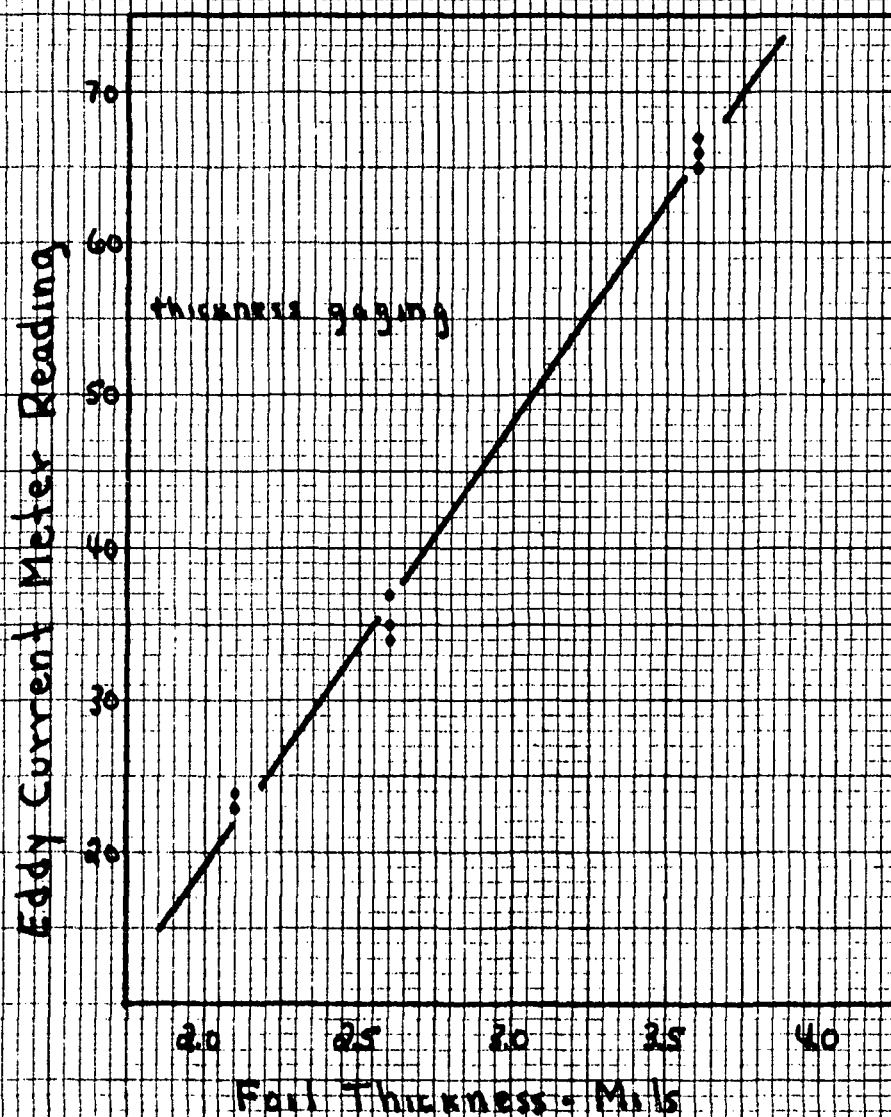


Table IV

Eddy Current Gaging Of "Unknown"

6061 Aluminum Foil Samples

Sample Identity	Eddy Current Meter Reading	Thickness Per Calib. Graph-mils	Micrometer Thick.-mils	Agreement- ± mils
6061-B	23, 23, 23	2.12	2.05	0.07
6061-C	24, 24, 24	2.15	2.1	0.05
6061-D	24, 24, 24	2.15	2.1	0.05
6061-E	24, 24, 24	2.15	2.0	0.05
6061-G	64, 64, 64	3.55	3.6	0.05
6061-H	65, 65, 65	3.58	3.6	0.02
6061-I	65, 65, 65	3.58	3.6	0.02
6061-J	65.5, 65.5, 65.5	3.60	3.6	0.00

* See Figure 2

As can be seen, a linear response was obtained, with foil thickness measurement accuracies exceeding ± 0.0001 ". As expected, the electrical conductivity difference between the 1100 and 6061 foils produced different eddy current calibration curves. This means that the operator must calibrate to or know what type of aluminum foil is being tested. Since the data taken during this project needed to be relative, the eddy current circuit was initially set only once for an arbitrary response to both types of aluminum foil.

DISCUSSION

The results of this project verify that a small, portable eddy current instrument can be constructed for accurately gaging the thickness of aluminum (and other metal) foils. Since the response has been linearized, a direct readout of thickness is possible when the instrument is calibrated on a given alloy.

APPENDIX B

AN EXAMINATION OF FACTORS THAT AFFECT
TRANSVERSE PROPERTIES OF
ALUMINUM-BORON COMPOSITES

by

Robert Charles Kietzman

A Thesis Submitted to the Faculty of the
DEPARTMENT OF METALLURGICAL ENGINEERING
In Partial Fulfillment of the Requirements
For the Degree of
MASTER OF SCIENCE
WITH A MAJOR IN MATERIALS ENGINEERING
In the Graduate College
THE UNIVERSITY OF ARIZONA

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STATEMENT BY AUTHOR

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APPROVAL BY THESIS DIRECTOR

This thesis has been approved on the date shown below:

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Associate Professor of Metallurgical
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July 12, 1972
Date

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ABSTRACT

Transverse tensile strength properties of boron-filament reinforced aluminum composites with various heat treatments and stress relief cycles were studied in an effort to determine the procedures necessary to improve these properties. Tensile specimens were fabricated by chemically etching to free the filament ends outside the reduced gage length section and by bonding of metal grips with adhesive onto the ends of the specimens. Initially a range of strain rates was used to determine the optimum strain rate to use for this experiment. The data were evaluated in terms of percent elongation, yield strength, ultimate tensile strength, modulus of elasticity, and the mode of failure. The experimental results indicated that the T6 heat treatment provided the highest transverse strength properties of the eleven different procedures evaluated.

CHAPTER I

INTRODUCTION

The transverse strength properties, of unidirectionally reinforced boron-aluminum matrix composites, has been much lower than expected, according to early investigators in 1969. This behavior has been attributed to longitudinal splitting in the boron filaments, with the cracks consequently propagating into the metal matrix causing premature tensile failure. The splits are inherent in the filaments, due to the high temperature of vapor deposition of the boron onto the 1/2 mil tungsten wire core. This is due to the thermal contraction differences between the two materials when they cool. These cracks have also developed during vapor deposition of coatings such as silicon carbide or boron nitride on the boron which are applied to increase the chemical compatibility of the filaments with the matrix at high temperature exposure.

Recent industrial programs to resolve this splitting problem have produced a 5.6 mil-diameter boron filament which is reported to have relatively few splits such that higher, transverse strength properties can be realized (Kreider, Dardi, and Prew, 1970). Kreider, et al., reported on a tensile specimen preparation technique where the metal matrix around the filament ends, of transverse tensile specimens, are chemically etched to remove the metal. This produces a reduced gage length tensile specimen with the filaments extending out the sides

beyond the gage length test portion of the specimen. The result is that cracks in the filaments at the ends that developed during shearing of the specimens are removed from the test area of the specimen.

Other very recent industrial efforts on this splitting problem have resulted in development of glass core and carbon-filament core upon which the boron is vapor deposited. These types of boron filament are reported to have less thermal contraction differences and consequently fewer inherent cracks or splits.

For the research program reported herein, the only available improved filaments were the 5.6 mil diameter boron without a coating. This type filament, therefore, was used with the two most common aluminum alloys (6061 and 2024) and were fabricated into a two-filament layer composite using a diffusion method in a heated-platen press. The fabrication of the panels was performed by Harvey Aluminum Company, one of the foremost developers of filament reinforced metal composites, since fabrication was not within the scope of this research effort.

To evaluate other methods to improve transverse strength properties, assuming once again that the strength depended on the matrix and not the splitting of filaments, various thermal treatment techniques were used on the matrix. These methods used were natural and artificial aging, solution heat treatment, thermal stress relief, thermal cycling as a stress-relief procedure, and strain cycling as a stress-relief procedure.

CHAPTER 2

LITERATURE SURVEY

2.1 Critical Areas for Transverse Strength

Transverse strength of unidirectionally reinforced boron-fiber-aluminum-matrix composites has been of concern for many applications since low transverse strengths on the order of 12,000 psi to 22,000 psi have been experienced, as reported by Christian (1969) and Dolowy (1969b). These applications utilizing a composite with longitudinal tensile strengths in excess of 200 ksi in the direction of the boron fibers, also have need for transverse strengths which would be at least equal to the conventional aluminum strength of 45 to 65 ksi (6061 and 2024, respectively). Specific applications such as aircraft or missile structural members including outer skins need good transverse strength properties. Adsit and Forest (1969) reported on testing performed at Convair on aluminum-boron composites for structural stringers to reinforce and stiffen skin structures. Transverse compression tests were performed to establish design strengths to be expected from aluminum-boron composites. Christian (1969) reported on results of a Convair study program for components for the F-111 fuselage involving twelve major structural components including bulkheads, frames, longerons, door panels, shear panels, fittings, and a shear beam. In most cases, substantial weight savings, ranging from 18% to 60%, were identified by this study. In several cases, however, the low shear and transverse strength of the composite prevented a significant

weight saving. In other cases, it was apparent that the weight payoff could be substantially increased, by a factor of two in some cases, if transverse properties could be increased by relatively small amounts.

The Convair Division of General Dynamics Corporation has been flight testing aluminum-boron-composite access doors on F-102, F-106, and F-111 airplanes and has been fabricating and evaluating aluminum-boron composite satellite payload adapters of conical shape 60 inches in diameter and 42 inches in length.

Another application where low transverse strength has been of concern, is for aircraft gas turbine compressor and fan blade usage. Tsareff (1969) of the Allison Division of General Motors Corporation evaluated transverse strength properties of aluminum-boron composites since the blades experience a cantilever-beam-fatigue type loading. Alloy 7178 was used for the matrix in these tests and a transverse tensile strength of 43 ksi was reported, mainly due to the high shear strength of the solution treated and aged 7178 matrix. Axial filament splitting was observed in these tests due to the inherent radial sub-surface cracks in the filaments.

Kreider, et al., (1970) of the Pratt & Whitney Aircraft Division of United Aircraft Corporation has also been evaluating aluminum-boron composite transverse strength for third-stage compressor blades that are exposed to a 600^o F. operating temperature. The blades in these turbine engines also experience the cantilever-beam-fatigue-type loading. It was reported that all the filaments in the fracture plane showed splitting in the transverse tests.

Hanby (1971a) reported that NASA's planned space shuttle will help to maintain the present high interest and activity in the development of aluminum-boron composites. Hanby also reported that the Hamilton Standard Division of United Aircraft Corporation has been evaluating aluminum-boron composites for helicopter blades and propeller blades and the Bendix Corporation has been evaluating this material for landing gears.

It was concluded from these reports that transverse strength of unidirectional boron fiber-aluminum matrix composites is of utmost importance to the success of these various applications, and that much test effort is being exerted in R&D programs to improve this property.

2.2 Boron Filaments

One of the parameters of aluminum-boron composites that is being evaluated and improved is the boron filament itself. Original boron filaments consisted of boron vapor deposited from a boron trichloride (halide) and hydrogen gas mixture on to a one-half mil diameter tungsten wire substrate. This continuous filament has a tensile strength of about 450 KSI and a modulus of elasticity of about 56×10^6 psi. One problem associated with this type of filament during filament fabrication has been the thermal contraction difference between the tungsten wire core and the deposited boron. This difference has caused subsurface radial cracks in the boron that are detrimental to composite physical properties. Transverse testing almost always results in splitting of the filaments on the fracture plane, thus limiting the transverse tensile strength of the composite, as reported by Long (1969)

and Hanby (1971b). Use of a silicon-carbide coating on the boron filament, to decrease chemical reaction between the aluminum matrix and the boron at high temperature, has increased the problem with additional radial cracks created during application of the coating on the filament.

Two new developments in boron filaments are the use of a glass-based core substrate and a carbon monofilament core substrate upon which the boron is vapor deposited. These combinations provide a low density for the continuous filament as well as offer a lower cost potential. It has been found with these types of filaments that radial cracks are substantially reduced. This is especially important in transverse strength where premature failure has been attributed to the propagation of the radial crack into the metal matrix thus reducing transverse strength. The average properties of these glass or carbon core filaments tend to be lower (300 KSI tensile strength) than those of the boron-on-tungsten type, but due to the lower density, the specific strength and modulus are about comparable in the boron-on-tungsten composite.

Another recent development in boron filaments is a 5.6-mil-diameter boron filament with the boron vapor deposited on a one-half-mil-diameter tungsten wire. No coating is used on this type of filament in an effort to reduce radial splits. Information concerning the fabrication processes utilized for this filament that make it have less tendency to split is not available. Usage of this type of filament, however, in aluminum matrix composites was reported by Kreider, et al., (1970), where 49 KSI transverse tensile strength was reported (2024 matrix) with no filament splitting in the test specimens.

It was concluded that full potential strength of the aluminum has not been realized during transverse testing since the filaments split with the cracks propagating into the matrix thus causing low transverse tensile strength of the matrix. Therefore, to improve composite transverse strength, the filament splitting had to be eliminated so that the transverse strength could once again be dependent upon the maximum strength that could be developed in the matrix. For this reason, the 5.6 mil diameter filaments produced by AVCO were utilized in the research program reported herein.

2.3 Processes of Composite Manufacture

There are various processes that are being used to fabricate metal-matrix composites. Experimental methods include electroplating, powder metallurgy, explosive bonding, and vapor deposition. For most small research programs, it is usually most convenient to use hot pressing, plasma spraying, or liquid infiltration, which produce higher temperature exposure for the filament with subsequent undesirable inter-metallic compounds created at the filament-matrix interface. However, Lockheed Aircraft has developed a continuous casting method where the filament (protected by a boron nitride coating) contacts molten aluminum. This process permits fabrication of composites with filament contents to 70% by volume, and reduces the overall fabrication costs. Most commercial fabrication, though, of aluminum-boron composites has been by diffusion bonding to reduce the temperature to which the filaments are exposed during fabrication.

A processing technique review was presented by Cornsweet (1971) which outlined many practical possibilities for advanced fabrication methods for aluminum-boron composites. However, they were too numerous and had too many variations to include in this literature survey.

The manufacture of composites is a complex procedure beyond the scope of this test program. So, to avoid fabricating composites that could produce questionable results, a major manufacturer of composites, Harvey Aluminum Company, was contacted and they agreed to fabricate the composite panels for this program using both 2024 and 6061 aluminum alloys with the AVCO 5.6-mil filaments. These panels were fabricated using the diffusion bonding process, details of which are not available.

2.4 Volume Percent of Filaments

Another factor that influences transverse tensile strength is the volume percent of filaments present in the composite. A study reported by Lin, Chen, and Dibenedetto (1969) developed the set of tensile curves shown in Figure 1 for annealed, as well as, aged boron-6061 aluminum composite. The modulus curves for the material are shown in Figure 2. Lenoe (1967a) showed data of modulus versus volume percent boron as shown in Figure 3. Davis (1969) of Harvey Aluminum Company also reported on transverse tensile strength influenced by volume percent filaments as shown in Table 1.

As verified by the test data from the above four sources, transverse tensile strength is greatly influenced by volume percent of filaments.

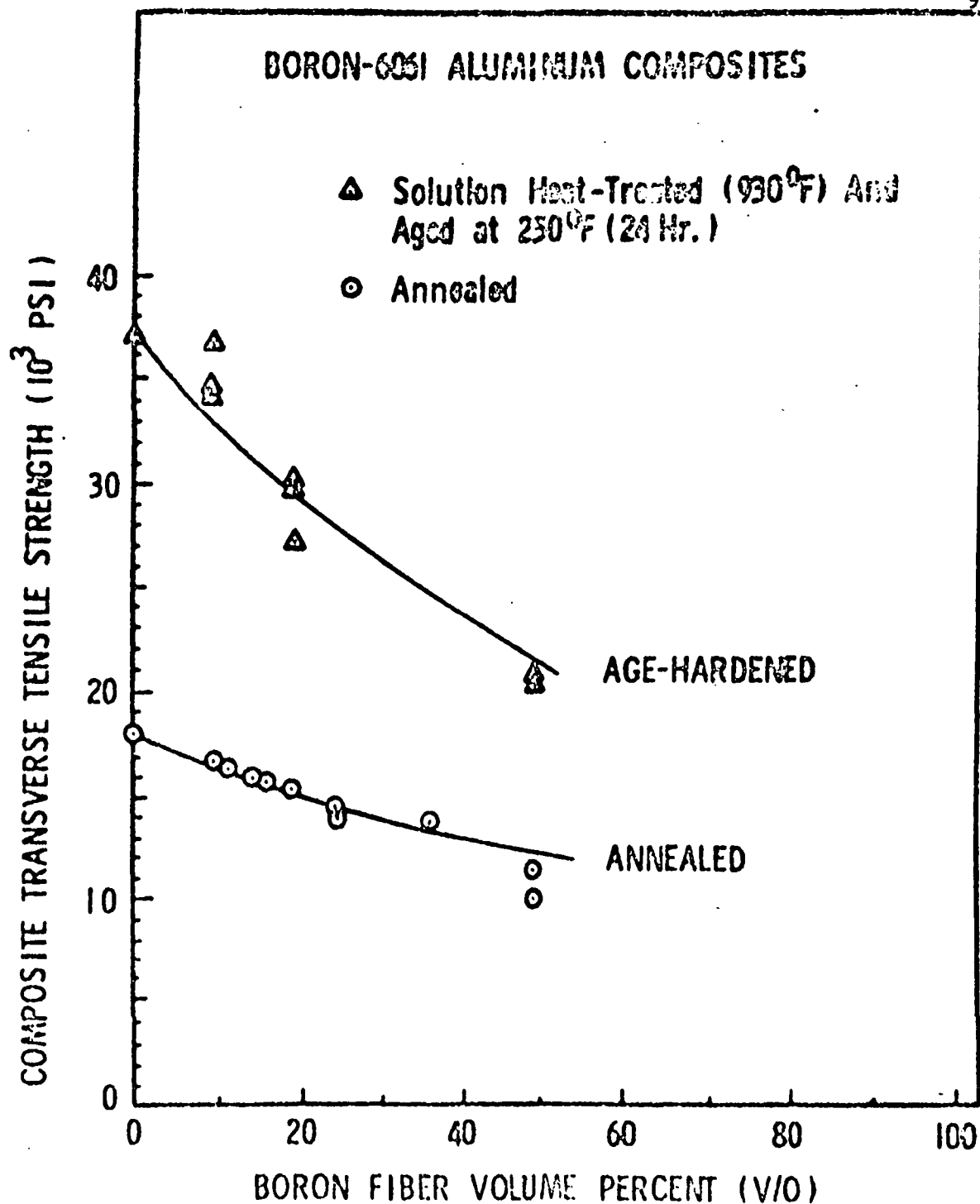


Figure 1. Boron-6061 Aluminum Composite Transverse Tensile Strength vs. Fiber Volume Percent under Different Heat Treated Conditions (Lin, et al., 1969)

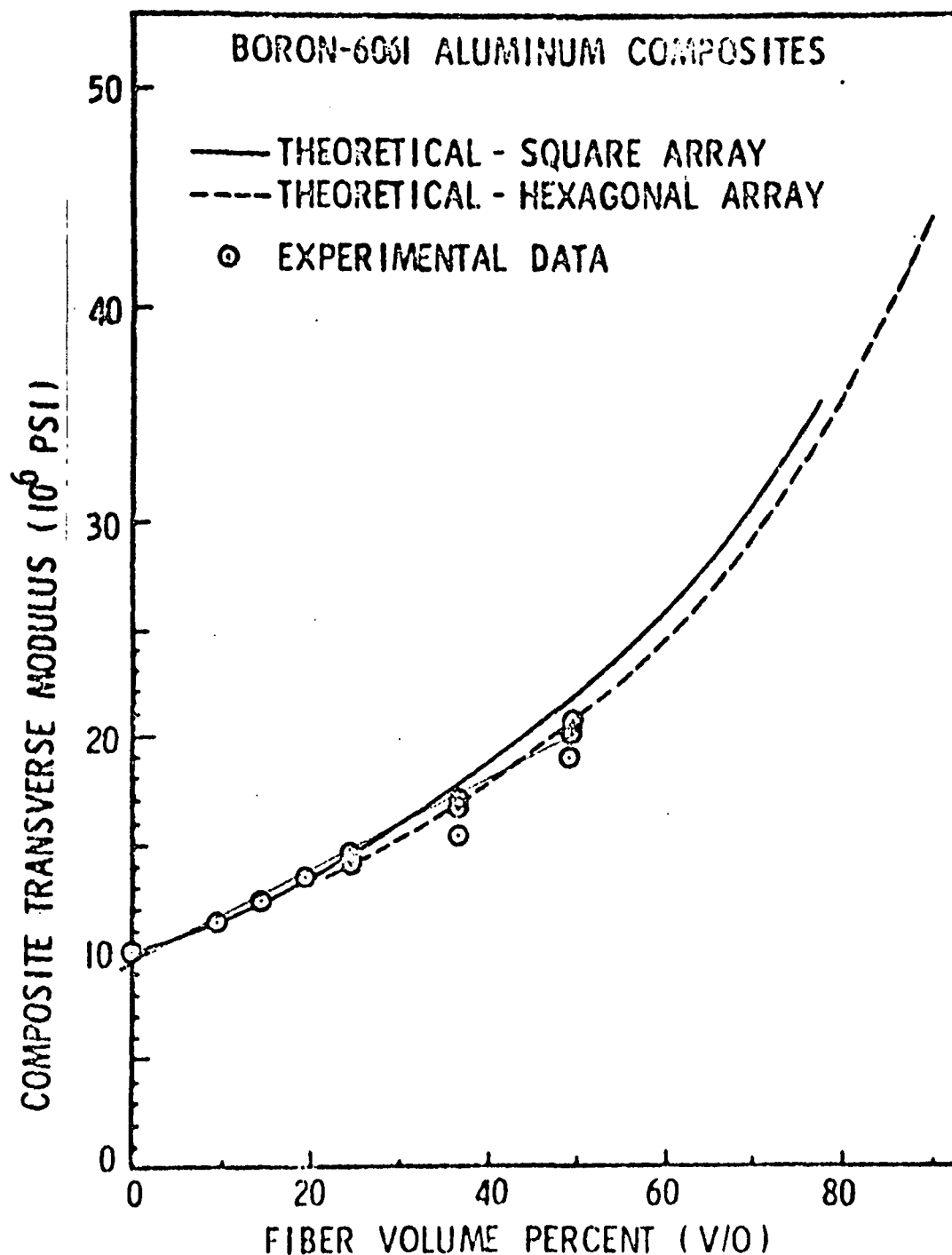


Figure 2. Boron-6061 Aluminum Composite Transverse Modulus vs. Fiber Volume Percent--Comparison Between Analytical Results and Experimental Data (Lin, et al., 1969)

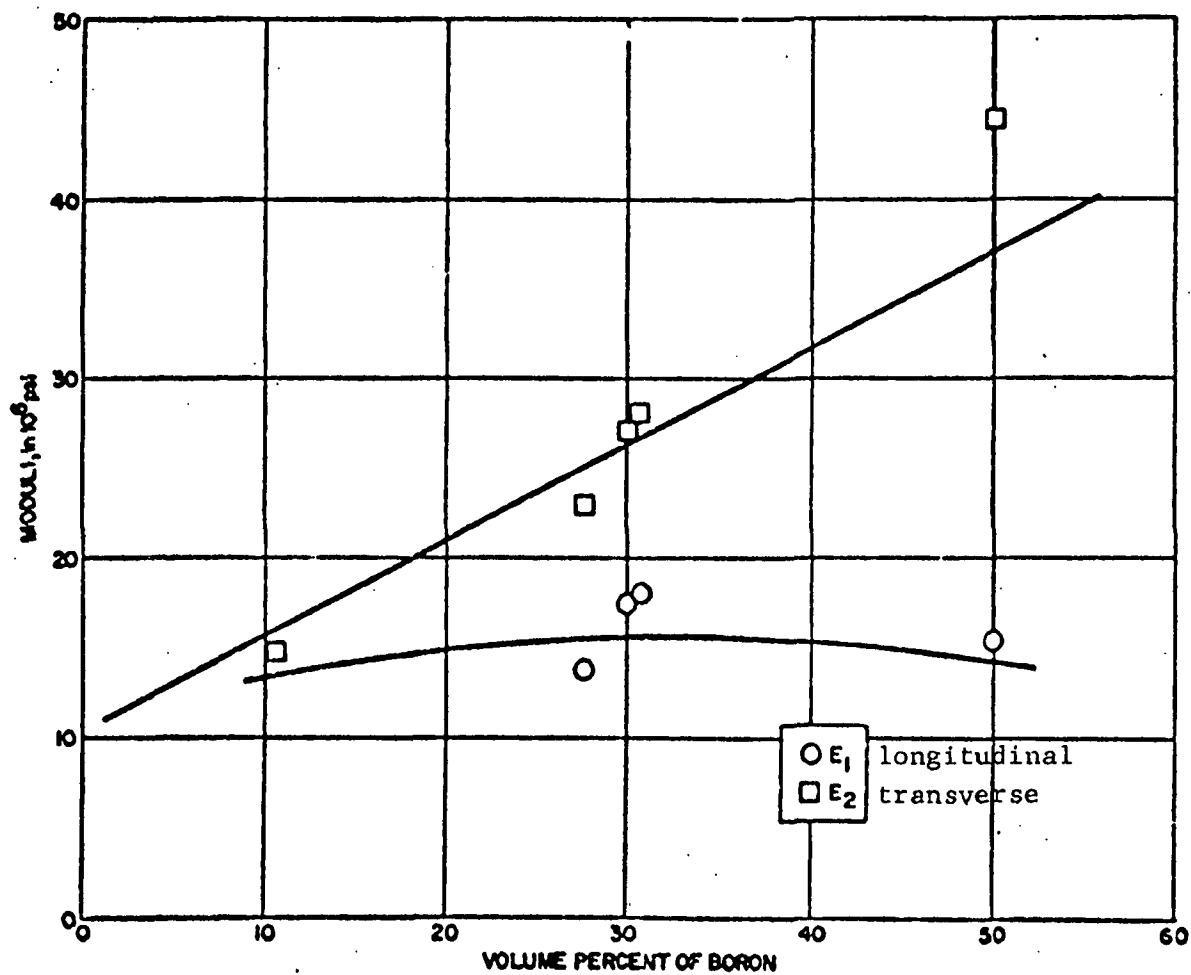


Figure 3. Longitudinal and Transverse Moduli vs. Volume-Percent Boron (Lenoe, 1967a)

Table 1. Transverse Tensile Strength (Davis, 1969)

Matrix	Average Transverse Tensile Strengths (KSI)*			
	10 v/o B	25 v/o B	40 v/o B	50 v/o B
1100	8.5	20.8	12.9	10.5
6061	16.0	7.7	13.7	14.5
2024	30.0	15.3	14.9	12.8

* All failures contained longitudinal split filaments.

The transverse tensile strength decreases as filament volume percent increases. This is most likely due to the increase of filament splitting as the percentage of filaments increases. The transverse modulus, however, increases with increase of filament addition to the composite. This increase in modulus is the result of the Law of Mixtures with the boron filaments having a higher modulus than the aluminum matrix, so that increasing the percentage of filaments increases the composite transverse modulus. For the research program for this thesis, volume percents of 25% and 50% were chosen for study.

2.5 Stress Relief by Thermal Cycling

Taylor, Shimizu and Dolowy (1969) of The Marquardt Corporation evaluated the effect of temperature cycling on relief of residual stresses in composites of Alloys 1145 and 6061 with boron filament reinforcement. The following cycles were utilized:

- a. 20 cycles from 70°F. to 700°F.
- b. 284°F. for 24 hours.
- c. 338°F. for 24 hours.

It was concluded by Taylor, et al., however, that the thermal cycling degraded the strength of the composite. A report by Hamilton and Ebert (1969) discussed a test program to reduce or alter residual stresses. It was found that prestrain was effective in improving tensile strength by 20%. Davis (1969) evaluated a stress relief cycle on 6061 alloy matrix consisting of 200°F. for 16 hours followed by a slow cool. This produced a transverse strength of 17,000 psi (for 37 v/o B) which was much lower than the T6 condition of 31,900 psi.

It was decided to utilize stress relief cycles of 284°F. for 24 hours and 338°F. for 24 hours, as well as the 20 cycles of 70°F. to 700°F. in this thesis test program.

Residual stress measurements were made by Lenoe (1967b) by machining off one side of a composite and then measuring the amount of force required to straighten out the warped composite. The test data for four samples for various thicknesses are shown in Figure 4.

2.6 Heat Treatment and Aging

Shimizu and Dolowy (1969) of The Marquardt Corporation reported on various thermal treatments and their effect on transverse strength. The study showed that the highest transverse strength was in the range of 12 to 16 KSI for the T6 treatment; however, it was felt that the data were not representative of the true capability of the composite.

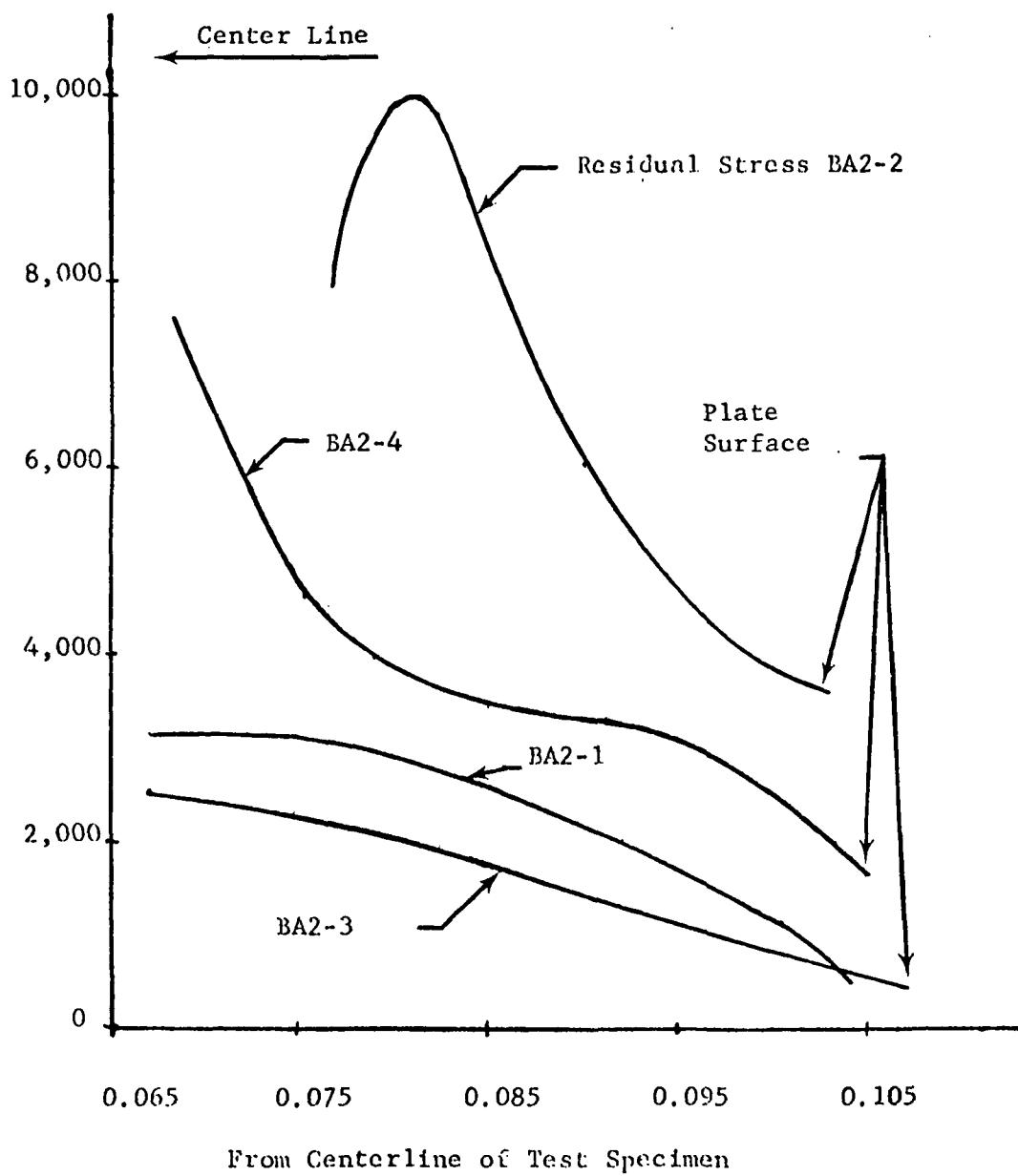


Figure 4. Residual Stress Versus Thickness (Lenoe, 1967b)

This was due to the sensitivity of transverse tests to edge effects, of splits in the fiber ends due to fabrication.

Swanson and Hancock (1971) also evaluated various heat treatments for 30 v/o boron-7075 aluminum and found that the T6 condition produced the highest UTS. The test data are shown in Figure 5; however, the transverse specimens failed by longitudinal splitting of the filaments.

Hanby (1971b) reported on some very recent research conducted at Midwest Research Institute that evaluated various heat treatments and their effect upon transverse tensile properties of 7075 aluminum matrix with 30 v/o boron filaments. These data indicated that the standard T6 treatment produced the highest strength as shown in Figure 6 although fractures contained excessive boron filament splitting, suggesting that the composite's transverse strength was limited by the properties of the filaments (splitting).

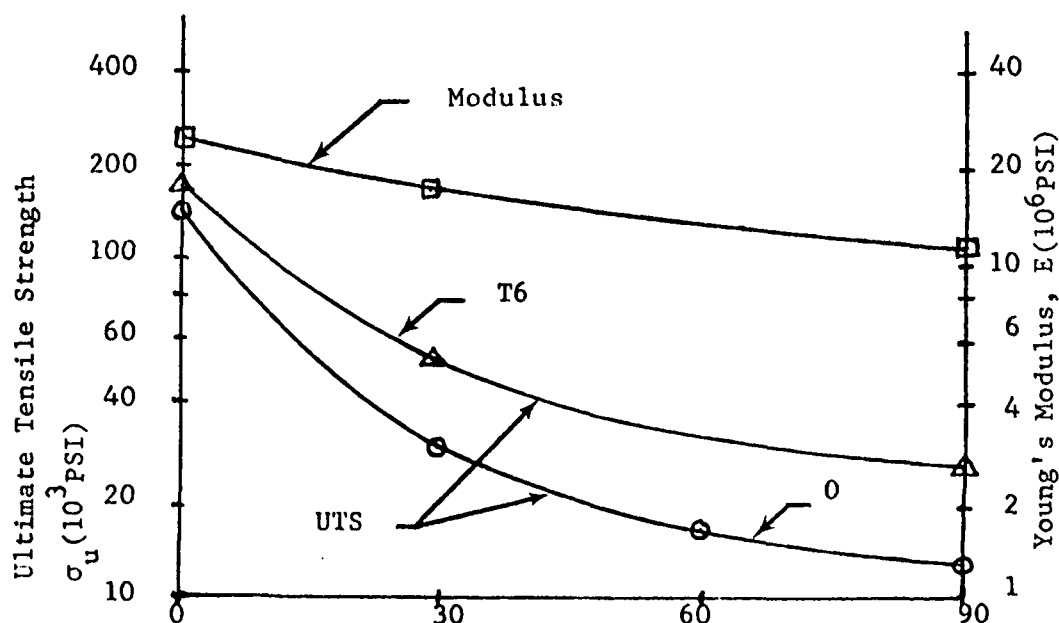


Figure 5. Filament Orientation vs. Ultimate Tensile Strength and Young's Modulus for Composite Specimens of 7075-T6 and 7075-0 Aluminum-Boron (Swanson and Hancock, 1971)

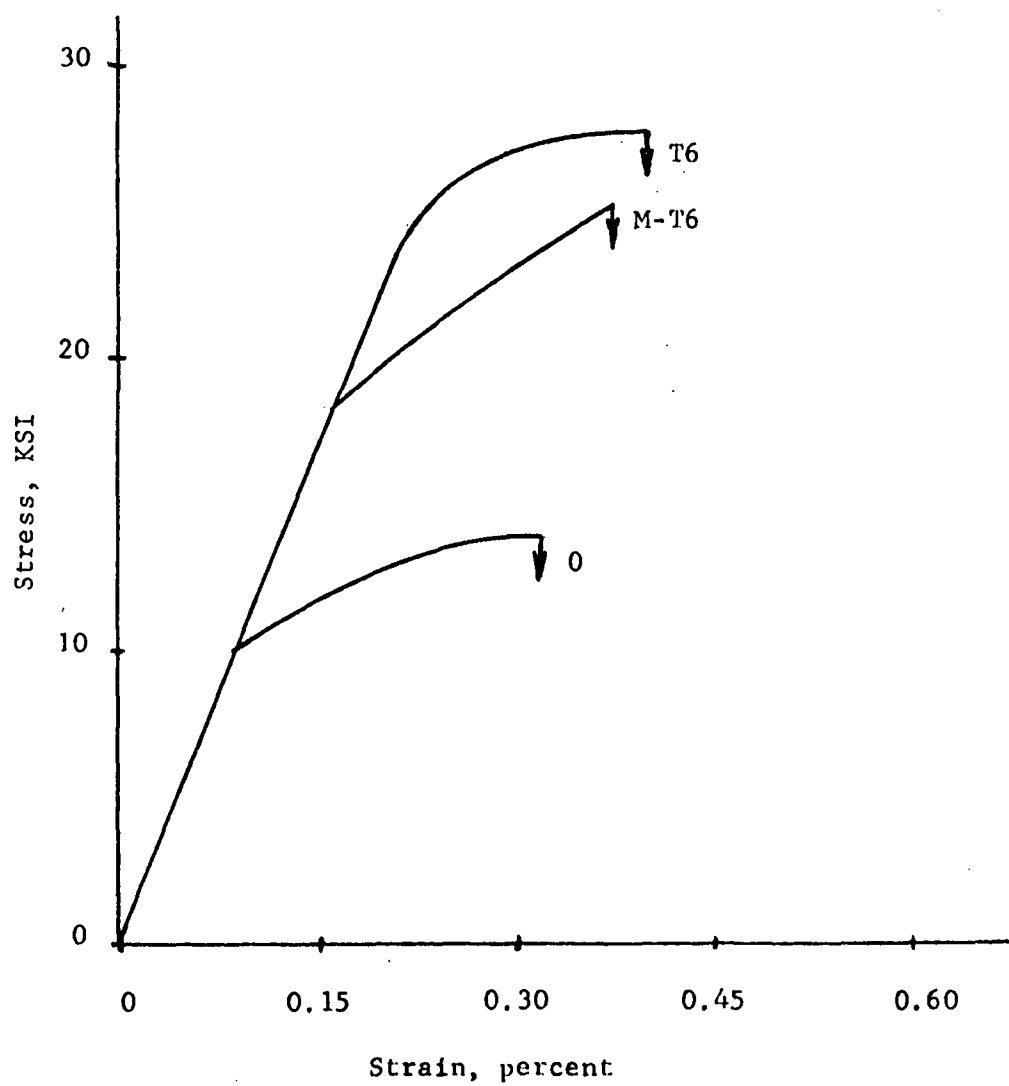


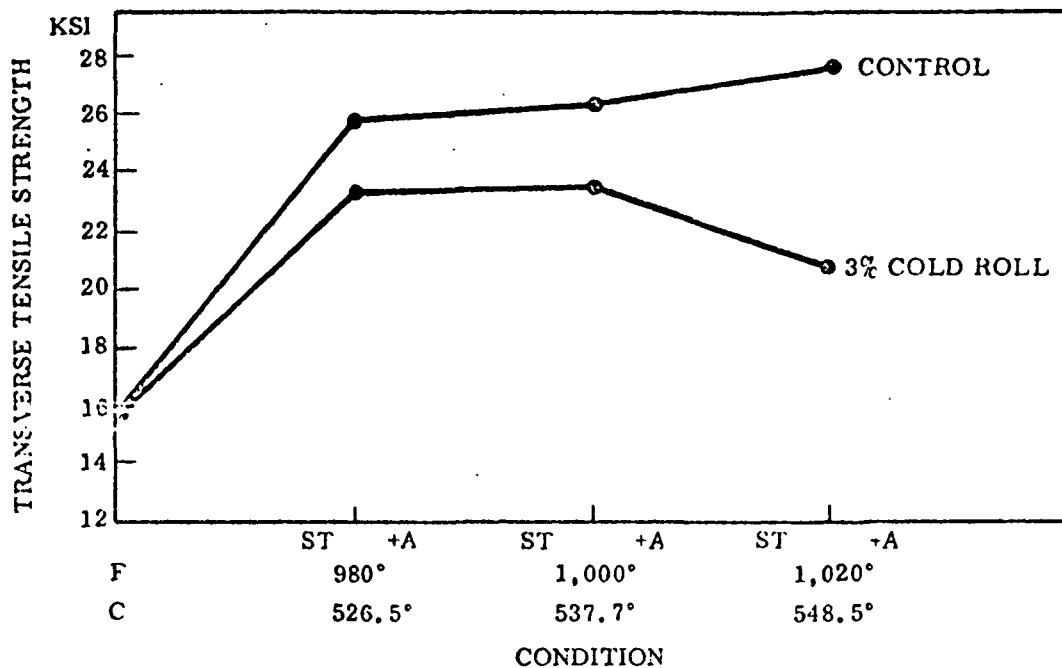
Figure 6. Effect of Heat Treatment on Representative Stress-Strain Curves for 7075 Aluminum/30 Vol % Boron Composite Tested Transversely to the Fiber Direction (Hanby, 1971b)

Many of the other reports in the literature survey reported herein evaluated the effect of T4 and T6 heat treatment and aging procedures on the transverse tensile strength. In all cases, the T4 and T6 conditions were reported to be beneficial, so that these treatments were included in this experiment.

2.7 Cold Rolling

A 10% reduction by cold rolling transverse to the filaments was reported by Taylor, et al. (1969) to show an increase in transverse strength. Christian (1969) reported on test data where 3% transverse cold rolling decreased transverse tensile strength of 6061 alloy composite as shown in Figure 7. Christian also reported that 5-6% of cold working on this composite structure resulted in matrix crazing. Dolowy and Taylor (1969) described transverse 10% cold rolling of 6061 matrix-boron composite that increased the longitudinal tensile strength by 30 KSI; however, the effect upon transverse strength was not described. Another report of the effect of transverse cold rolling upon longitudinal tensile strength was reported by Getten and Ebert (1969) with the test results plotted as shown in Figure 8.

Dolowy (1969a) reported that although transverse 10% cold rolling increased longitudinal ultimate tensile strength by 10%, the transverse ultimate tensile strength was reduced for the 6061 matrix composite. Forest (1968) of Convair reported that composites obtained from both Marquardt and Harvey Aluminum Corporation showed cold working was actually deleterious to transverse strength.



ST = Solution heat treating at the subscript temperature

(i.e. 980°F, 1000°F, or 1020°F; 526.5°C, 537.7°C, 548.5°C) for 30 minutes

Aging (350°F, 176.7°C) for 8 hours).

Figure 7. Effect of Cold Working on Transverse Tensile Strength

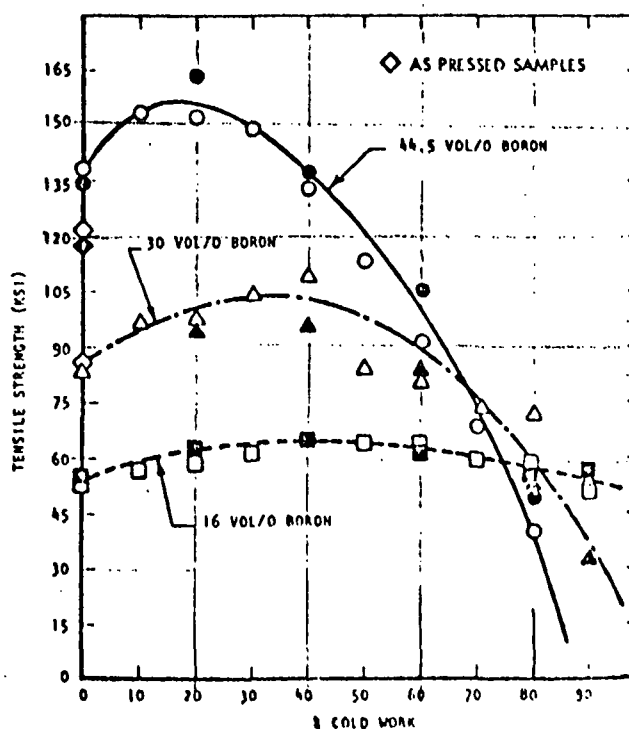


Figure 8. The Effect of Transverse Rolling on the Tensile Strength of Aluminum-Boron Composites (Open Points: 10% Reduction per Pass--Closed Points: 20% Reduction per Pass)

A review of the available data of the effect of transverse tensile strength of transverse cold rolling seemed to indicate that this thesis test program should evaluate at least 5% and 10% transverse cold rolling.

2.8 Pre-stretching of Filaments

A review of the literature in regard to research in pre-stretching the filaments during composite fabrication indicated that no effort has been exerted to evaluate this variable. Since the pre-stretching is a common procedure used in pre-stressed concrete, it would seem logical that the same pre-stretching would be beneficial to aluminum matrix composites. However, although this technique is within the realm of fabrication of composites, it was not considered for this test program since only post-fabrication variables were to be evaluated in this program.

2.9 Stress Relief by Stress Cycling

No reference could be found in the literature for metal matrix composites where stress cycling had been evaluated as a method for stress relief. This method, however, has been used for metals and it was felt that this method may be beneficial to transverse tensile strength of the aluminum-boron composite materials to be tested in this thesis. A stress cycling of 10% of the ultimate tensile strength (transverse) was used in this thesis with ten cycles applied from no load to 10% of the UTS, prior to the specimens being tested to failure.

2.10 Matrix-to-Fiber Bond

No reference could be found in the literature where matrix-to-fiber bond was evaluated in relation to transverse strength properties. Failure mode up to this time has been splitting of the fibers where the splits propagated into the aluminum matrix causing premature failure of the matrix. However, with the new glass core and carbon monofilament core filaments, as well as the new 5.6-mil-diameter filaments, relatively few splits of the boron is experienced. Therefore, higher transverse strengths have been obtained from the matrix. Even with the higher strengths, no mention has been found of bond failures between the matrix and the filaments.

There have been several reports in the literature evaluating the chemical reaction between the aluminum matrix and the boron filaments associated with high-temperature exposure. The intermetallic compounds found at the matrix-fiber interface, degrade the bond strength of the matrix to the filament. These reports have attempted to identify the intermetallic compounds, degree of boron attack or decomposition, and use of coatings such as silicon carbide on the boron filament to decrease the filament-matrix reaction. However, there were no data found in the literature search relating transverse tensile strength to bond strength of the matrix to the filament.

It was decided that this bond factor was not to be within the scope of this thesis program unless the bond factor became a prominent mode of failure in the tests to be performed.

2.11 Addition of Transverse Fibers

The literature search disclosed that several programs had evaluated the effect of adding a small percentage (5%) of stainless steel filaments in the transverse direction to increase transverse tensile strength. Christian (1969) discussed the usage of 5% by volume of AM-355 stainless steel wire (cross-ply) in 6061 aluminum matrix. These composites produced data as shown below in Table 2, with the transverse strength approximately twice that reported by Christian (1969).

Dolowy (1969b) reported also about double the transverse tensile strength to 30-43 KSI with the addition of 5% stainless steel transverse wire.

Again, this factor of adding transverse filaments or wire to increase transverse tensile strength is a factor involved in fabrication of the composites and therefore was not considered to be within the scope of this thesis. This thesis evaluated only post-fabrication variables that affect transverse properties.

2.12 Summary of Literature Search

The foregoing literature survey was developed in some detail in an effort to take in the wide scope of experimental work carried out in the area of transverse properties of boron-filament-reinforced-aluminum composites. In the course of sorting out the many approaches used by the investigators, it became evident that there was no common agreement about which approaches produce the best results.

Table 2. Mechanical Properties of Al-B-SS Composites

Condition	Long. Tensile		Trans. Tensile			Shear F_{su} (KSI)
	F_{tu} (KSI)	E(MSI)	F_{tu} (KSI)	F_{ty} (KSI)	E(MSI)	
<u>Al-35B-5SS:</u>						
F	115(80.8)	22.5(15,820)	42.7(30.0)	19.1(13.5)	11.5(8,080)	24.2(17.0)
ST&A	124(87.1)	23.3(16,380)	41.3(29.0)	29.3(20.6)	13.9(9,770)	22.6(15.9)
<u>Al-45B-5SS:</u>						
F	175(123)	29.7(20,880)	36.2(25.4)	15.0(10.5)	14.2(9,980)	18.4(12.9)
ST&A	159(112)	31.3(22,000)	32.1(22.5)	23.0(16.2)	15.8(1,110)	19.2(13.5)

NOTE: All figures in parentheses are kgf/mm^2 .

This was particularly true since some test results contradict results from other investigators. Thus, at this juncture, it appears that all the post-fabrication approaches should be investigated in this thesis within the limits of the material available. There were several analytical studies made of composites and transverse strength properties such as that by Chen and Lin (1968) and Ebert (1970). However, an analytical study of composites was not within the scope of this test effort and no analysis was made of the analytical studies.

CHAPTER 3

OBJECTIVES OF THIS INVESTIGATION

The general purpose of this investigation was to study the transverse physical properties possessed by boron-filament-unidirectionally-reinforced-aluminum-matrix composites with various post-fabrication treatments to determine the nature of the relationship between the post-treatments and the behavior resulting from these treatments.

The detailed objectives of this study were:

1. To determine the effectiveness of a new transverse tensile specimen preparation technique first reported by Kreider, et al., (1970) improving transverse strength. This technique involves the freeing of the filament ends at the edges of the specimen by chemically etching the aluminum away from the machined edges. This technique reduces the possibility of cracks at the ends of the filaments (created during specimen machining) propagating into the reduced gage length section of the specimen.

2. To evaluate observed behavior of the transverse strength properties with the post-treatments of solution heat treatment, natural and artificial aging, cold-rolling, thermal stress relief cycles, and strain cycling for stress relief. These processes were evaluated in an effort to improve the transverse strength properties of the composites.

3. To evaluate the new 5.6-mil-diameter boron filaments reported to produce transverse tensile specimens that do not fail prematurely from filament splitting.

CHAPTER 4

THEORETICAL CONSIDERATIONS RELATED TO TRANSVERSE STRENGTH IMPROVEMENT

The two most commonly used aluminum alloys are 6061 and 2024; therefore, these alloys were evaluated in this thesis. These alloys have nominal compositions as indicated in Table 3.

Table 3. Alloy Compositions

Alloy	Si	Cu	Mn	Mg	Cr	Al
6061	0.6%	0.25%	--	1.0%	0.25%	97.9%
2024	0.5%	4.5%	0.6%	1.5%	0.1%	92.8%

The 6061 alloy is popular since it is characterized by excellent corrosion resistance and is more workable than other heat-treatable alloys. The 2024 alloy develops the highest strengths of any naturally aged aluminum-copper alloy.

4.1 Precipitation Hardening Process

The general principle of precipitation hardening is to make use of the supersaturation condition that exists when a solid solution is preserved by a rapid cooling process. This is prominently encountered with low carbon steel, beryllium copper, precipitation hardening steels,

and most prominently with many of the aluminum alloys. Supersaturation can only be induced when the phase diagram has a solid solubility line that has a significantly positive slope, that is, solubility increases with increasing temperature. The solid solubility line separates the single-phase (α) region from the two-phase region of the phase diagram.

Since the matrix materials in this thesis are two precipitation hardening aluminum alloys, the precipitation hardening process will be defined specifically for these materials. The precipitation hardening process is divided into three basic steps as follows:

- a. Solution heat treatment step--In this process, it is necessary to heat the alloy into the solid solution temperature range for a time long enough to permit solid state diffusion processes to occur and produce a completely homogeneous solid solution. It is imperative that the solution heat treatment temperature never exceeds the eutectic temperature to prevent melting of the eutectic that may be present along the grain boundaries. This is known in industrial heat treating as "burning the alloy." The solution heat treatment temperature for 6061 alloy is 985°F. and for 2024 alloy is 920°F. Times required for this process are a function of the thickness of the material, and the inherent diffusion coefficients of the alloying elements.
- b. Preservation of a homogeneous solid solution--The state of supersaturation is produced by rapidly cooling (water quench) the solid solution alloy to prevent precipitation of the now-excess insoluble phase.

c. Aging to enhance mechanical properties--Aging may be defined as a heat treatment of the supersaturated alloy that utilizes a temperature/time combination sufficient to precipitate the critical size sub-microscopic particles that produce optimum properties of strength and ductility. Currently accepted theory explains this hardening and strengthening phenomenon as a result of the formation of Guinier-Preston Zones along the $\{100\}$ planes (two-dimensional platelets one atom thick and 30 to 50 Angstroms in diameter for GP [1] zones and several atoms thick for three-dimensional GP [2] zones), (Van Horn, 1967). Dislocation theory accepts the Guinier-Preston theory and explains the optimization of strength and hardness by the impeding of dislocation movement when the limiting radius of the dislocation loop is equal to the distance between zones (approximately 100 Ångstroms). In order for the dislocation to progress through the aluminum lattice, it is necessary for the dislocation to shear the zone, that is, to overcome the elastic strain energy produced in the aluminum lattice by the coherent platelets of foreign atoms (precipitate as a Guinier-Preston Zone); or, it is necessary to glide by overcoming the interaction energy of the zone.

In the case of the 2024 alloy, ordinary room temperature (70° - 90° F.) is high enough to permit precipitation to occur at a significant rate. This condition is described as 2024-T4 and is representative of the "natural aging" process. In the 2024 alloy, the magnesium addition accelerates and intensifies the natural aging and the

precipitation zones are believed to consist of groups of magnesium and copper atoms. The apparent acceleration of the natural aging by the addition of magnesium may result from complex interactions between vacancies and the two solutes.

In the case of the 6061 alloy, it is necessary to "artificially age" the alloy at elevated temperature in order to achieve optimum precipitation conditions. In artificial aging (heat treatment) of the 6061 alloy to obtain the T-6 condition, a temperature cycle of 320°F. for 18 hours is utilized. In artificial aging of the 2024 alloy to obtain the T-6 condition, a temperature cycle of 375°F. for 9 hours is utilized.

4.2 Cold Rolling Process

Metals and alloys can be strengthened by cold working (strain hardening) below the recrystallization temperature. Certain precipitation-hardenable alloys can be further strengthened by aging after cold working.

Work hardening causes the generation and multiplication of dislocations and the subsequent locking (impeding of movement) of these dislocations due to elastic interaction of the dislocation strain fields. Several of the locking mechanisms are as follows:

- a. There can be both interactions with strain fields of dislocations parallel to each other as well as interactions of the strain field of a dislocation with forest dislocations.
- b. There can be elastic interaction with stress fields of

piled-up groups of dislocations creating Cottrell-Lomer sessile dislocations.

c. There can be elastic interaction with stress fields of high energy dislocation networks and tangles.

d. There can be elastic interaction with "debris" produced by dislocation movement. Debris consists of edge dislocation dipoles and loops.

e. There can be energy required to form a jog at a dislocation intersection.

f. There can be energy required to form vacancies and interstitial atoms by non-conservative motion of jogs on screw dislocations.

The additional energy required to produce dislocation movement, as described above, is the major contributing factor for the increase of strength realized in strain hardening.

4.3 Thermal Stress Relief

Thermal stress relief is a heat treatment that tends to uniformly redistribute the stress fields thereby preventing premature failure that could occur if highly stressed local conditions existed. This is accomplished because the higher temperature permits localized movement of individual dislocations (movement starts and is most active at high stress centers). During the movement and migration of the dislocations, annihilation of dislocations, glide, movement along low angle grain boundaries, and relief of violent tangles (stress concentrations) is realized.

The basic cause of these high stress concentrations is due to the difference in the thermal contraction rates between the boron filaments and the aluminum matrix when the composite is cooled from the fusion temperature. The aluminum tries to contract more than the boron, and therefore, stress concentrations are established at the interface. The thermal stress relief therefore has a tendency to relieve these high stress centers at the bond interface between the boron filament and the aluminum matrix as well as at other points within the matrix.

4.4 Strain Cycling

The strain cycling process accomplishes the same stress relief as does the thermal stress relief process. However, instead of using elevated temperature to provide the energy to allow dislocation movement, this process uses small tensile strain cycling applications (10% of UTS) within the elastic range to provide the energy for the dislocation movement.

CHAPTER 5

EXPERIMENTAL PROCEDURE

In this investigation, tensile specimens of the aluminum-boron composites were prepared by shearing, cold-rolling or thermally treating, masking, chemical etching, and adhesive bonding. Eleven (11) different treatment conditions were chosen to provide the experimental data, and tests were performed on three specimens for each condition for both 25% and 50% boron in 6061 and 2024 aluminum alloy matrixes. The treatment conditions consisted of 5% and 10% cold rolling (T3) with and without additional heat treatment (T36), natural age hardening (T4), artificial age hardening (T6), two thermal stress relief procedures, one thermal-cycling-stress-relief procedure, and one strain-cycling-stress-relief procedure. The test conditions are tabulated in Table 4.

Tensile specimens of one-inch gage length were used throughout, thereby making the term "pulling speed" equivalent in magnitude to strain rate. Aluminum pads were epoxy adhesive bonded on each side of each end of the specimens so that grip areas were provided for the Instron serrated jaws.

5.1 Material

One panel 0.022 inch thick of each 6061 and 2024 aluminum alloy containing 25% by volume of boron filaments was obtained from Harvey Engineering Laboratories for Research and Development,

Table 4. Aluminum 6061-F Composite Test Results

Condition	Heat Treatment	Stress Relief	Percent Boron (Vol.)	Elongation at Break		Yield Strength		Ultimate Tensile Strength		Modulus of Elasticity x 10 ⁶		Comments of Fracture Appearance
				%	Ave	PSI	Ave	PSI	Ave	PSI	Ave	
Annealed (T-0)	None	None	25%	2.0 1.45 1.1	1.52	11,730 12,400 12,450	12,190	19,050 17,900 16,850	17,950	8.9 10.75 12.7	11.1	
			50%	0.45 0.32 0.60	0.46	9,800 5,680 11,800	9,090	11,200 6,300 13,000	10,200	15.25 11.55 12.25	13.0	
Annealed Soln. Treat	Age at R.T. for 4 days (T-4)	None	25%	1.4 1.33 1.52	1.42	23,600 25,600 22,750	23,980	28,800 30,500 29,500	29,600	12.90 14.70 13.10	13.6	
			50%	0.69 0.57 0.84	0.70	23,400 21,000 20,900	21,800	25,500 21,000 26,700	24,400	20.35 19.6 20.15	20.0	
Annealed Soln. Treat	320°F. 18 hrs. (T-6)	None	25%	1.45 1.40 1.37	1.42	40,800 39,600 31,500	37,300	40,800 39,600 31,500	37,300	14.9 15.0 12.2	14.0	One fiber split 100%.
			50%	0.92 0.48 0.55	0.65	33,400 15,800 18,900	22,700	33,400 15,800 18,900	22,700	19.25 17.50 18.20	18.3	One fiber split 90%. One fiber split 60%.
Annealed Soln. Treat	320°F. 18 hrs. (T-6)	284°F. 24 hrs.	25%	0.89 0.90 1.32	1.04	23,000 22,300 31,700	25,700	23,000 22,300 31,700	25,700	13.7 13.15 12.7	13.2	One fiber split 40%.
			50%	0.88 0.88 0.76	0.84	29,000 29,700 26,500	28,400	29,000 29,700 26,500	28,400	17.5 19.2 17.8	18.2	
Annealed Soln. Treat	320°F. 18 hrs. (T-6)	338°F. 24 hrs.	25%	1.20 1.30 1.30	1.27	33,400 32,800 36,100	34,100	33,400 32,800 36,100	34,100	14.7 13.35 14.7	14.2	
			50%	0.68 0.81 0.63	0.71	21,500 29,600 21,700	24,300	21,500 29,600 21,700	24,300	16.8 19.4 18.3	18.2	One fiber split 100%. No fiber splits. One fiber split 60%.

Table 4 (continued)

Annealed Soln. Treat	320°F. 18 hrs. (T-6)	20 cycles 70°F. to 700°F.	25%	1.50 1.29 1.70	1.52	12,300 13,500 12,800	12,900	18,200 18,900 20,200	19,100	10.85 11.9 11.65	11.5	
			50%	0.69 0.69	0.69	7,250 8,160	7,705	10,550 11,400	10,980	12.0 11.8	11.9	
Anneal Soln. Treat Cold Roll 5% (T-3)	None	None	25%	1.42 1.11 1.70	1.41	28,000 21,600 26,600	25,400	32,200 26,200 34,300	30,900	12.55 12.7 12.8	12.7	One fiber split 15%. One fiber split 80%. No fiber splits.
			50%	0.71 0.79 1.05	0.85	23,900 27,400 24,500	25,300	25,000 31,000 32,700	29,600	18.9 23.4 18.5	20.3	One fiber split 25%. No fiber splits. No fiber splits.
Anneal Soln. Treat Cold Roll 5%	320°F. 18 hrs. (T-36)	None	25%	1.67 1.10 1.41	1.39	44,300 30,800 39,900	38,300	44,300 30,800 39,900	38,300	14.05 15.3 15.0	14.8	One fiber split 15%. No fiber splits.
			50%	0.50 0.88	0.88	14,300 29,200	29,200	14,300 29,200	29,200	15.15 17.5	17.5	One fiber split 50%. No fiber splits.
Anneal Soln. Treat Cold Roll 10% (T-3)	None	None	25%	1.22 0.90 1.02	1.05	26,300 16,950 23,000	22,100	28,900 18,400 24,700	24,000	13.05 11.5 13.4	12.7	No fiber splits. No fiber splits. No fiber splits.
			50%	0.70 0.65 0.65	0.67	16,200 17,700 18,900	17,600	16,200 17,700 18,900	17,600	12.3 14.4 15.4	14.0	No fiber splits. One fiber split 30%. No fiber splits.
Anneal Soln. Treat Cold Roll 10%	320°F. 18 hrs. (T-36)	None	25%	0.98 1.07 0.98	1.01	24,350 24,400 24,600	24,450	24,350 24,400 24,600	24,450	13.2 12.1 13.3	12.9	
			50%	0.60 0.80	0.70	17,800 21,900	19,800	17,800 21,900	19,800	15.7 14.5	15.1	No fiber splits. One fiber split 40%.

a division of Harvey Aluminum, Inc. Sections 1-1/2 inches by 4 inches were sheared from these panels with the filaments in the 1-1/2-inch direction. These sections then were processed by solution treating, cold-rolling, age hardening, or stress relief as required.

5.2 Tensile Specimen Fabrication

Test specimens 1/2 inch by 4 inches were sheared from each of the conditioned sections. For specimens to contain 50% by volume filaments, the thickness of the material was chemically etched from the original 0.022-inch thickness to approximately a 0.013-inch thickness, only in the necked down portion of the specimen (the grip areas were masked with Scotch No. 56 Mylar Masking Tape). Difficulty was experienced with this etching operation since some of the specimens (particularly the 2024 specimens) had a tendency to pit and etch unevenly. The original etchant was composed of four volumes HCl, one volume HF, and twelve volumes of water. The etchant was changed to a 0.1 normal NaOH solution. However, the aluminum pitted just as badly with this alternate etchant. For this reason, much of the 50%-boron-composite strength data are lower than expected.

The next specimen operation in fabrication consisted of masking the reduced-width section of the specimens. It was found that a Teflon pressure-sensitive tape was adequate for masking specimens which were not heat treated. However, the hardened aluminum alloys required longer etching times and this increased the tape exposure time in the etchant. The tape adhesive could not resist these longer etching times. An Eastman-Kodak photo-sensitive maskant, Photo-Resist

Type 3, was utilized to provide adequate masking for the long etching times (as long as 45 minutes). The panels which contained 50 volume percent boron (where the surfaces were etched to the 0.0135-inch thickness) were so rough that two coats of the Photo Resist were required for protection. The steps in the fabrication are described in Figure 9. The completed test specimen is shown in Figure 10.

5.3 Metallographic Examination

During various phases of the fabrication of test specimens and after tensile tests, sample pieces were taken from the composite material and from tensile specimens for microscopic examination and evaluation of microstructures. These sample cross-sections are shown in Figure 11. There was no visual indication of deterioration of the boron filaments with long heat exposures. There was some difficulty in grinding and polishing these metallographic samples due to the relative softness of the aluminum compared to the hardness of the boron. The aluminum had a tendency to be undercut from the surface of the boron, and the boron had a tendency to propagate cracks or create new cracks during the grinding and polishing. One sample was ground down 1/4 inch from the initial polished surface but the cracking tendency persisted.

5.4 Measurement of Tensile Strength

The tensile testing was performed with an Instron Table Model No. 1130 Universal Testing Machine, using a 1000 pound load cell. Initially to determine what strain rate should be used, tensile tests

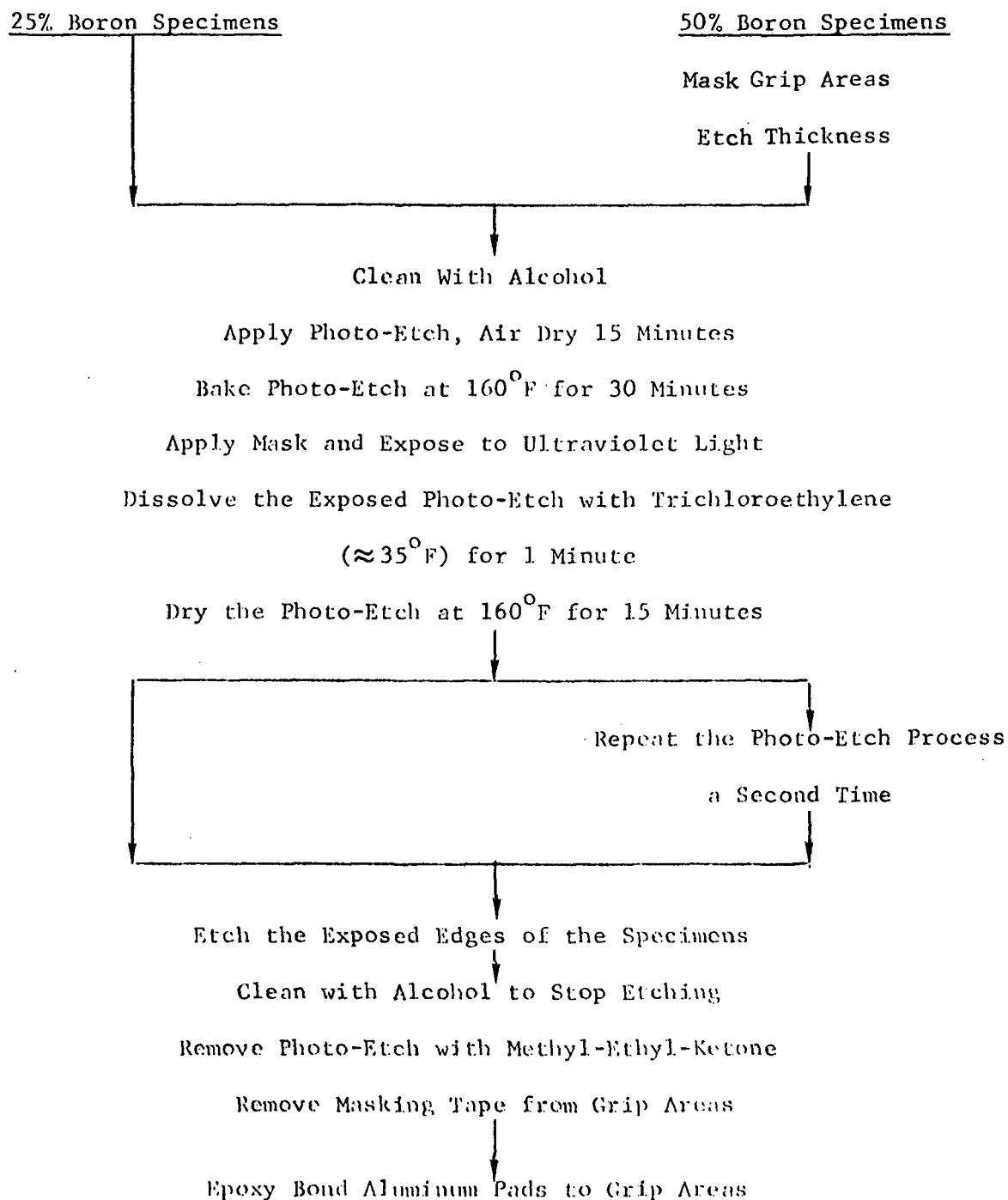


Figure 9. Flow Chart of Processes Performed to Fabricate Tensile Test Specimens

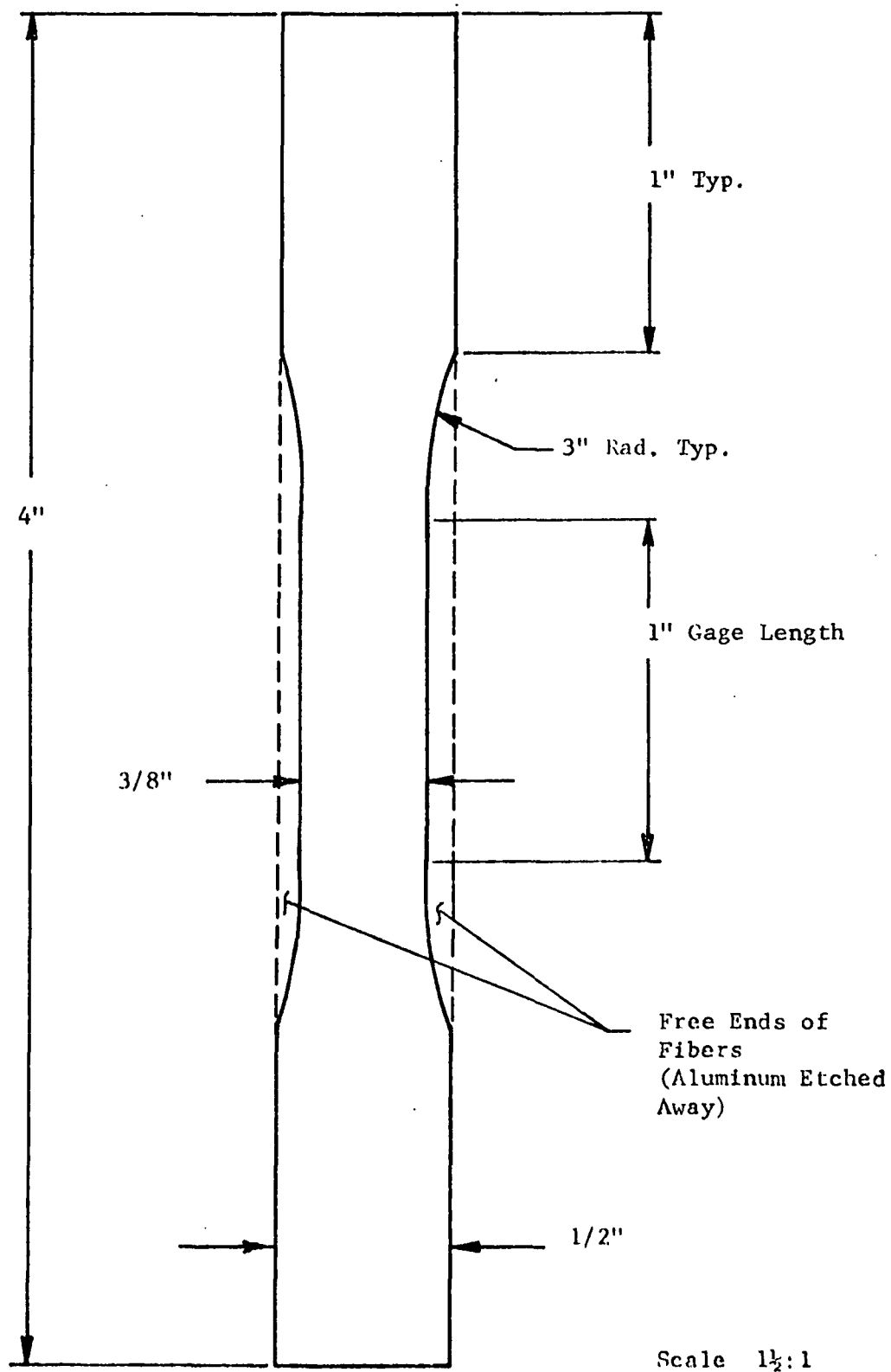


Figure 10. Configuration of Typical Tensile Specimen

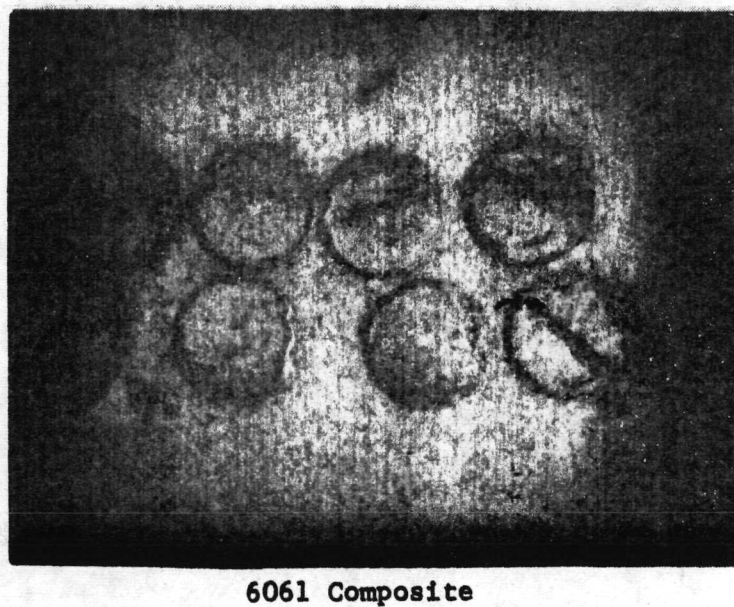
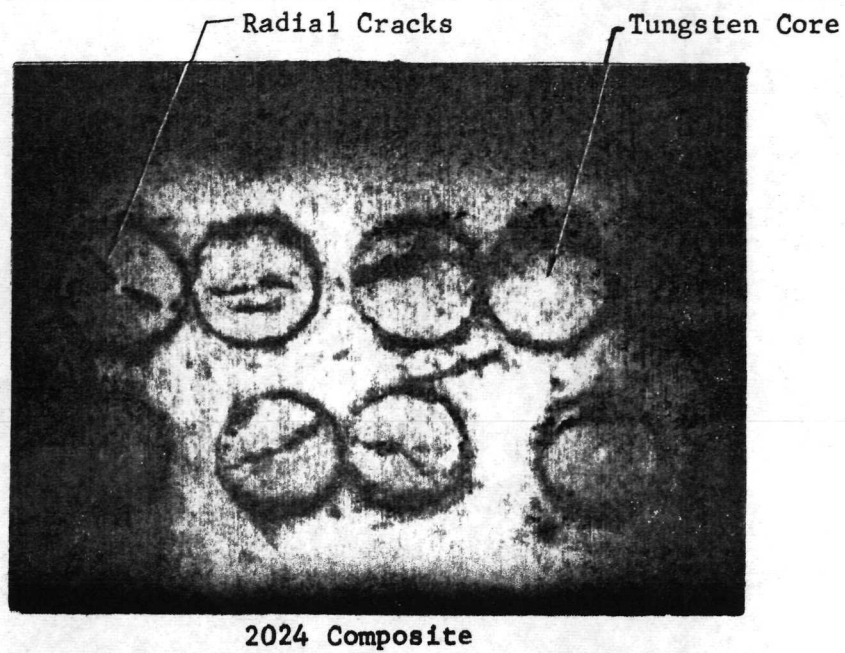


Figure 11. Photomicrographs of Composite Cross-sections
≈ 100X.

were performed on both 6061-F and 2024-0 test specimens (all 25% boron in the as-received condition) using strain rates of 2, 0.2, 0.05, 0.02, 0.01, and 0.005 using an Instron Model No. TT-C Universal Testing Machine. The percent elongation did not essentially change regardless of which strain rate was used. A strain rate of 0.2 in/min. was selected since this was the slowest rate that could be performed with the Model 1130 Instron. The test data for this initial strain-rate investigation are tabulated in Table 5.

Table 5. Strain Rate Evaluation

Strain Rate In/Min	Yield Strength, KSI		Ultimate Strength, KSI		Percent Elongation, %	
	6061-F	2024-0	6061-F	2024-0	6061-F	2024-0
2.0	11.80	-	11.80	-	1.50	-
0.2	12.70	15.15	16.70	23.40	1.44	2.00
0.05	13.35	20.00	14.95	21.65	1.25	0.95
0.02	13.25	21.40	14.90	24.90	1.38	1.12
0.01	12.50	15.15	16.95	22.05	1.48	1.09
0.005	11.70	13.35	16.00	23.80	1.25	1.40

All further testing was performed on the Model 1130 Instron, using 0.2 inches/minute loading rate and 20 inches/minute chart speed, with the 1000 pound load cell set for 500 pounds full scale. Elongation was measured by the distance measured on the chart times the ratio of the cross-head travel speed divided by the chart speed which then produced elongation in terms of in/min since the specimen gage length was one inch.

CHAPTER 6

RESULTS AND DISCUSSION

The results obtained in this investigation are presented and discussed in the following order:

First, the directly-observed values of percent elongation, yield strength, ultimate tensile strength, and modulus of elasticity are presented.

Second, visual observation of the fractures is described, since several modes of failure can be present, such as filament splitting, unbonding at filament-matrix interface, and matrix failure.

Third, a correlation is then made of the test results and the treatment conditioning used on the various specimens.

6.1 Test Data Results

For each test specimen, the percent elongation at fracture, the yield strength, the ultimate tensile strength, and the modulus of elasticity were calculated. The calculated results were then tabulated and are shown in Table 4 for the 6061 composite and in Table 6 for the 2024 composite.

The modulus data were found to be below the expected range by a factor of about five, due probably to the elongation of the epoxy adhesive used to bond the aluminum pads in the grip areas of the specimens.

Table 6. Aluminum 2024-0 Composite Test Results

Condition	Heat Treatment	Stress Relief	Percent Boron (Vol.)	Elongation at Break		Yield Strength		Ultimate Tensile Strength		Modulus of Elasticity $\times 10^6$		Comments of Fracture Appearance
				%	Ave	PSI	Ave	PSI	Ave	PSI	Ave	
Annealed (T-0)	None	None	25%	1.55	1.31	18,600	18,600	29,000	28,600	12.5	13.3	
				1.35		19,900		33,500		12.9		
				1.02		17,200		23,400		14.5		
			50%	1.05	0.85	21,700	20,800	28,200	24,100	16.4	16.5	One fiber split 80%.
				0.71		20,800		22,100		17.0		
				0.78		19,800		22,100		16.1		
Soln. Treat	Age 4 days at RT. (T-4)	None	25%	1.55	1.59	48,900	47,400	48,900	47,400	16.7	15.8	
				1.62		47,700		47,700		15.6		
				1.60		45,500		45,500		15.1		
			50%	0.90	1.05	33,300	35,700	33,300	35,700	19.6	18.1	
				1.09		32,400		32,400		15.7		
				1.15		41,300		41,300		19.1		
Soln. Treat	375°F. 9 hrs. (T-6)	None	25%	1.92	1.79	53,500	49,300	53,500	49,300	14.7	14.6	One fiber split 50%.
				1.57		44,400		44,400		15.0		
				1.88		49,900		49,900		14.0		
			50%	0.77	0.85	27,200	31,000	27,200	31,000	18.8	19.3	One fiber split 90%. One fiber split 100%.*
				0.97		36,300		36,300		19.9		
				0.81		29,400		29,400		19.3		
Soln. Treat	375°F. 9 hrs. (T-6)	284°F. 24 hrs.	25%	1.40	1.49	41,800	47,300	41,800	47,300	15.9	16.8	
				1.48		47,000		47,000		16.9		
				1.60		53,000		53,000		17.7		
			50%	0.98	0.89	35,500	34,800	35,500	34,800	19.3	20.8	
				0.80		32,800		32,800		21.7		
				0.89		36,100		36,100		21.4		
Soln. Treat	375°F. 9 hrs. (T-6)	338°F. 24 hrs.	25%	1.22	1.19	40,600	38,300	40,600	38,300	17.6	17.0	
				1.17		38,200		38,200		17.3		
				1.20		36,100		36,100		16.1		
			50%	0.93	1.06	35,100	40,100	35,100	40,100	20.0	20.2	
				1.19		44,100		44,100		19.7		
				1.05		41,000		41,000		20.8		

* Fiber split in neckdown section but not in etched areas at ends, indicating no end splits were present before tensile test.

Table 6 (continued)

Soln. Treat	375°F. 9 hrs. (T-6)	20 cycles 70°F. to 700°F.	25%	1.88 1.50 1.90	1.76	15,200 16,500 14,500	15,400	24,800 24,800 25,100	24,900	14.4 13.5 12.8	13.6	
			50%	1.14 0.88 1.35	1.12	14,500 15,700 15,700	15,300	23,950 21,600 24,100	23,200	14.0 16.7 14.6	15.1	
Soln. Treat Cold Roll 5% (T-3)	None	None	25%	1.67 1.42 1.38	1.49	23,400 25,800 27,400	25,500	31,200 30,600 29,700	30,500	11.5 12.0 11.6	11.7	
			50%	0.88 0.63 0.84	0.78	20,600 16,850 20,600	26,000	24,200 16,850 22,700	21,300	15.6 14.2 15.4	15.1	No fiber splits. One fiber split 100%. No fiber splits.
Soln. Treat Cold Roll 5% (T-81)	375°F. 12 hrs. (T-81)	None	25%	1.64 1.53 1.70	1.62	38,400 39,000 44,500	40,600	42,000 39,000 46,000	42,300	14.4 14.3 14.7	14.5	
			50%	0.70 0.85 0.71	0.75	20,200 25,700 22,600	22,800	20,200 25,700 22,600	22,800	15.3 16.1 16.9	16.1	
Soln. Treat Cold Roll 10% (T-3)	None	None	25%	1.50 1.52 1.62	1.55	35,000 39,900 40,400	38,400	35,000 39,900 40,400	38,400	12.3 13.9 13.2	13.1	
			50%	0.57 0.72	0.64	15,600 19,100	17,300	15,600 19,100	17,300	14.5 14.1	14.3	
Soln. Treat Cold Roll 10% (T-81)	375°F. 12 hrs. (T-81)	None	25%	1.38 1.38 1.38	1.38	35,200 33,300 32,300	33,600	35,200 33,300 32,300	33,600	13.5 12.8 12.4	12.9	
			50%	**								
Soln. Treat	375°F. 12 hrs.	Stretch Cycle Ten Cycles 10% UTS	25% (Stretch 350 lbs.)	1.63 1.41 1.42	1.49	50,500 38,600 27,600	38,900	50,500 40,000 34,000	41,500	16.4 15.6 13.6	15.2	Both fibers split 50% & 100%.
			50% (Stretch 15 lbs.)	1.08 0.83 0.75	0.89	40,400 32,800 29,000	34,100	40,400 32,800 29,000	34,100	19.8 21.0 20.5	20.4	One fiber split 100%. Both fibers split 50%. One fiber split 100%.

** All specimens were ruined due to severe pitting during chemical etching of thickness.

To support this hypothesis, aluminum specimens were machined from 6061-T4 (.025 mil thick) and 2024-T3 (.032 mil thick) and aluminum pads were epoxy adhesive bonded to them in the same manner used for the composite test specimens. Tensile tests performed using identical procedures as those used for testing the composite specimens, produced the data shown in Table 7.

Table 7. Modulus of Elasticity of Aluminum Specimens with Epoxy Bonded Grips

Material	Modulus of Elasticity	Average Modulus of Elasticity
6061 (.025" thick)	1.9×10^6	1.92×10^6
	1.94×10^6	
2024 (.032" thick)	1.88×10^6	1.86×10^6
	1.83×10^6	

Comparing the modulus measured on the aluminum specimens to the known modulus of 10.0×10^6 psi, it is shown that a ratio of 5.3 is needed to correct the modulus values determined for the composite specimens, because of the elongation in the epoxy. The modulus values in the graphs are corrected by this ratio. It was unfortunate that an extensometer was not available to attach directly to the specimens.

Photographs of the tensile test specimens are shown in Figure 12. The top photograph shows a completed test specimen with the aluminum

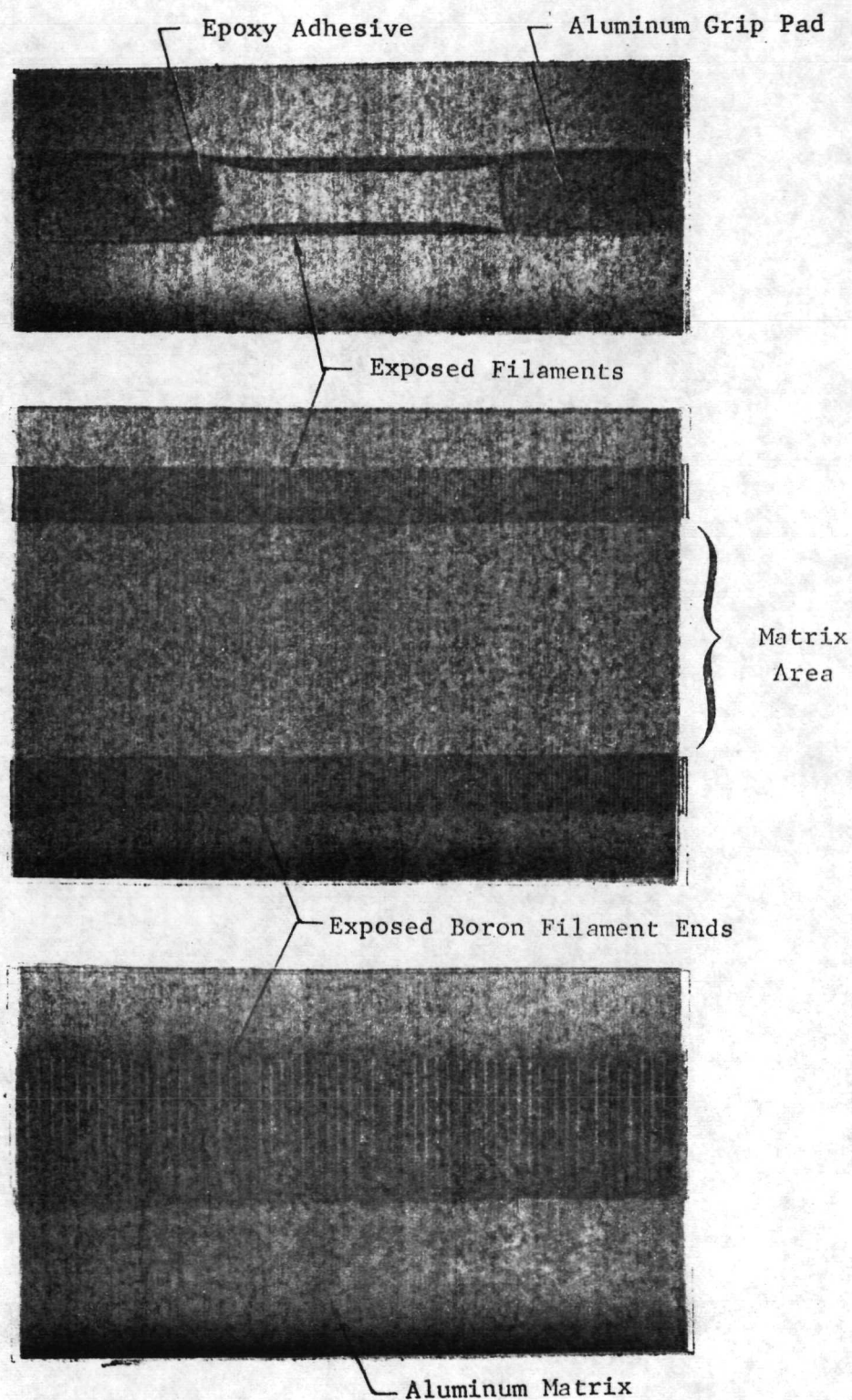


Figure 12. Photographs of Tensile Test Specimen

matrix etched away from the boron filament ends in the area of the gage length. The top photograph also shows the epoxy bonded aluminum pads used for the serrated test jaws of the Instron test machine that prevented jaw teeth damage to the composite. The other two photographs in Figure 12 show enlarged views of the test specimen.

6.2 Effect of Thermal Conditioning

The effects of the various aging, heat treatment, and thermal stress relief procedures on the boron reinforced aluminum composites, are shown in Figure 13 for yield strength and in Figure 14 for ultimate tensile strength for 2024 matrix. In both the yield strength as well as the ultimate tensile strength, the standard T6 treatment of the 25%-filament alloy produced the highest strengths, with the thermal cycling of 70°F. to 700°F. producing the lowest strengths. The lower T6 strengths shown by the 50% boron specimens are believed due to the pitting problem experienced during chemical etching of the specimen thickness. Figure 15 shows the yield strength for 6061 matrix and Figure 16 shows the ultimate tensile strength for 6061 matrix, and again the standard T6 treatment produced the highest strengths of all the treatments tested.

A plot of the transverse yield strength and tensile ultimate strength versus percent filament reinforcement for 2024-T6 aluminum matrix is shown in Figure 17. A similar plot for 2024-T4 is shown in Figure 18. For the aluminum matrix of 6061-T6, a plot of the transverse yield strength and tensile ultimate strength versus percent

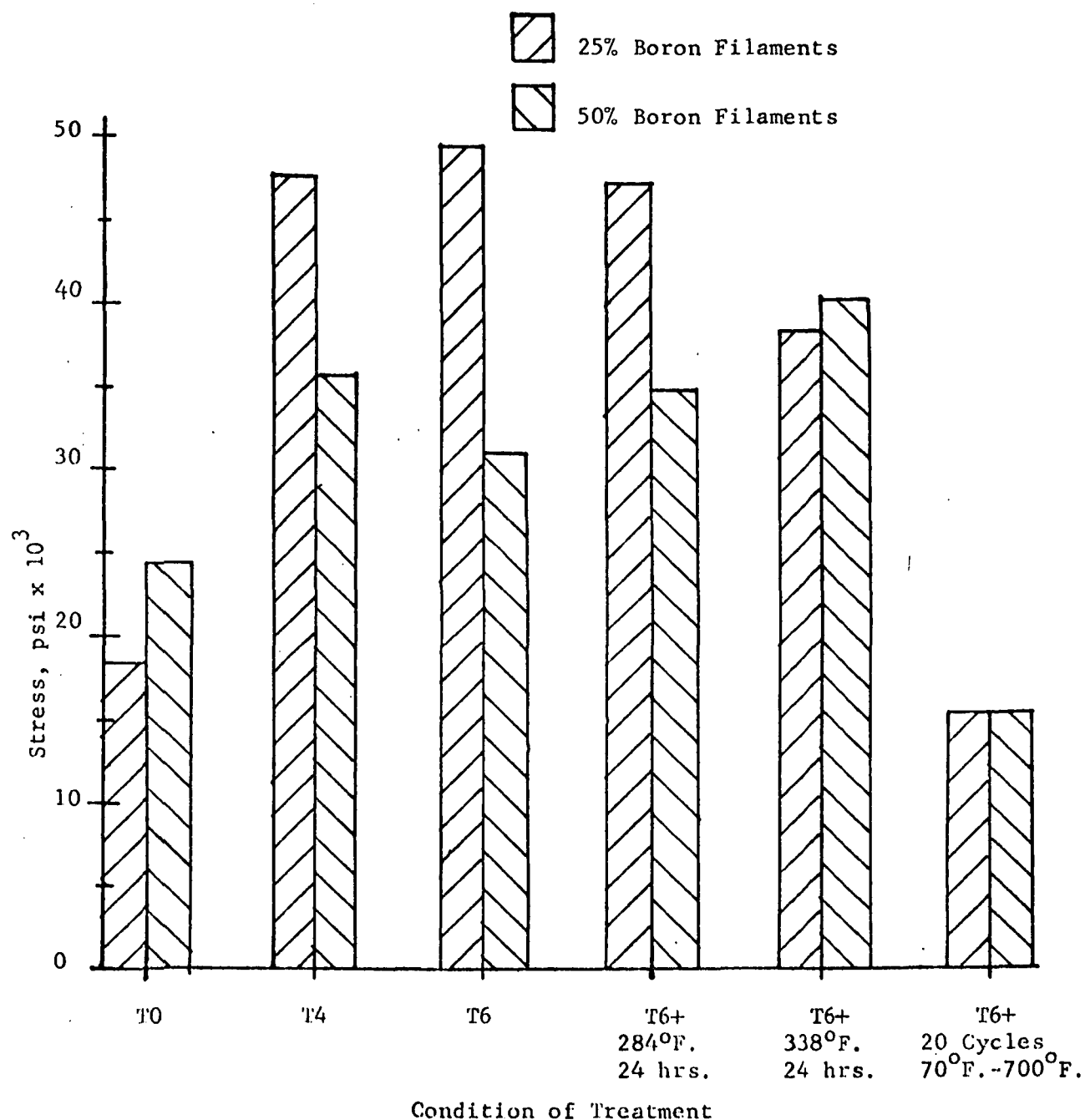


Figure 13. Transverse Tensile Yield Strength Versus Heat Treatment or Stress Relief (2024 Alloy)

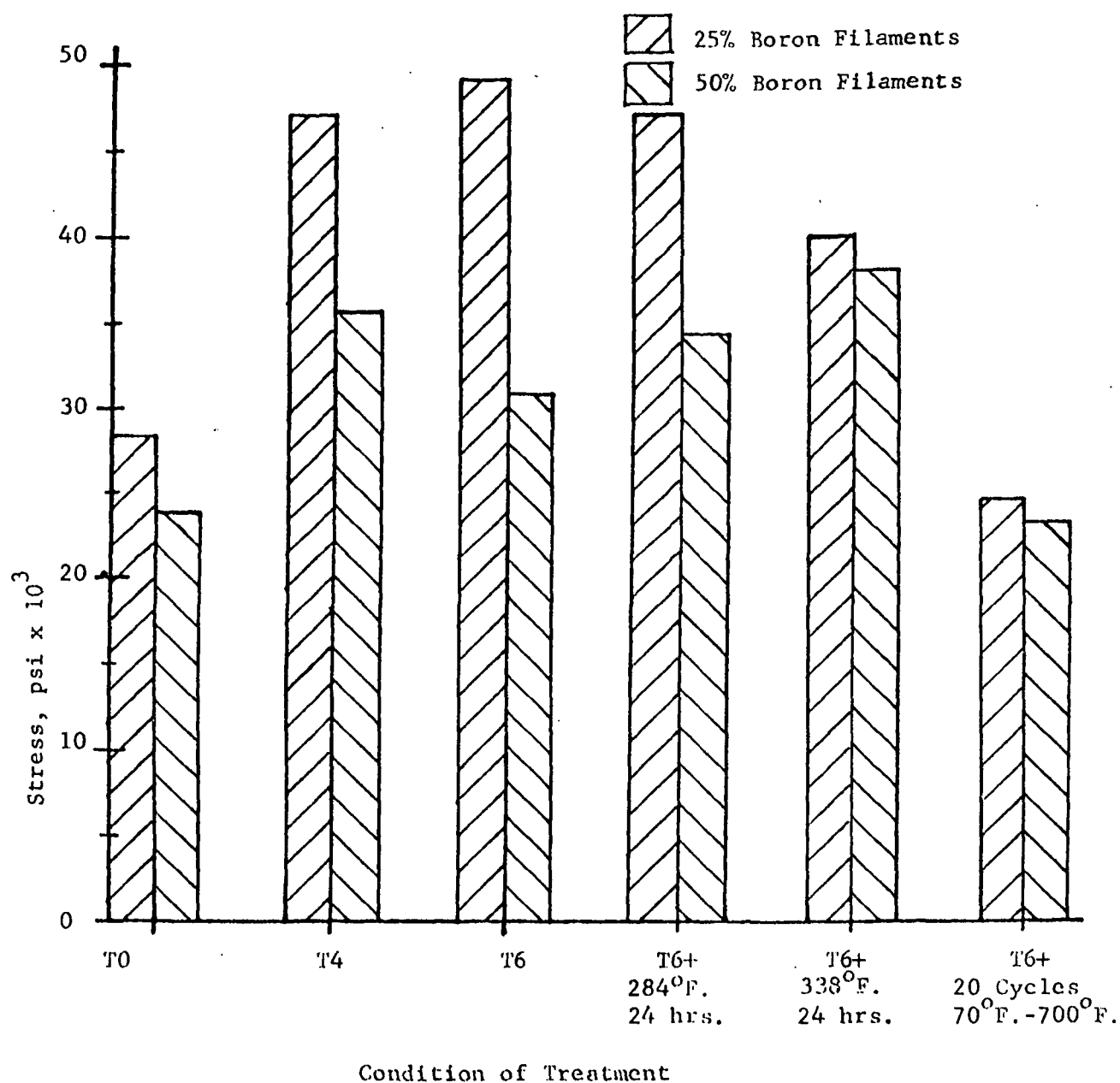


Figure 14. Transverse Ultimate Tensile Strength Versus Heat Treatment or Stress Relief (2024 Alloy)

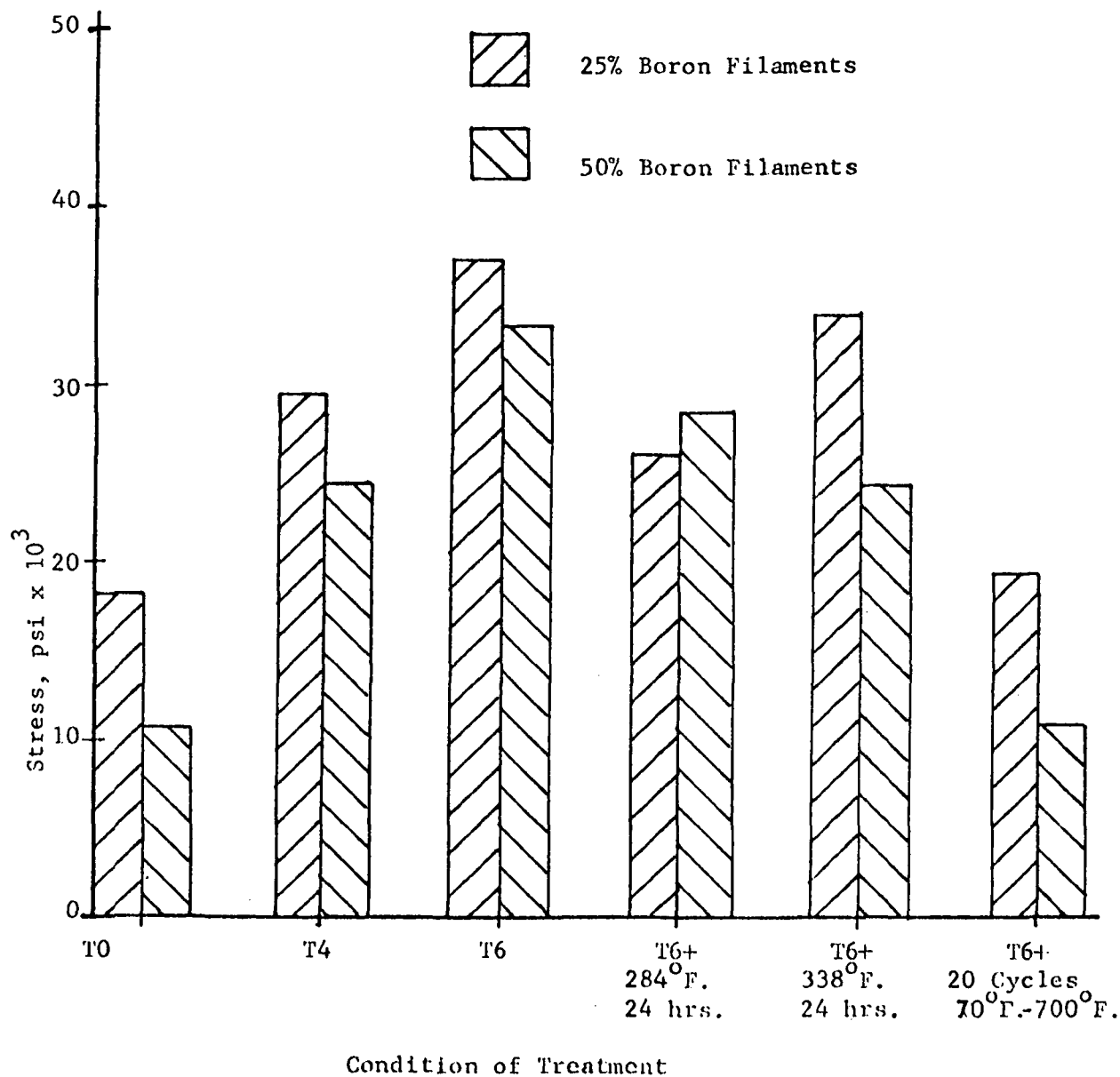


Figure 15. Transverse Tensile Yield Strength Versus Heat Treatment or Stress Relief (6061 Alloy)

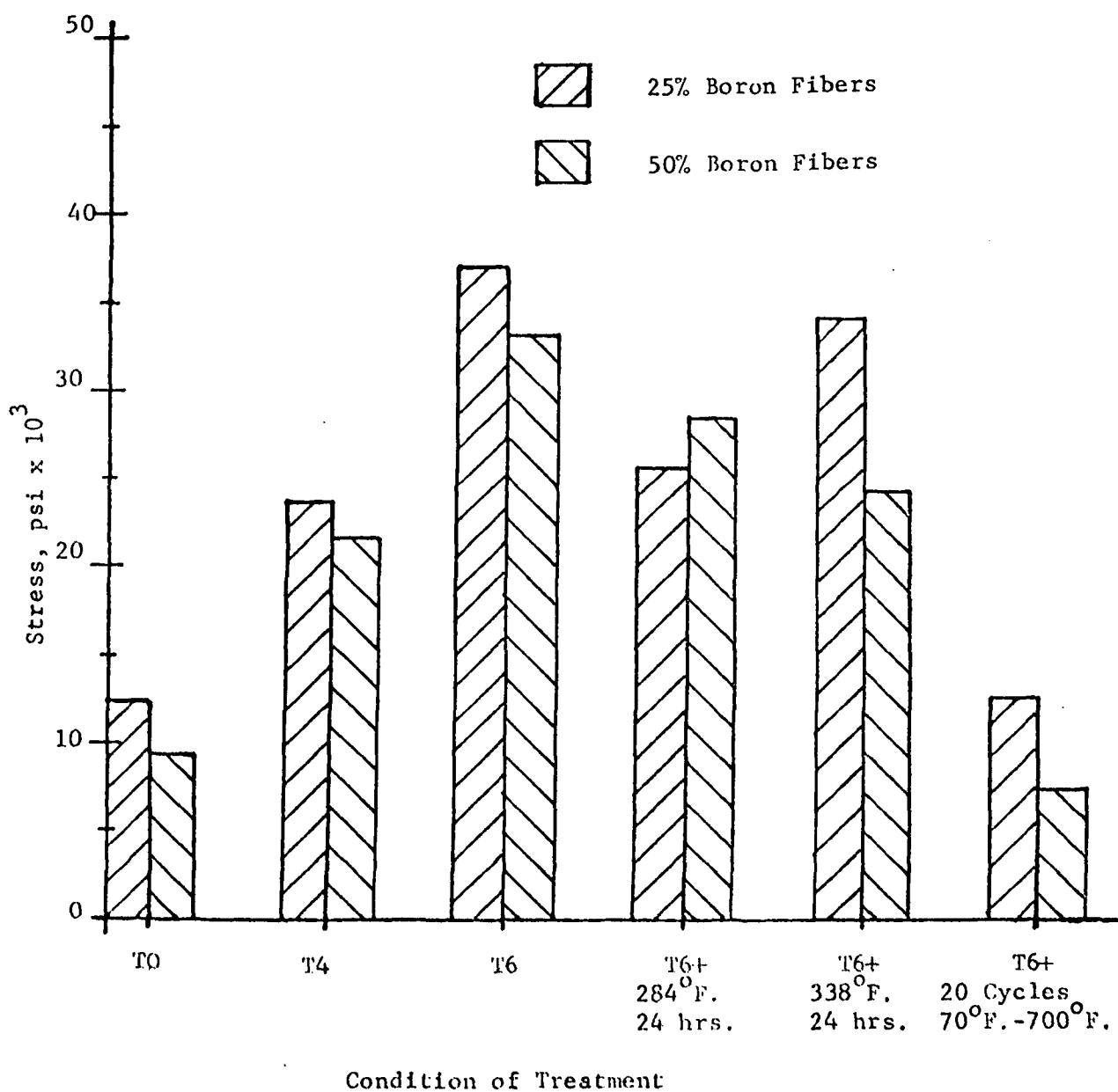


Figure 16. Transverse Ultimate Tensile Strength Versus Heat Treatment or Stress Relief (6061 Alloy)

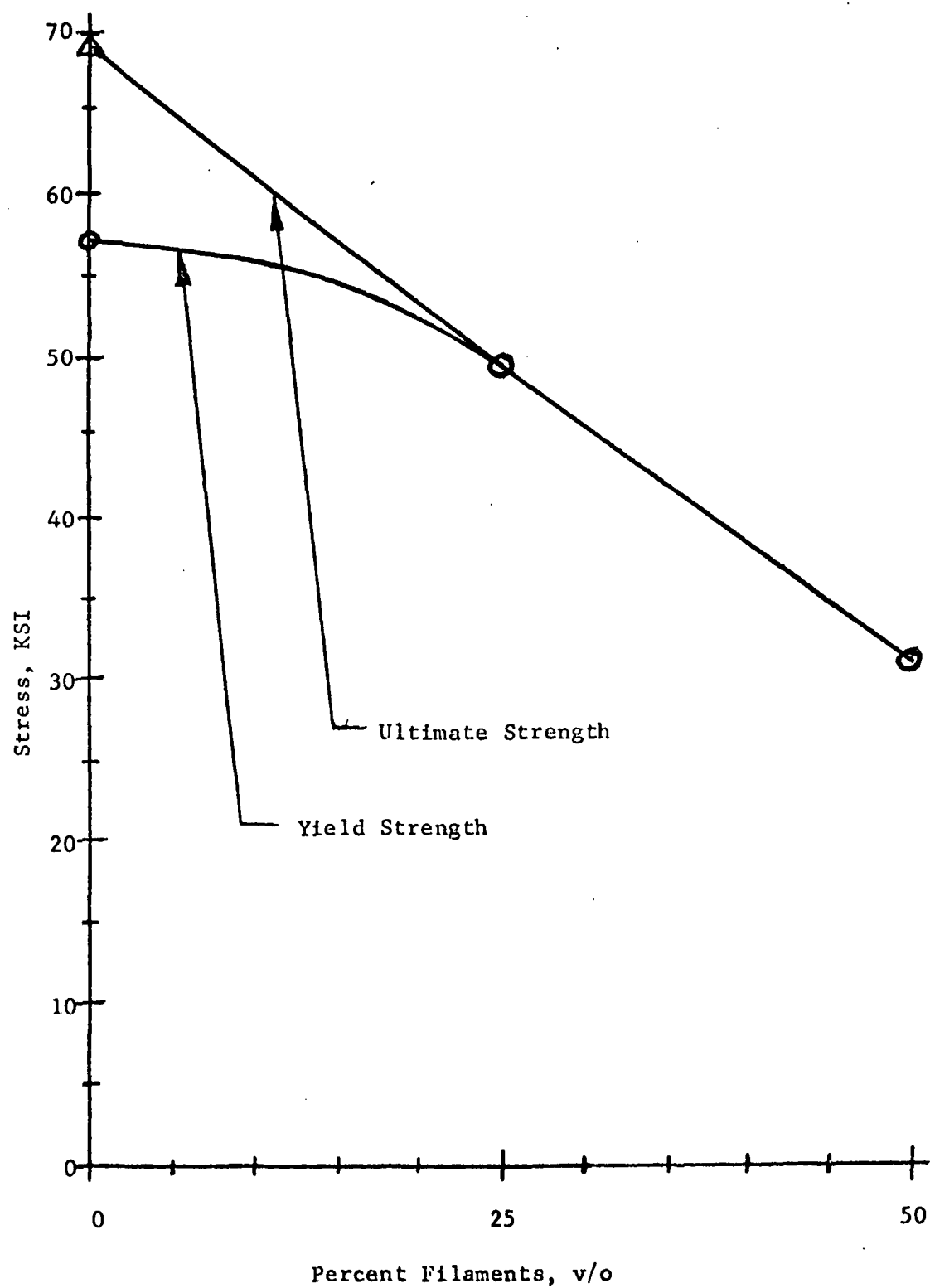


Figure 17. Transverse Tensile Strength Versus Percent Reinforcement
(Material: 2024-T6 Matrix)

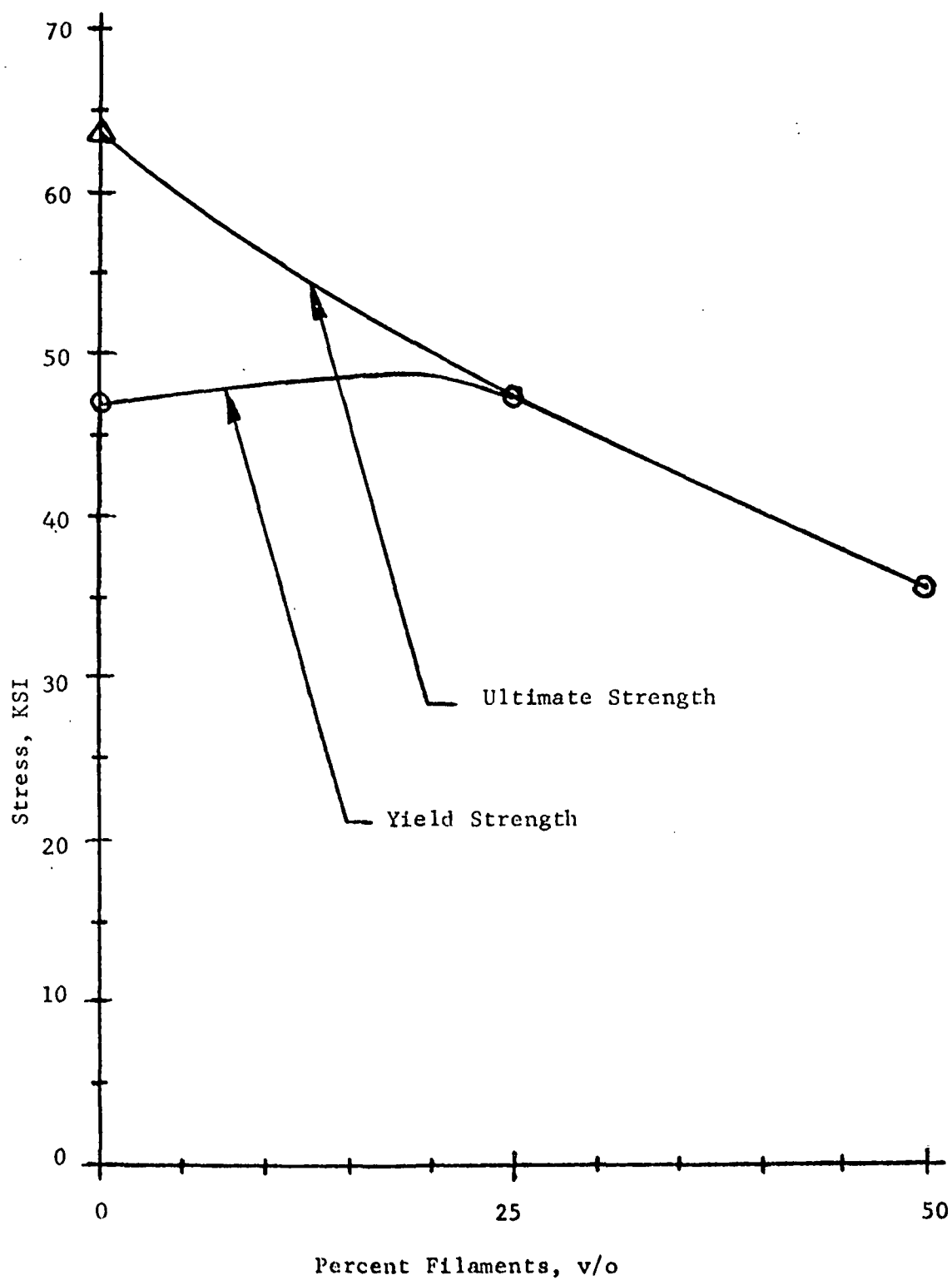


Figure 18. Transverse Tensile Strength Versus Percent Reinforcement
(Material: 2024-T4 Matrix)

filament reinforcement, is shown in Figure 19. The matrix 6061-T4 plot is shown in Figure 20.

The change of transverse modulus of elasticity due to percent filament reinforcement for both 2024 and 6061 aluminum composites is shown in Figure 21. As expected from the rule of mixtures, the high modulus of the boron increases the composite transverse modulus as the percent boron increases.

6.3 Effect of Cold Rolling

The effect of cold rolling of the aluminum composite to the T3 condition using 5% and 10% cold rolling (decrease in thickness), and also heat treating after cold rolling to the T36 condition is shown in Figures 22 through 27. For alloy 6061 with 25% boron filaments, the effect upon yield strength due to cold rolling is shown in Figure 22. For this same 6061 alloy with 25% boron, the effect upon ultimate tensile stress due to cold rolling, is shown in Figure 23. In both cases, it is noted that 5% cold rolling indicates that it increases these mechanical properties somewhat ($\approx 6\%$).

Cold rolling of 6061 alloy composite with 50% boron filaments, using the 5% and 10% cold rolling (T3) as well as the added heat treatment (T36), has the effect upon transverse yield strength as shown in Figure 24. The effect upon transverse ultimate tensile strength is shown in Figure 25. As noted for the 25% boron composites, the 5% cold rolling procedure appears to produce the highest strengths for the 50% boron composites as well.

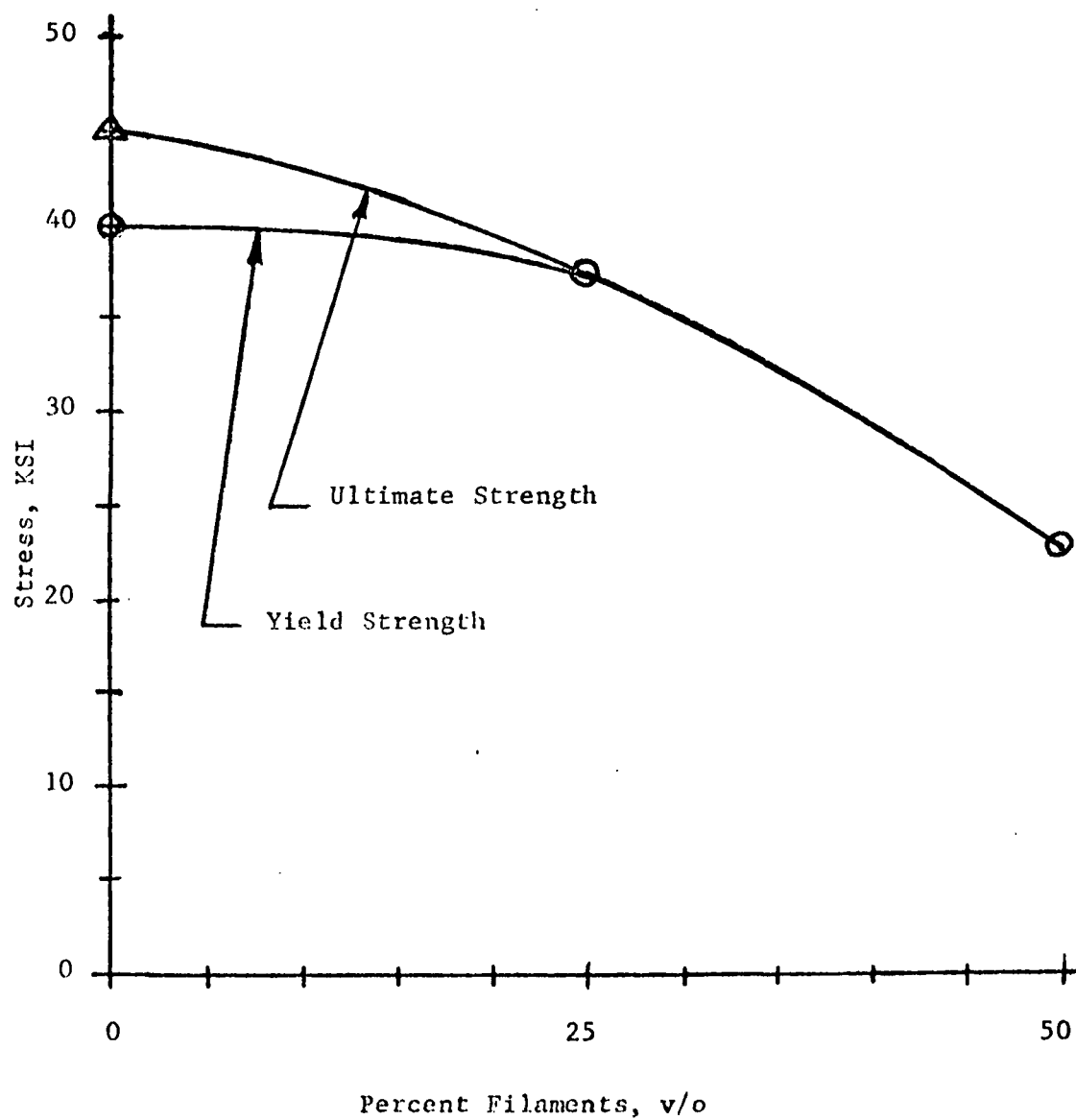


Figure 19. Transverse Tensile Strength Versus Percent Reinforcement
(Material: 6061-T6 Matrix)

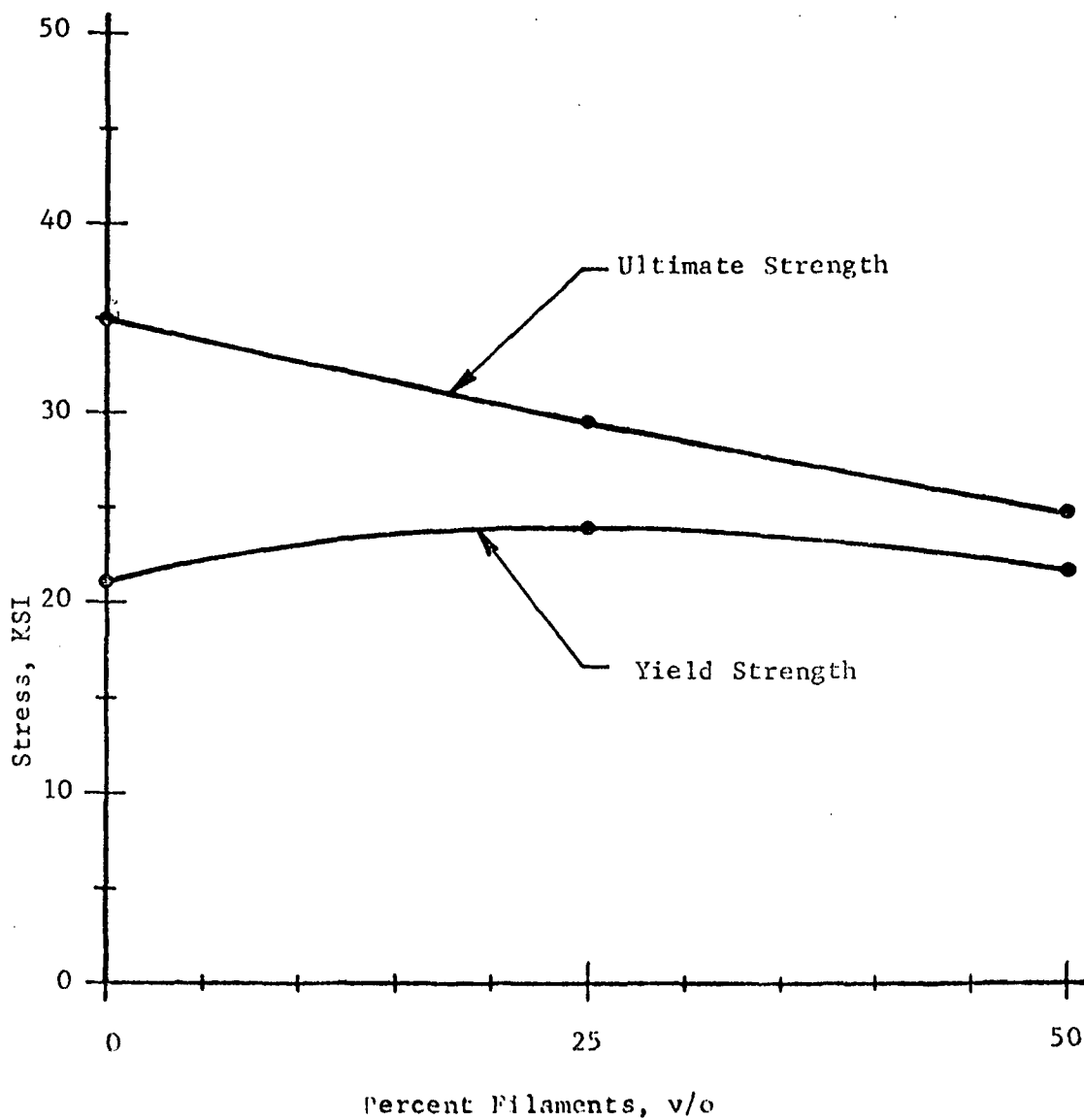


Figure 20. Transverse Tensile Strength Versus Percent Reinforcement
(Material: 6061-T4 Matrix)

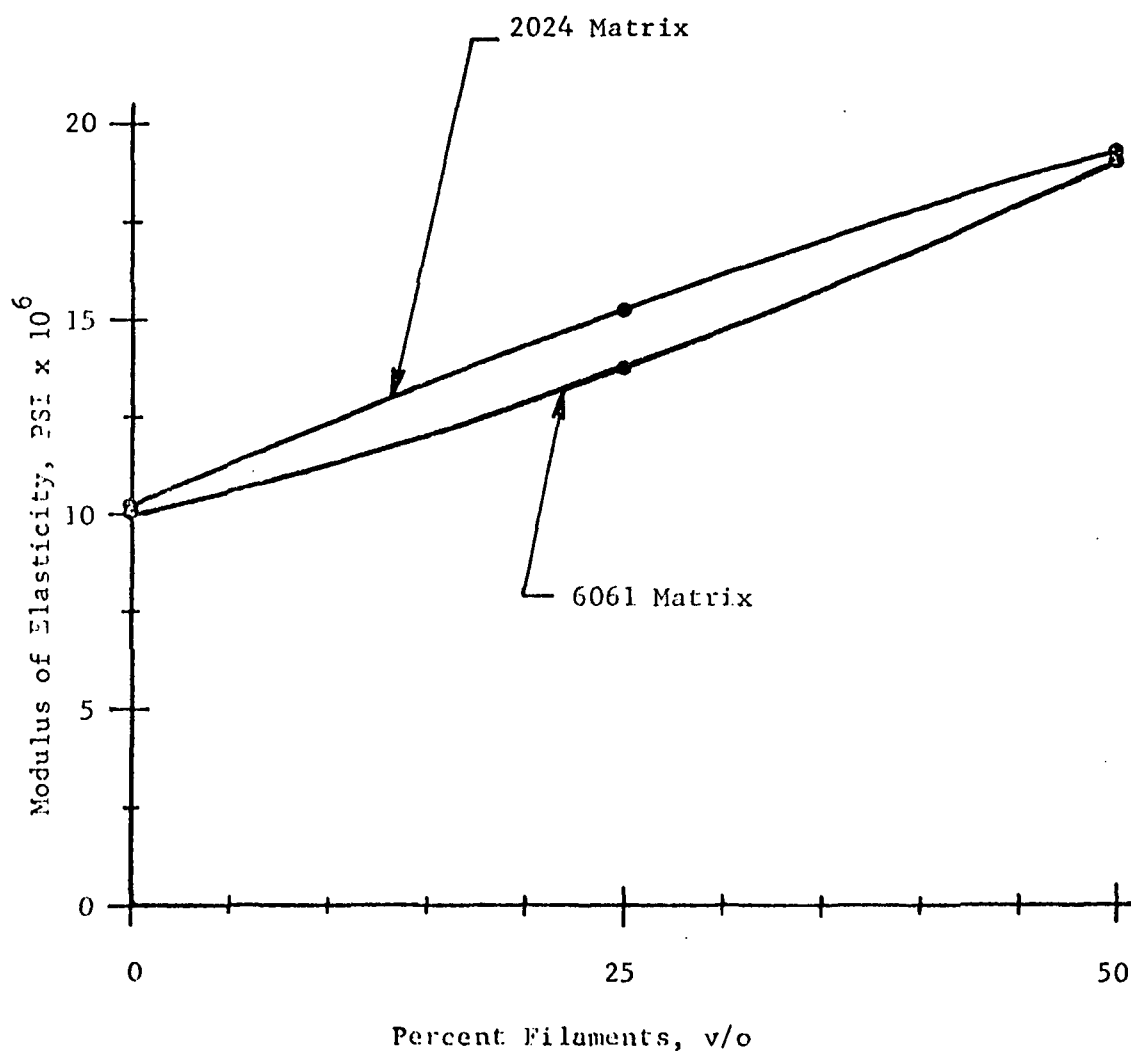


Figure 21. Transverse Modulus of Elasticity Versus Percent Reinforcement

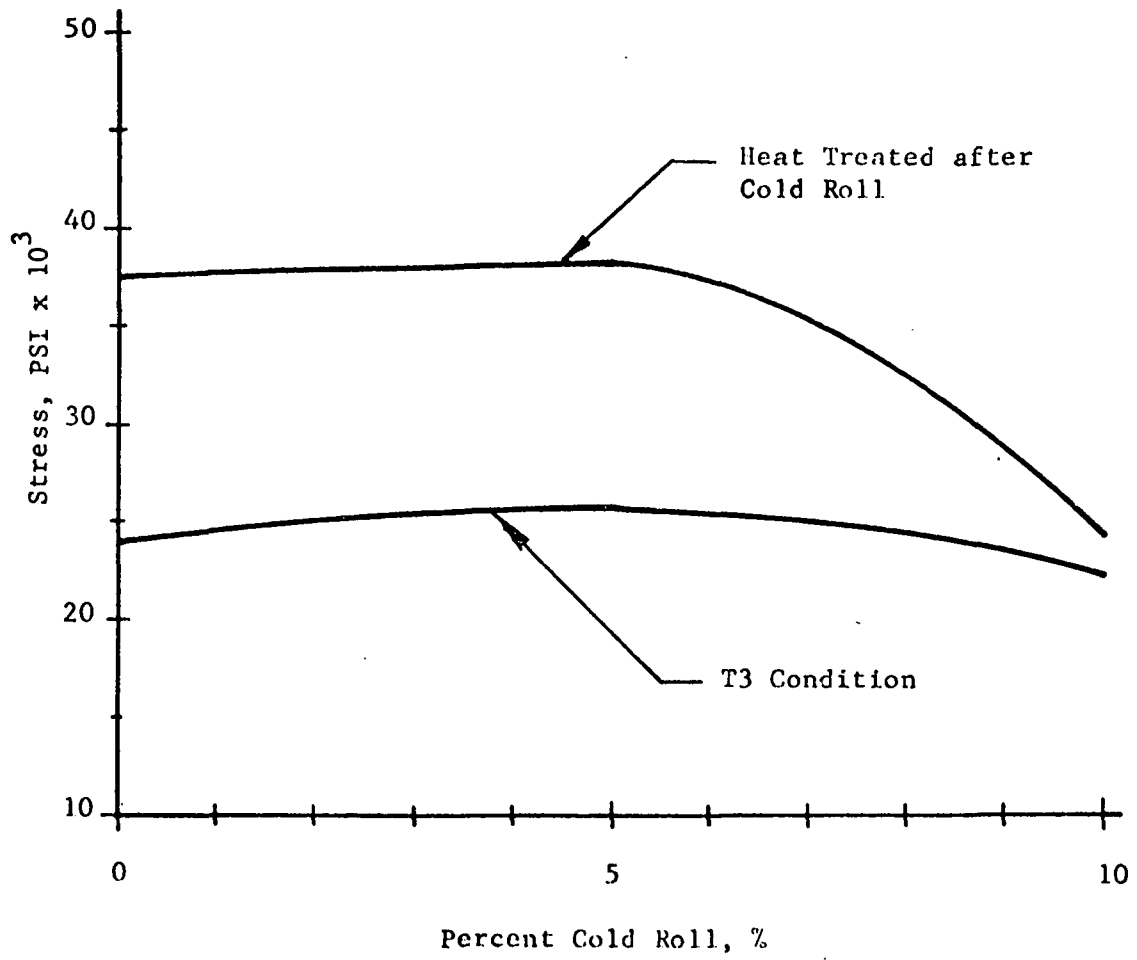


Figure 22. Effect of Cold Rolling on Transverse Yield Strength (6061 Alloy--25% Boron Filaments)

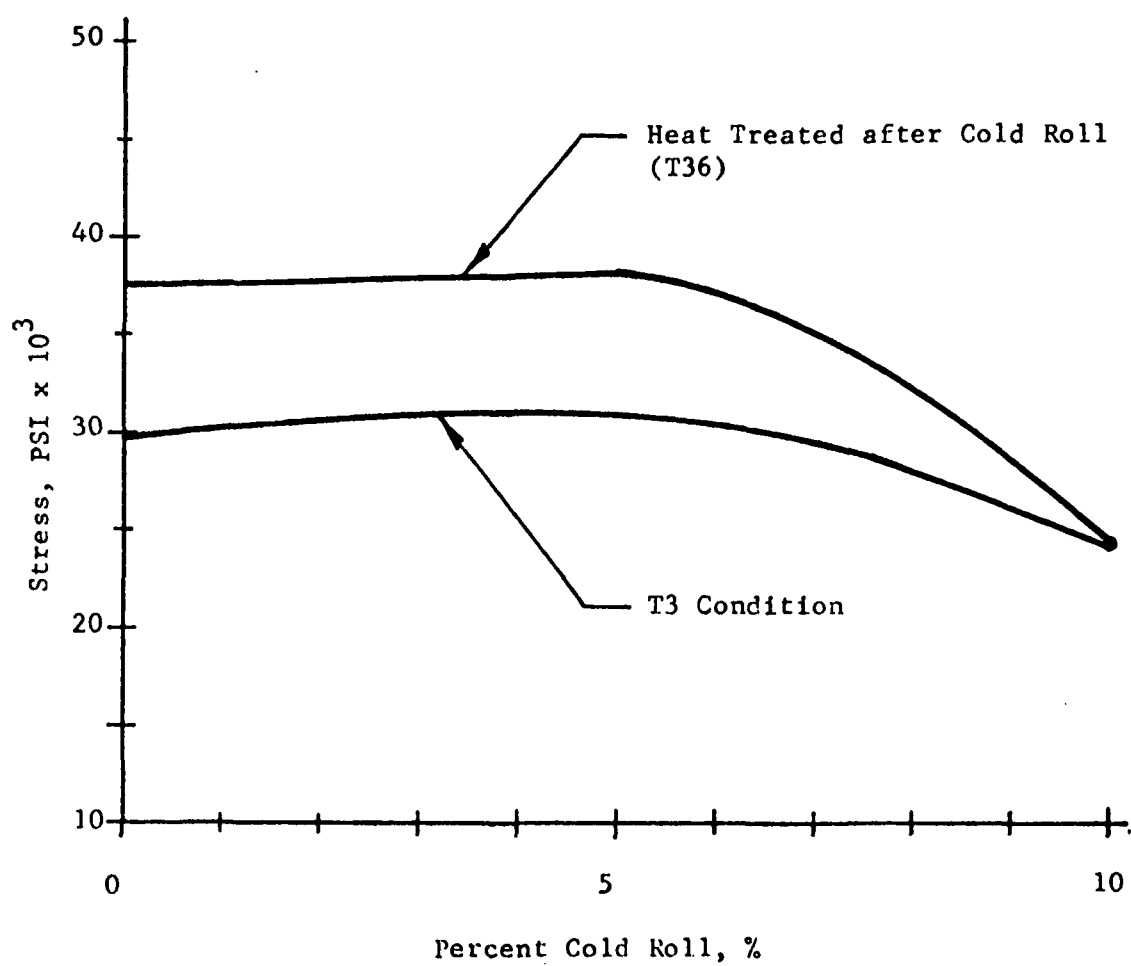


Figure 23. Effect of Cold Rolling on Transverse Ultimate Strength (6061 Alloy--25% Boron Filaments)

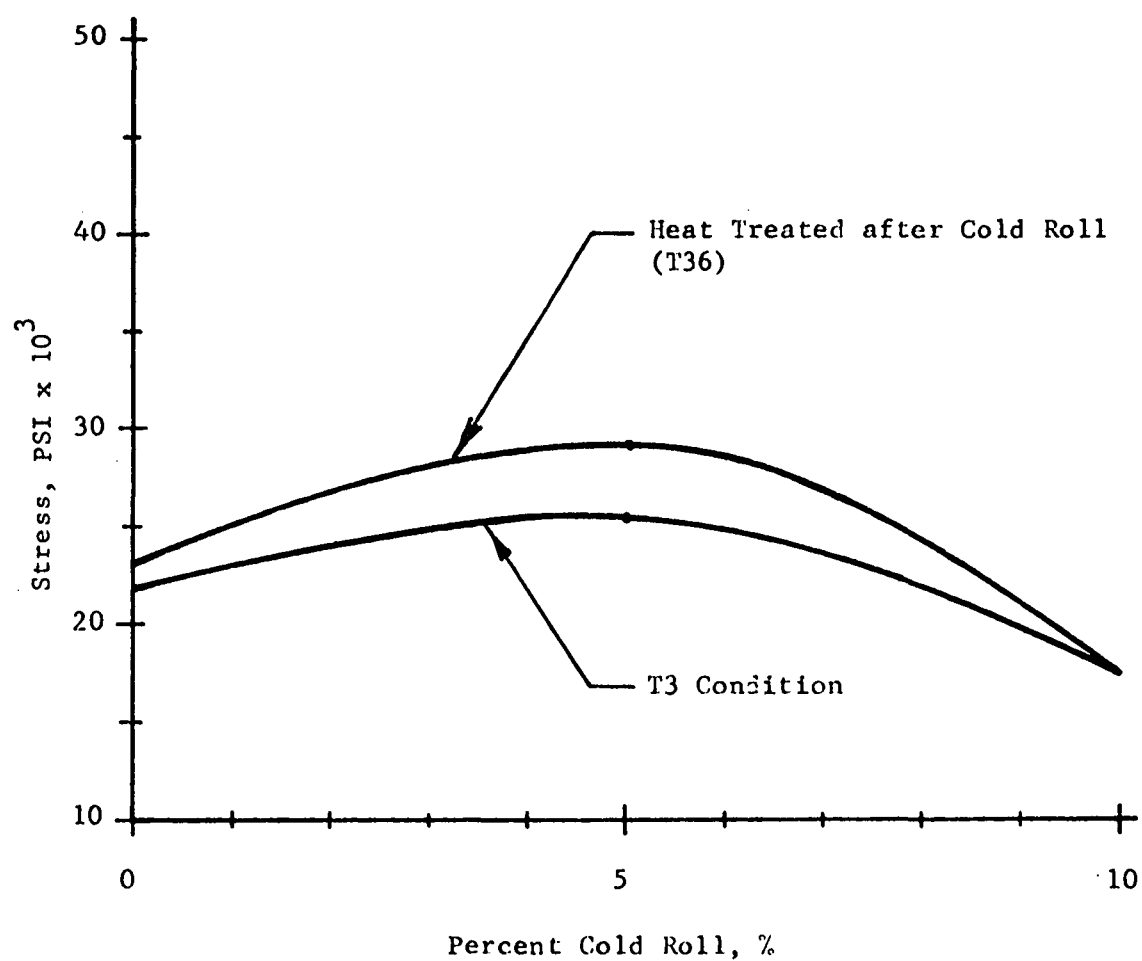


Figure 24. Effect of Cold Rolling on Transverse Yield Strength (6061 Alloy--50% Boron Filaments)

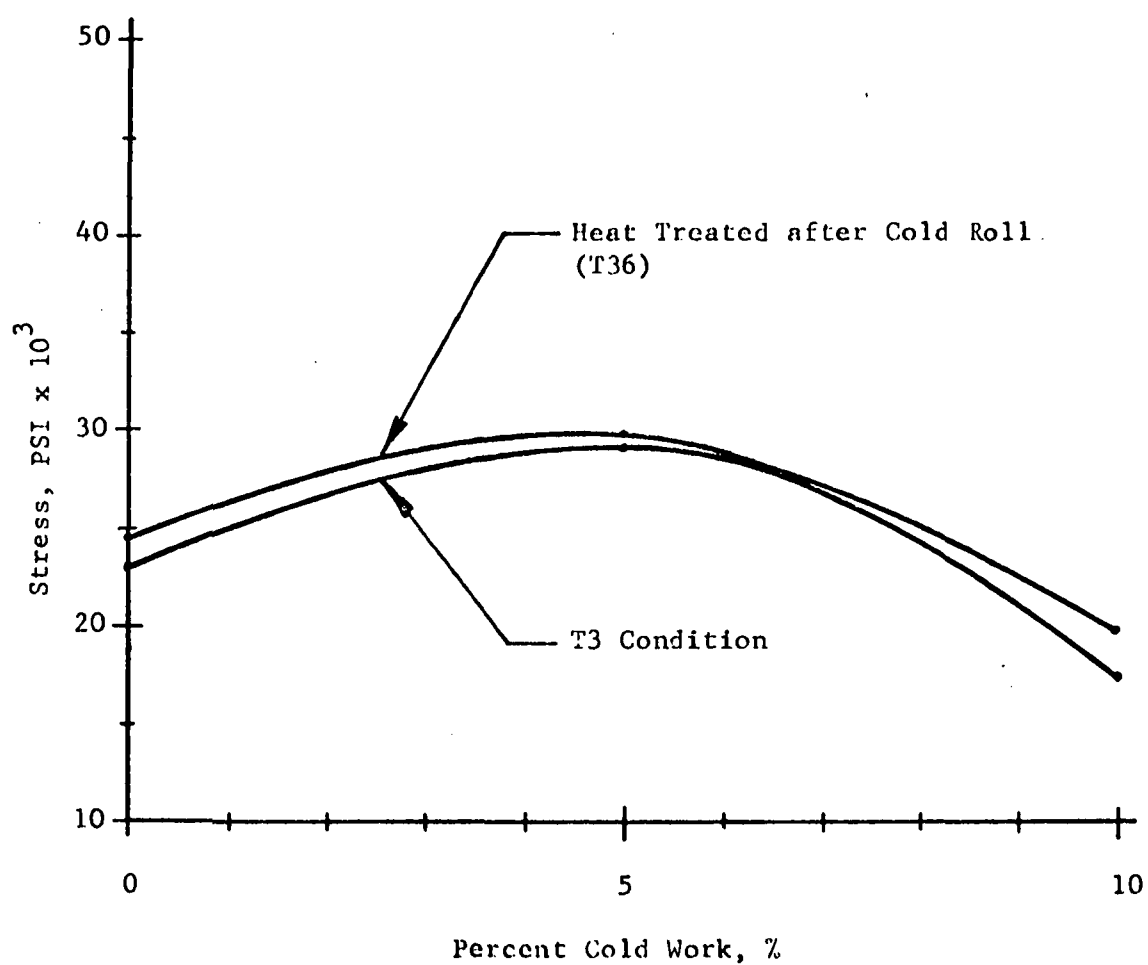


Figure 25. Effect of Cold Rolling on Transverse Ultimate Strength (6061 Alloy--50% Boron Filaments)

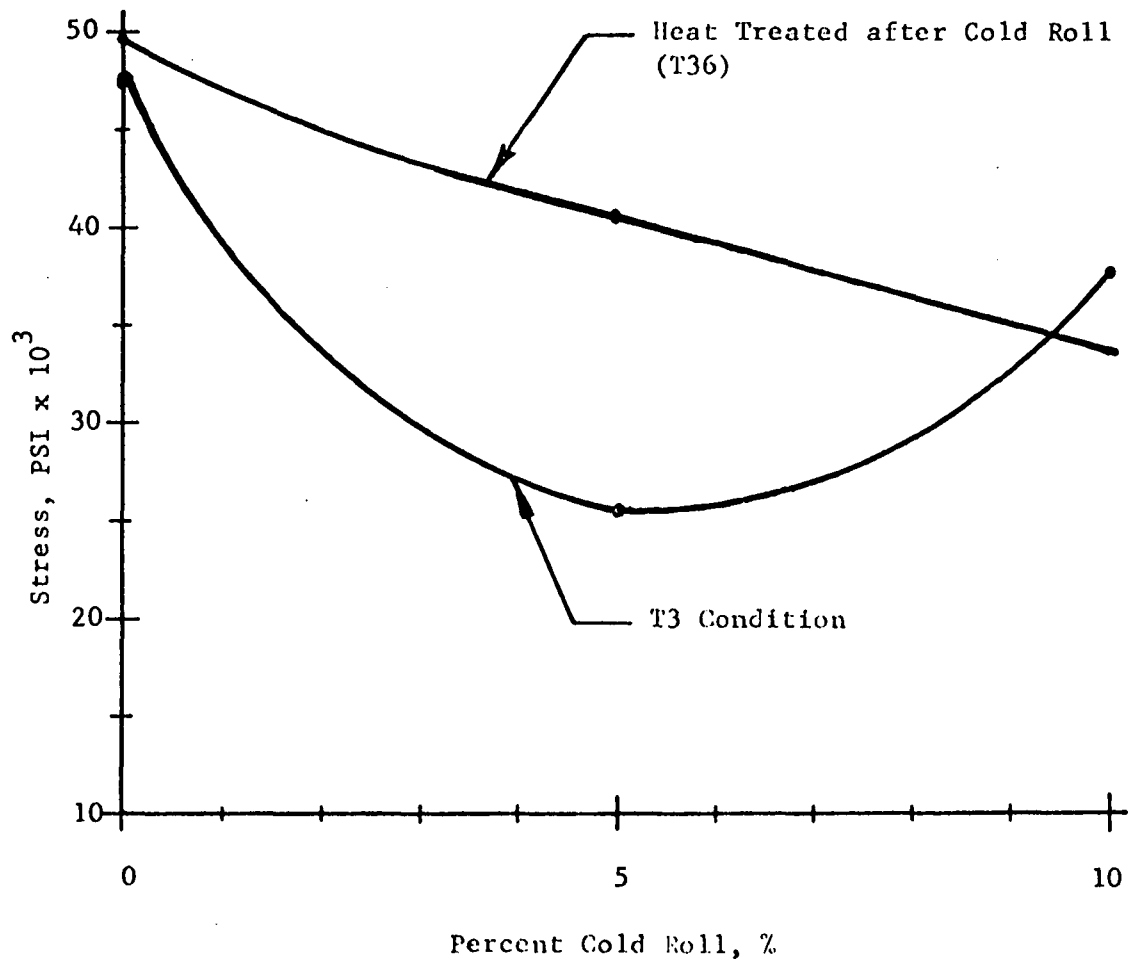


Figure 26. Effect of Cold Rolling on Transverse Yield Strength (2024 Alloy--25% Boron Filaments)

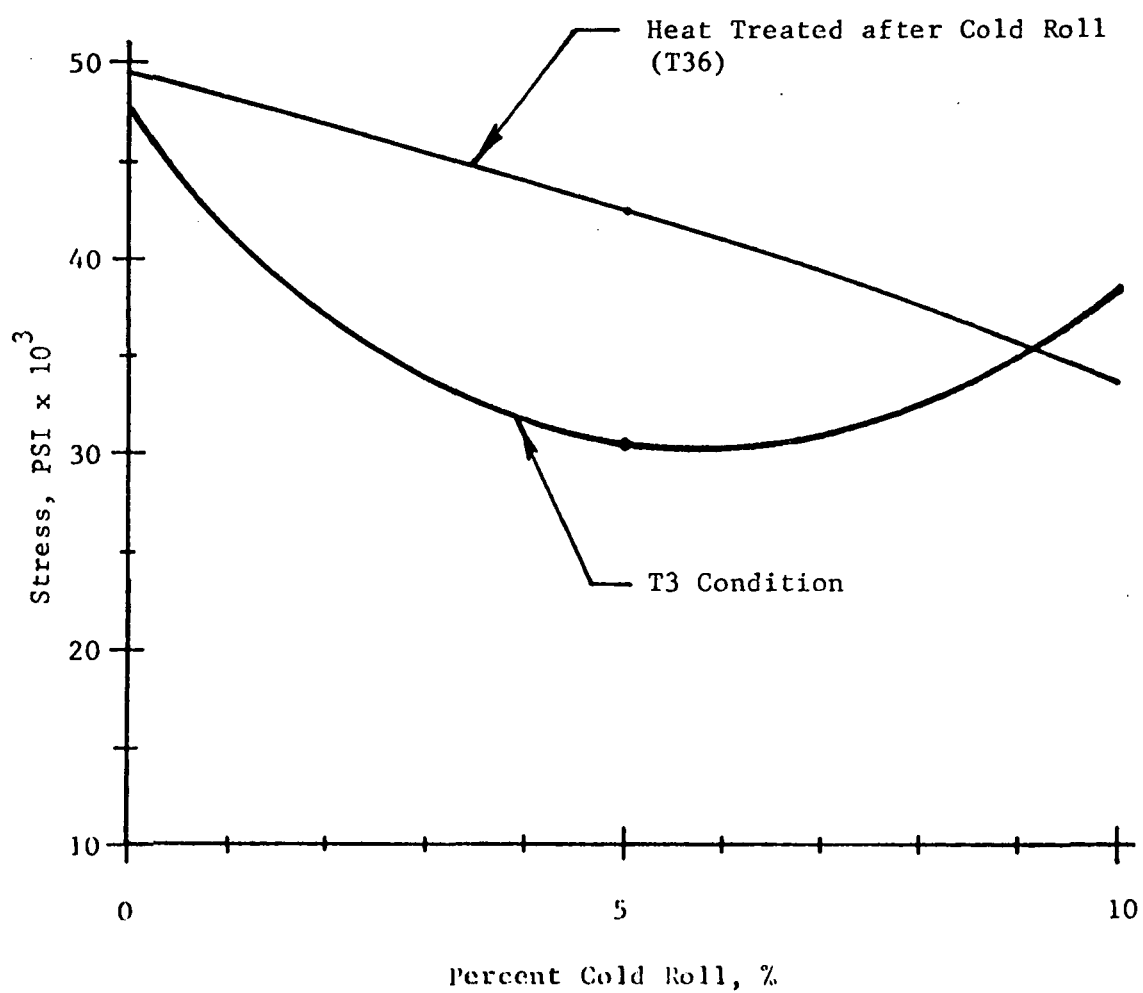


Figure 27. Effect of Cold Rolling on Transverse Ultimate Strength (2024 Alloy--25% Boron Filaments)

Cold rolling of 2024 alloy composite with 25% boron filaments, using 5% and 10% cold roll (T3) as well as subsequent heat treatment (T36), has the effect upon transverse yield stress as shown in Figure 26. The effect upon transverse ultimate tensile strength is shown in Figure 27. For this composite, the cold rolling procedure appears to degrade the mechanical properties.

The effect upon transverse yield and ultimate strength of 2024 alloy composite with 50% boron filaments, of cold rolling using 5% and 10% cold roll (T3) as well as subsequent heat treatment (T36), is as shown in Figure 28. Again, for the 2024 alloy composite, cold rolling appears to degrade the strength properties.

The apparent modulus of elasticity in the transverse direction of 6061 alloy composite with either 25% or 50% boron filaments, is affected by cold rolling as shown in Figure 29. The composite of 2024 alloy is affected by cold rolling as shown in Figure 30. In the 6061 case, one must conclude that cold rolling to 5% has a tendency to increase the apparent modulus somewhat. In the 2024 composite, the cold rolling was definitely detrimental to the modulus of elasticity.

6.4 Effect of Strain Cycling

As shown in Figures 13 and 14 for the 2024 composite and in Figures 15 and 16 for the 6061 composite, the strain cycling of the cycles of 10% of the UTS before testing the specimens was very detrimental to the material. The results were about one-half of those obtained from the standard T6 condition.

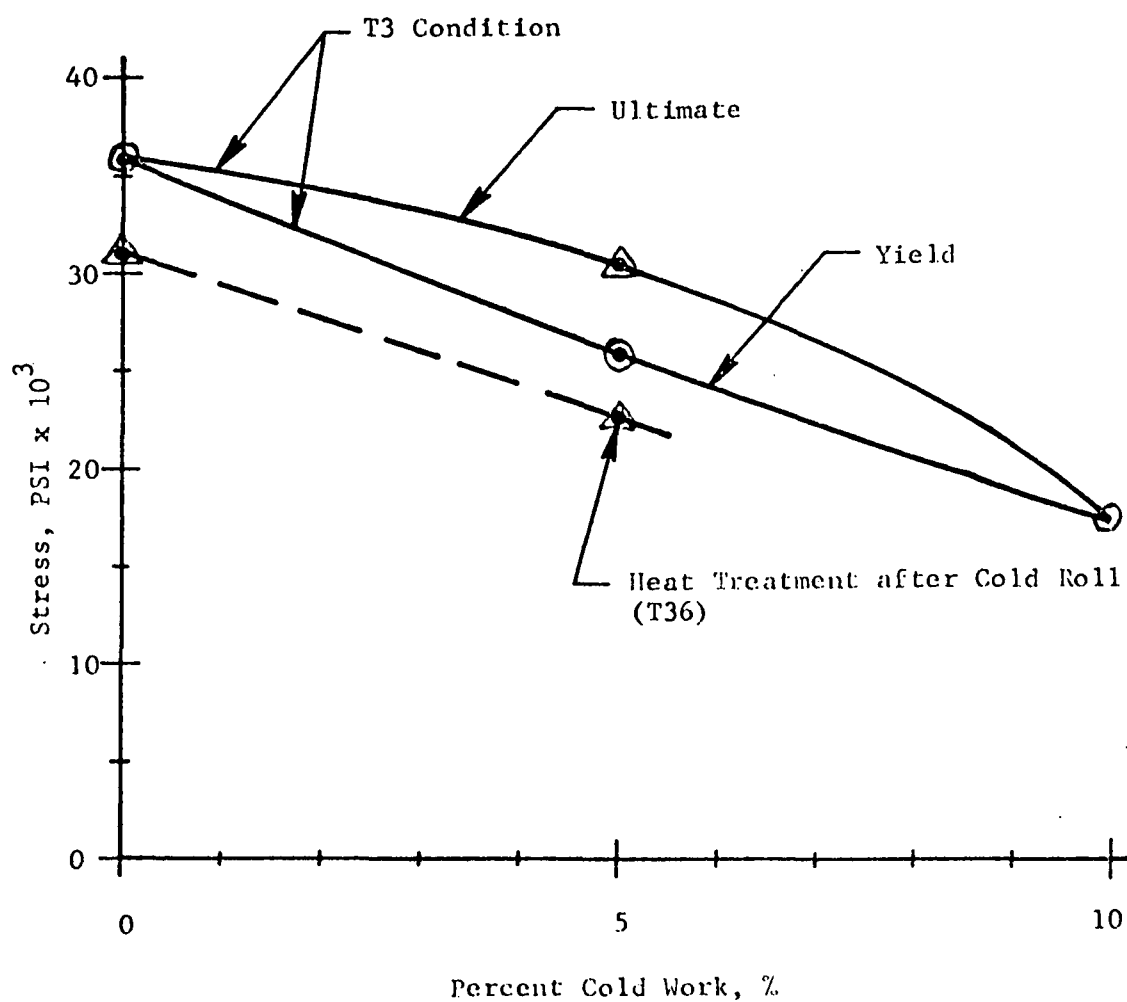


Figure 28. Effect of Cold Rolling on Transverse Ultimate and Yield Strength (2024 Alloy--50% Boron Filaments)

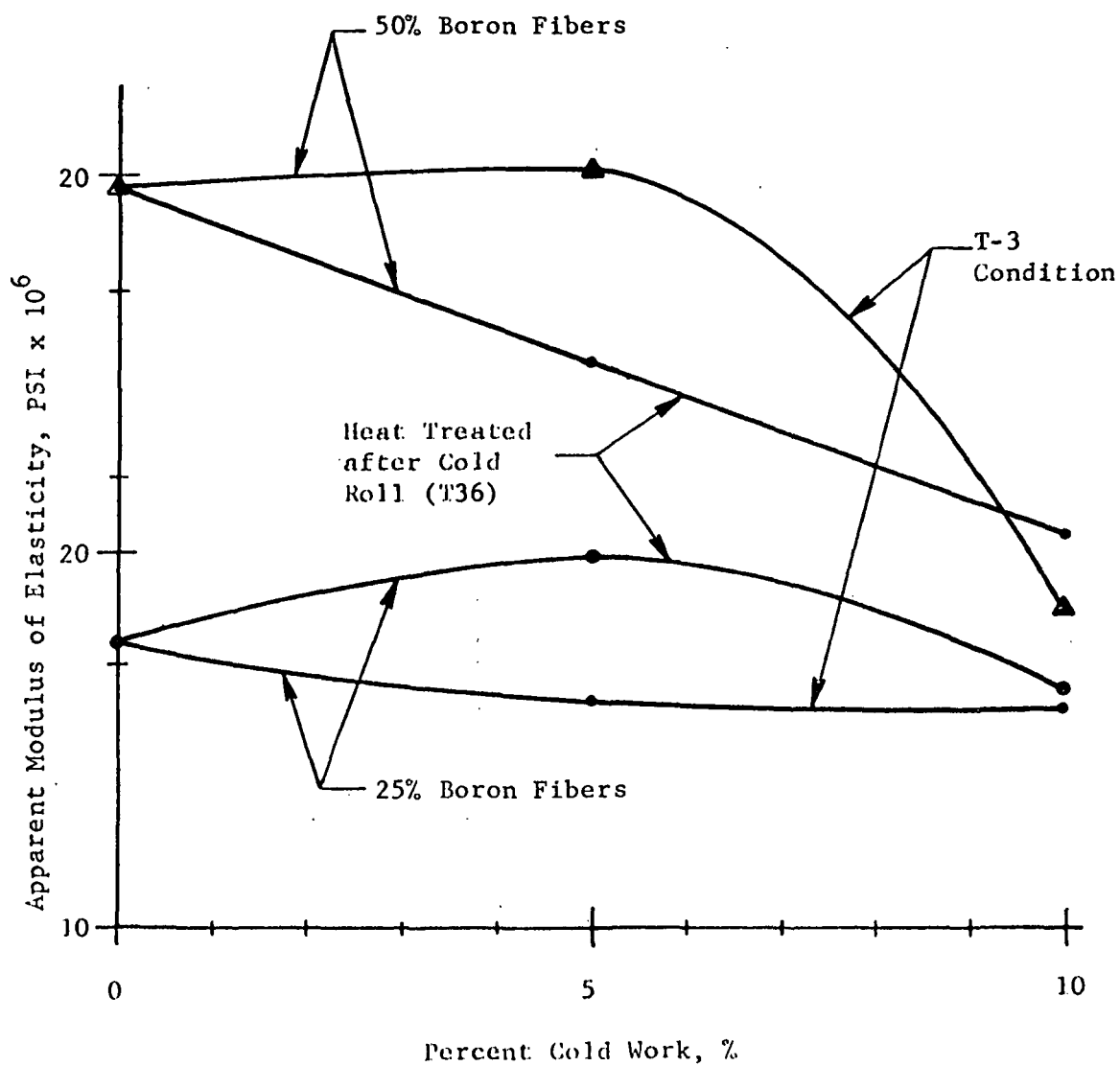


Figure 29. Effect of Cold Rolling on Apparent Transverse Modulus of Elasticity (6061 Alloy)

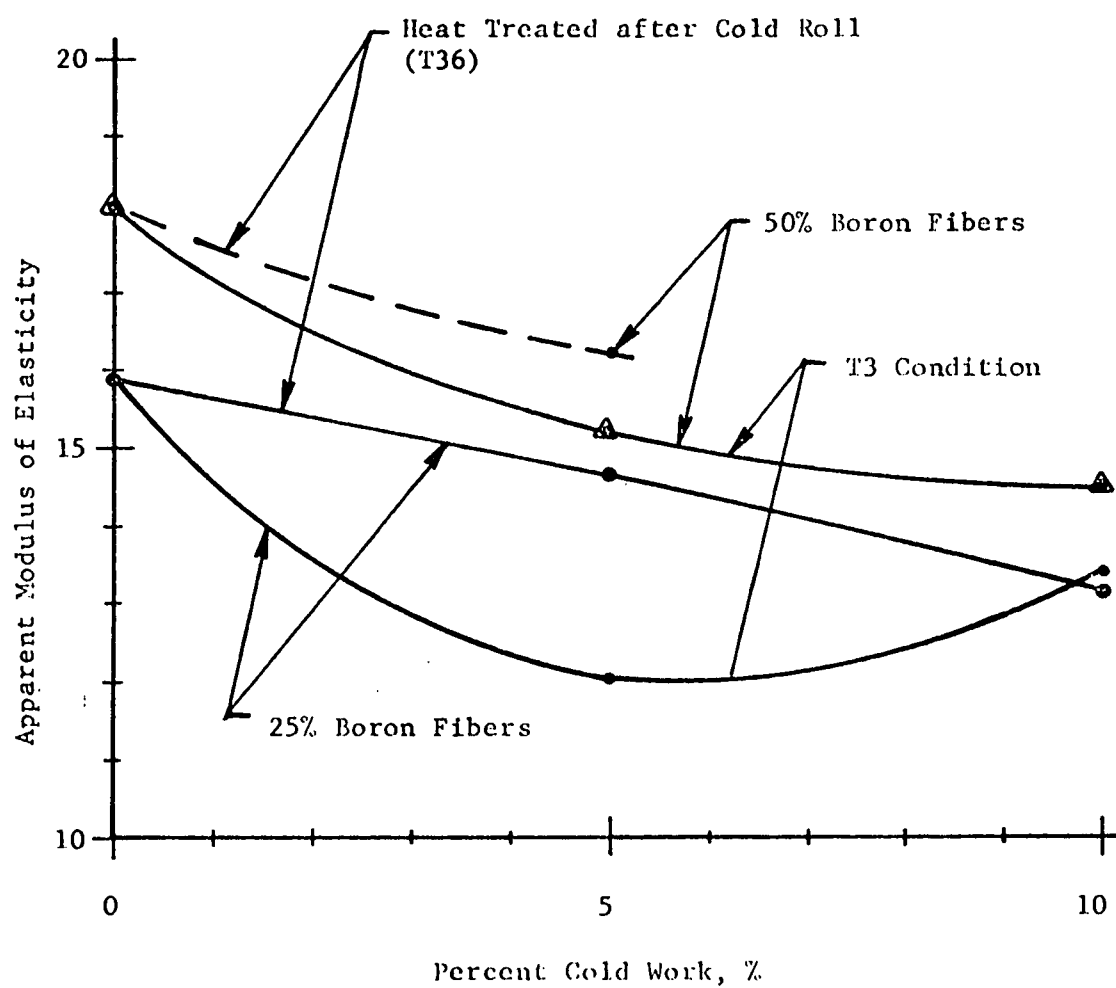


Figure 30. Effect of Cold Rolling on Apparent Transverse Modulus of Elasticity (2024 Alloy)

6.5 Mode of Fracture

The mode of fracture in general was fracture of the matrix. No bond failures of the aluminum matrix to the boron filaments were observed. There was some splitting of the filaments in the fracture surfaces, however, except for one case; there was no way to determine if the splitting initiated at the filament free ends (due to fabrication cracks) or originated within the test area due to inherent subsurface cracks. In one case, noted in Table 6, there was one fiber completely split in the reduced width test area but not in the free ends.

A fractographic analysis of the test specimens was not performed, since fractography was not within the scope of this program.

6.6 Discussion

Of the various thermal treatments evaluated in this test program, the standard T6 process appeared to produce the best results for both the 6061 and 2024 aluminum alloy matrix composites. Cold rolling of 5% appeared to be slightly beneficial for 6061 composite while 10% cold rolling was detrimental. For the 2024 composite, all cold rolling appeared to be detrimental.

The strain cycling procedure evaluated in this test program was very detrimental to physical properties.

The test data obtained from the 50% boron test specimens were not as reliable as expected. This was caused by pitting and uneven etching of the thickness of the specimens especially of the 2024 matrix composite specimens. Therefore, any of the results from these 50% boron specimens should not be considered for design purposes.

Since one of the major objectives of this investigation was to evaluate the splitting of the Avco 5.6-mil-diameter filaments, it was encouraging to note that the mode of fracture was chiefly confined to matrix failure.

CHAPTER 7

CONCLUSIONS

The first objective of this investigation, namely, investigate the new transverse tensile specimen preparation technique, produced very consistent test results. The isolation of the filament end splits from the reduced-width test area on the specimens proved to be effective since few filament splits were observed in the test area of fractured specimens. The elimination of this splitting problem produced high transverse strengths since failure was primarily matrix fracture. The fabrication procedure for the 3/8-inch reduced width test specimens described herein was straightforward and was comparable to the data reported by Kreider, Dardi, and Prewo (1970) for one-inch wide specimens etched to a 1/2-inch reduced width test area. The overall technique, however, is highly recommended as a standardized laboratory test procedure for transverse tensile testing.

The second objective of this investigation, namely, observe behavior of transverse strength properties with various thermal post-treatments, showed that the standard T6 heat treatment produced the highest transverse strengths. (The effect of the T6 heat treatment on longitudinal tensile strength was not within the scope of this program.)

The third objective of this investigation, namely, evaluate the new 5.6-mil-diameter boron filaments, showed that they indeed produce few splits as recently reported in the literature (Kreider, Dardi and Prewo, 1970).

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QUALITY ASSURANCE SPECIFICATION

NO. XCQ-1

Inspection of Aluminum Foil for Composites

1.0 SCOPE

1.1 This specification establishes the requirements for visual inspection of aluminum foil for composites both as-received and processed.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 MMA Spec XCM-1 - Aluminum Foil for Composites

2.1.2 Fed. Spec QQA-250 - Aluminum Alloy Plate, Sheet & Foil

2.1.3 MMA Spec XCPA-1 - Preparation of Aluminum Foil for Production of Diffusion Bonded Composites

2.1.4 MMA Spec XCQ-3 - Measurement of Surface Resistivity of Aluminum Foil for Composites

3.0 REQUIREMENTS

3.1. RECEIVING INSPECTION

3.1.1 Supplies Certification. The certification shall be checked for conformity to the requirements of QQA-250 and XCM-1 prior to acceptance.

3.1.2 Damage and Defects. The foil package and contents shall be examined for obvious defects and damage. Foil which has defects or has been damaged to the extent that it may not be suitable for production of composites shall be reviewed prior to acceptance or rejection.

3.2. Inspection of Processed Foil

3.2.1. In-Process Inspection

3.2.1.1 Damage and Defects. At each step in the processing cycle (eg. cleaning, the foil shall be examined for proper processing and for damage and defects which might make it unsuitable for production of composites. Questionable material shall be subjected to material review procedures.

3.2.1.2 Cleaning. A check shall be made to assure that all solutions have been properly maintained prior to each usage.

3.2.1.3 Equipment Check. A check shall be made to assure that all equipment is functioning properly prior to each usage.

3.2.2.1 Cleanliness. The foil shall be free of all grease, dirt, and other contaminants in accordance with the requirements of MMA Spec No. XCPA-1. The surface resistance shall not exceed 300 micro-ohms as determined in accordance with MMA Spec No. XCQ-3

3.2.2.2 Thickness. Thickness of all foil shall be within 10% of the nominal thickness specified on the drawing. Measurements shall be made in a manner to assure that the thickness is within this tolerance over the entire area of the foil.

3.2.2.3 Defects. The foil shall be free of holes, tears and other defects.

QUALITY ASSURANCE SPECIFICATION

NO. XCQ-3

Measurement of Surface Resistivity of Aluminum Foil for Composites

1.0 SCOPE

1.1 This specification establishes the requirements for measurement of surface resistivity of aluminum foil prepared for production of composites.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 MMA Spec. No. XCPA-1 - Preparation of Aluminum Foil for Composites

2.1.2 MMA Spec. No. XCQ-1 - Inspection of Aluminum Foil

3.0 REQUIREMENTS

3.1 Foil Preparation. Foil shall be prepared in accordance with MMA Spec. XCPA-1 prior to making resistance measurements.

3.2 Sampling. Representative samples shall be selected from at least five sheets of each lot of foil processed for production of composites. The samples shall be taken immediately prior to lay-up and after inspection in accordance with the other requirements of MMA Spec. XCQ-1.

3.3. Equipment. Equipment shall consist of Leeds and Northrup Model 4287-2 Kelvin Bridge or equivalent.

3.4 Measurement of Resistance

3.4.1 Measurement of surface resistance shall be performed in accordance with the following procedure:

1. Clean .5 dia. gold plated copper contact points
2. Apply 1000-lb. load
3. Read indicated resistance
4. Open contact points and insert foil sample
5. Apply 1000-lb load
6. Read indicated resistance

4.0 RECORDS

4.1. A record shall be made of all resistance measurements on the job card for each composite lot for which the foil is used.

4.2. The resistivity measurement recorded shall be the average of all measurements taken on the samples for the lot of foil.

QUALITY ASSURANCE SPECIFICATION

NO. XCQ-7

Leak Testing Vacuum Packages for Diffusion Bonding Composites

1.0 SCOPE

1.1 This specification establishes the requirements for leak testing vacuum packages for diffusion bonding composites.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 H.A. Spec. XCP-7 - Welding Vacuum Packages for Diffusion Bonding Composites

2.1.2 H.A. Spec. XCPA-5 - Diffusion Bonding Aluminum Matrix Composite

3.0 REQUIREMENTS

3.1 Within 2 hours after welding in accordance with H.A. Spec. XCP-7, vacuum packages shall be inspected for leaks.

3.2 Pressure Test

3.2.1 The package shall be purged with argon by filling and evacuating 3 times after which the outlet shall be sealed and the package pressurized to + 53 psi. This pressure shall be maintained while all welds are inspected for leaks by swabbing with a liquid soap solution.

3.2.2 Detection of any bubbles shall be cause for rejection.

3.2.3 Rejected packages shall be repair-welded and again subjected to the pressure test.

3.2.4 Packages which pass the pressure test shall be subjected to the vacuum test.

3.3 Vacuum Test

3.3.1 Packages which pass the pressure test shall be evacuated to a pressure of 0.5 mmHg and sealed.

3.3.2 Packages which do not hold the above vacuum for a minimum of 4 hours shall be rejected.

3.3.3 Rejected packages shall be repaired and retested until the required vacuum is maintained.

4.0 ENGINEERING REVIEW

4.1 Packages which cannot be repaired without dis-assembly, shall be reviewed by the responsible Project Engineer prior to repair.

4.2. Packages which show evidence of leaks after three repairs and re-tests shall be reviewed by the responsible Project Engineer during the third re-test.

5.0 HANDLING AND STORAGE

5.1 Care must be exercised during handling and storage to avoid damage which might produce leaks or disrupt the lay-up in the package.

5.2. Packages shall be purged, pressurized with argon and sealed prior to storage.

MATERIAL SPECIFICATION

NO. XCM-1

Aluminum Foil for Composites

1.0 SCOPE

1.1 This specification establishes the requirements for aluminum foil to be used in production of diffusion bonded aluminum-boron composite material.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on the date of manufacture, unless otherwise specified, form a part of this specification to the extent specified herein.

Fed Spec QQ-A-250	- Aluminum Alloy Sheet & Plate
ASTM E8	- Tension Testing of Metallic Materials
ASTM E34	- Chemical Analysis of Aluminum and Aluminum Base Alloys
ASTM E227	- Spectrochemical Analysis of Aluminum
ASTM E112	- Estimating the Average Grain Size of Metals

3.0 REQUIREMENTS

3.1 Chemical Composition: The composition of the foil shall conform to the requirements of the applicable QQ-A-250 specification for the alloy required. Determination shall be made in accordance with ASTM E8.

3.2 Temper: The foil coiled sheet shall conform to "F" or "H" temper as indicated by a tear test. Tear or notch the edge of a foil strip 15-in. wide in two places approximately 1/2-in. apart. Holding the edge of the strip down with two fingers placed outside the notches, grasp the section between the notches and pull across the width of the roll. Under acceptable conditions the strip should tear clean the full width of the roll without breaking, and should not curl more than two coils.

3.3 Grain Size: The material shall possess an ASTM grain size of six or finer at foil converter gauge.

3.4 Defects: The foil shall be free of scratches, tears, excessive oxidation and other defects that would adversely effect the quality of the composite.

3.5 Dimensional Requirements

3.5.1 Thickness: The foil converter stock shall be supplied in nominal thickness required. Variations from the nominal thickness shall not exceed 10%.

3.5.2 Width: The foil converter stock shall be supplied in widths of 12-in., 15-in., 18-in., 24-in., 48-in., and 54-in. The tolerances for each width are shown in the following table.

<u>Ordered</u>	<u>Width</u> <u>Tolerance</u>
Under 30-in.	1/32-in.
Over 30-in.	1/16-in.

3.5.3 Sheet Profile: Maximum thickness variation from edge to edge of sheet shall be no more than 2%. The desired profile is symmetrical with the maximum thickness in the center of the sheet.

3.6 Coil Size and Condition

3.6.1 Coil Size: The maximum outside diameter shall be 25 inches.

3.6.2 Oscillated Wraps: Oscillated wraps shall not exceed 1/8-in. in the body of the coil and 1/4-in. in the first inch of build-up.

3.6.3 Coil Edge Quality: The coils shall have slit edges free of edge cracks and slivers.

3.6.4 Winding: The coils shall be wound tight enough to prevent telescoping or cinching during unwind or handling.

3.6.5 Splices: The splices are to be identified with no more than three splices per coil.

3.7 Surface Condition

3.7.1 Water and Oil Stain: Visible water or oil stain is not permitted on the metal surface.

3.7.2 Surface Uniformity: Surface roughness and appearance must be uniform from one edge of the sheet to the other. Broken matte is not acceptable.

3.7.3 Surface Residue: The surface should possess a slight amount of oil such that water when sprayed onto the foil will bead up. If water clings to the surface of the foil, the surface is excessively dry and is not acceptable.

Upon quickly unrolling a layer of foil and smelling the surface, any burning sensation to the nostrils is not acceptable.

Surface contamination is not acceptable. A clean white tissue should not show discoloration when rubbing the surface of foil a minimum distance of six inches.

4.0 PREPARATION FOR DELIVERY

4.1 The 48-in. and 54-in. wide coils are to be packed in wooden boxes and axially supported not to exceed 10,000 lbs. gross weight. A resilient material should be packed against the coil edges and circumferentially around the coil to cushion it during in-transit vibrations.

4.2 The 12-in., 15-in., 18-in., 24-in. wide coils are to be packed in wooden boxes and axially supported. It is permissible to pack more than one coil in a box provided the edges are adequately protected and no edge damage incurred. A resilient material should be packed against the coil edges and circumferentially around the coil to cushion it during in-transit vibrations.

4.2.1 The coil shall be positioned within the box so that the wraps will unwind from the top when in the horizontal position.

4.3 Package Marking: Each package shall carry marking including the following information :

- a) Alloy and Temper
- b) Gauge and Width
- c) MMA Order Number
- d) Vendor Lot Number
- e) Gross and Net Weights

5.0 EXCEPTIONS

Any exceptions to this specification must first have prior approval of the Martin Marietta Aluminum Research and Development Department.

MATERIAL SPECIFICATION

NO. XCM-2

Boron Monofilaments

1.0 SCOPE

1.1 Scope: This specification establishes the requirements for continuous boron monofilament. The material specified herein is a composite filament consisting of a tungsten core upon which boron is vapor deposited. The resultant material is a high modulus-low density filament which is used as the reinforcement in composite structures.

1.2 Classification: The boron filaments may be coated or uncoated as specified on the purchase order and are classified as follows:

TYPE I boron filaments, uncoated

TYPE II boron filaments with a .0002 inch thick coating of silicon carbide.

2.0 APPLICABLE DOCUMENTS

2.1. The following documents (and subsidiaries thereof) of issue in effect on date of invitation for bid, unless otherwise indicated, form a part of this specification to the extent specified herein.

STANDARDS: Fed. Test Method Std. No. 400 - Plastics, Methods of Testing.

PUBLICATIONS: AFML-TR-274 Testing Techniques for Filament Reinforced Plastics - Session I - Boron Filaments and Composites - Their Evaluation and Potential.

2.2. Publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting activity.

3.0 REQUIREMENTS

3.1 Material Requirements: The boron monofilament shall be supplied on spools with a diameter of not less than eight inches, Hubbard IM20 or equivalent. Each spool shall contain 35,000 ft $\pm$ 10 percent of boron monofilament, if spliced material is specified. Unspliced random lengths (3000 ft - minimum length) wound separately on individual spools are permissible as an alternate to the 35,000 ft spliced spools.

3.2 Splicing Requirements: Splicing of the boron monofilaments shall be permitted to achieve the desired spool lengths. Such splicing shall be performed using the manufacturer's best techniques and with the additional requirements that:

- a) The splices shall be conspicuously marked to assure easy identification of their location by the filament user.
- b) The maximum number of splices shall not exceed ten per filament spool.
- c) The method of identification used to mark each splice, and the number of splices in each spool shall be recorded by the filament manufacturer and furnished to the user with each spool.

3.3 Winding Requirements: The boron monofilaments shall be wound on the spools using uniform tension to assure a transportable, handleable and usable material. All filaments shall be level wound with a minimum spacing between adjacent wraps, and shall be free to unwind without restrictions caused by overlapping or crossed over filaments.

3.4 Liner Interleaves: The filaments shall be wound with a paper liner interleaf inserted at the completion of each level wind. It is intended that the liner material be of such stock that no contaminants are imparted to the filament surfaces.

3.5 Mechanical Property Requirements: The boron material supplied shall meet the requirements of Table I.

TABLE I - Boron Filament Properties
Minimum Requirement

<u>Class</u>	<u>I</u>	<u>II</u>	<u>III</u>	<u>IV</u>
Property				
Lot Average UTS (ksi)	450	450	500	450
Spool Average (ksi)	400	350	450	400
Prod Run Average (ksi)	400	350	450	400

TABLE I - Boron Filament Properties Minimum Requirement (continued)

<u>Class</u>	<u>I</u>	<u>II</u>	<u>III</u>	<u>IV</u>
*Any Single Test	400	350	450	400
Modulus (10^6 psi)	(min) 55	(min) 55	(min) 55	(min) 55
Diameter (inches)	$\pm .0002$	$\pm .0002$	$\pm .0002$	$\pm .0002$
Density Maximum lbs/in ³	.098	.1	.095	.097
Twist, Max. Acceptable Turns/10 ft.	1	1	1	1

Classification: This material is classified by boron filament diameter as follows:

Class I 0.004-in. diameter boron filaments
 Class II 0.0042-in. diameter borsic ⁽¹⁾ filaments
 Class III 0.0056-in. diameter boron filaments
 Class IV 0.0057 in. diameter borsic ⁽⁹⁾ filaments

(1) boron filament with a thin coating of silicone carbide.

NOTE: All classes have single layer, unidirectional filaments.

* If any single tensile test is less than 400 ksi (350) in any production run, the coefficient of variation of all tensile tests in that production run shall not exceed 15%. No more than one individual test per production run shall be below 335 ksi (275).

Modulus of elasticity, tension, lot ave.	psi	57×10^6	55×10^6
Diameter, inch		0.0039 ± 0.0002	0.0042 ± 0.0002
Twist, maximum acceptable, turns/10 ft		1	1
Density, maximum lbs/in ³		0.098	0.100

3.6 Documentation Requirements: The supplier shall furnish with each shipment two (2) copies of a certificate of conformance confirming that all material in the shipment complies with the requirements of the specification.

3.6.1 Inspection records of examinations and tests shall be kept complete and available to purchaser. These records shall contain all data necessary to determine compliance with the requirements of this document.

3.7 Workmanship Requirements: The boron monofilaments furnished in compliance with this specification shall be a uniform high quality product free from contamination of surface defects which would adversely affect the suitability for its intended purpose.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection: Unless otherwise indicated, the supplier is responsible for the performance of all inspection and test requirements specified herein. The supplier may use his own facilities or any commercial laboratory acceptable to purchaser. Purchaser reserves the right to perform any or all of the inspections set forth herein where such inspections are deemed necessary to assure that the material to be furnished conforms to the prescribed requirements.

4.1.1 Before the material is accepted, purchaser or its material processor shall verify that:

- a) the material identification is correct.
- b) the quantity is correct
- c) the required documents are received.
- d) the test data shows the material to be within the requirements of this specification.

4.2. Certificate of Conformance: The supplier shall furnish with each shipment two (2) copies of a certificate of conformance confirming that all material in the shipment complies with the requirements of this specification.

4.2.1 Conformance Certification Data: The certificates of conformance furnished in accordance with the requirements of 4.2 shall include the following information:

- a) Manufacturer's name
- b) Manufacturer's stock or lot designation
- c) Material specification number, title and revision
- d) Purchase order number
- e) Date and description of tests performed
- f) All required test results
- g) Results of any retests

4.3 Test Methods and Procedures

4.3.1 Sampling: All material to be used for mechanical property testing will be taken from the ends of each production run. A production run is defined as all the boron monofilament produced continuously by a single reactor.

4.3.2. Tensile Tests: Ten (10) tensile tests shall be performed for each filament production run. Each tensile specimen shall have a one inch gage length as a minimum and shall be tested in accordance with the test procedures defined in AFML-TR-66-274-Testing Techniques for Filament Reinforced Plastics. All tension test results for all production runs on a single shipping spool shall be averaged to determine spool averages. These spool averages shall meet the requirements of Paragraph 3.5. Other suitable methods agreed upon by purchaser and the manufacturer may be substituted for this technique.

4.3.3. Modulus of Elasticity, Tension: The tension modulus of elasticity of the boron monofilaments shall be determined by tensile specimens using long (10 inches or greater) lengths from which stress strain values can be obtained. These specimens shall be tested in accordance with test procedures which are defined in AFML-TR-66-274-Testing Techniques for Filament Reinforced Plastics. At least one tension modulus test will be performed for each production run. All test results will be averaged to obtain the modulus of the material on each shipping spool.

4.3.3.1 The total number of spools shipped at one time by the filament manufacturer to purchaser or its material processor constitutes a manufacturer's lot. The average modulus for each manufacturer's lot shall meet the requirements of Paragraph 3.5 Manufacturer's lot average is defined as the average of all spool modulus values included in the shipment. Acceptance of this filament material shall be based upon manufacturer's lot average of 57×10^6 psi or greater, provided that no individual modulus

test result is less than 55×10^6 psi. Other suitable methods agreed upon by purchaser and the manufacturer may be substituted for this technique.

4.3.4 Diameter: The diameter of the boron monofilaments shall be measured in at least 3 places for every production run. Each of these measurements shall be within the tolerances specified in this document. The measurements shall be made metallographically using a calibrated eyepiece. Other suitable methods agreed upon by purchaser and the manufacturer may be substituted for this technique. Mean diameter and deviation from the mean shall be reported for each production run, but acceptance of the material will be based upon all measurements meeting the requirements of Table I.

4.3.5 Density: One (1) density test shall be performed for each spool of material supplied. These tests shall be performed in accordance with Federal Test Method Standard No. 406, Method 5011 or 5012. Average density of the manufacturer's lot will be obtained from these results and shall meet the requirements of Paragraph 3.5. Other suitable methods agreed upon by purchaser and the manufacturer may be substituted for this technique.

4.3.6 Twist: One (1) Twist Test shall be performed for each spool of material supplied. These tests shall be performed as follows:

- a) Unwind under tension approximately 30 feet of filament from the free end of the spool without permitting the remaining filament to untwist.
- b) Cut and discard the first 30 feet.
- c) Remove, without permitting to untwist, a ten foot section of the remaining filament.
- d) Secure in a vertical position
- e) Free the lower end and note the number of revolutions made about the vertical axis by the free end.
Results of the Twist Test shall be reported for each spool of material in the shipment and shall meet the requirements of Paragraph 3.5.

4.4 Retests: If the material fails any one of the tests specified in Paragraph 4.3, the test shall be repeated using the procedures and requirements defined in Paragraph 4.3. If the retest fails, the material may be rejected by purchaser. Material which fails two retests shall be rejected.

4.5. Inspection of Product: The supplier shall make such visual examinations as are necessary to determine conformance to the workmanship requirements of 3.7.

4.6. Quality Conformance Inspections: Quality conformance inspections for certification of conformance shall consist of all the tests and inspections included in Section 4.0 of this document.

5.0 PREPARATION FOR DELIVERY

5.1 Preservation and packaging of all material furnished under this specification shall be sufficient to afford adequate protection against contamination by dirt, grease, oils and other harmful substances which would adversely affect the diffusion bonding qualities of the filaments.

5.2 All material shall be packaged in such a manner as to prevent physical damage during handling, shipping and storage and shall be packaged in accordance with the manufacturer's best commercial practices.

5.3. A complete paper liner, as specified in 3.4, shall be placed over the exposed filaments on each spool. In addition, a protective foamed plastic outer cover shall be used to protect the filaments from damage.

5.4. Each shipment package or container shall contain only material from the same manufacturer's lot. Shipment packages may contain more than one spool of filaments.

5.5. Each shipping package or container shall be marked with the following information:

Manufacturer's name, trademark or symbol

Manufacturer's lot number

Purchaser number, title and revision, purchase order number.

Number of spools in lot.

5.6 Each spool shall be marked with the following information:

Manufacturer's name, trademark or symbol
Harvey specification number, title and revision
Manufacturer's lot number, spool number
Material identification and date of manufacture
Approximate length of material on spool
Diameter of filament and tolerance
Average ultimate tensile strength
Modulus of elasticity, tension, psi, density, lbs/in³
Purchase order number

6.0 NOTES

6.1 This material is intended to be used for the fabrication of high performance structural composites for use in airframe and aerospace primary structures and in similar assemblies where high stiffness and strength to weight ratios are required.

6.2. Ordering Data: Procurement documents shall specify the following data:

- a) Number, title and revision of specification.
- b) Quality and description of material.
- c) Specify spliced material or unspliced random lengths.
- d) Specify fully all deviations from this specification.

6.3 This specification supersedes all previous boron filament specifications as of the date of issue.

6.4 Definitions:

6.4.1. Spool Average: The average of all measurements required on the material wound on a single shipping spool.

6.4.2. Production Run: All the boron filament produced continuously by a single reactor.

6.4.3. Manufacturer's Lot: All the material shipped at one time by the filament manufacturer to purchaser or its material processor.

6.4.4 Coefficient of Variation = $\frac{\text{Standard Deviation}}{\text{Arithmetic Mean}} \times 100$
(Average)

PROCESS SPECIFICATION
NO. XCP-7

Welding Vacuum Packages for Diffusion Bonding Composites

1.0 SCOPE

1.1. This specification establishes the requirements for welding steel vacuum packages for diffusion bonding composites.

2.0 APPLICABLE SPECIFICATIONS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 MMA Spec XCPA-4 - Lay-up and Packaging of Constituents for Diffusion Bonded Composites

2.1.2 AWS Spec A5.9-69 Class ER 308 - Bare Stainless Steel Welding Rod

2.1.3 AWS Spec A5.14-69 Class ERNiCrFe-5 - Nickel Alloy Welding Rod (INCO 62)

3.0 REQUIREMENTS

3.1 Welding shall be performed in accordance with the requirements of the applicable drawing.

3.2 Unless otherwise specified, the GTA welding process shall be used.

3.3 For the mild steel portions of the package, the filler rod used shall conform to AWS Spec A5.9-69, Class ER 308.

3.4 For the stainless steel closure the filler rod used shall conform to AWS Spec A5.14-69, Class ER NiCrFe-5.

3.5 Welding shall be performed by a certified weldor using qualified equipment and procedures.

3.6 Weld quality shall conform to the requirements of the drawing and shall be such that the package is capable of passing the leak test.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 All completed vacuum packages shall be tested for leaks by filling with argon to a slightly positive pressure and applying a soap solution to the seams to detect escaping argon.

5.0 STORAGE AND HANDLING

5.1 Care shall be exercised during handling welded packages to avoid cracking welds especially at fitting attachments.

5.2. Packages shall be evacuated purged with argon gas and sealed prior to storage.

6.0 RECORDS. The date and time of completion of welding of closures shall be recorded on the job ticket.

PROCESS SPECIFICATION

NO. XCPA-1

PREPARATION OF ALUMINUM FOIL FOR PRODUCTION OF DIFFUSION BONDED COMPOSITES

1.0 SCOPE

1.1 This specification establishes the requirements for preparation of aluminum foil for production of filament reinforced aluminum matrix composite sheet by the diffusion bonding process.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on the date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 Aluminum Sheet and Foil

2.1.1.1 QQA-250/1d - 1100 Al Alloy Plate, Sheet & Foil

2.1.1.2 QQA-250/11d-6061 Al Alloy Plate, Sheet & Foil

2.1.1.3 FED STD 245C - Receiving Inspection for Al Foil

2.1.1.4 MMA XCM-1 - Aluminum Foil for Composites

2.1.2 Inspection

2.1.2.1 MMA XCQ-1 - Visual Examination of Processed Al Foil

2.1.2.2 MMA XCQ-2 - Surface Resistivity Measurements for Al Foil

3.0 REQUIREMENTS

3.1 Material - Aluminum foil shall conform to the requirements of the applicable Federal Material Specification. Receiving inspection shall be performed in accordance with the requirements of FED STD 245C.

3.2 Cleaning - Aluminum foil shall be solvent cleaned in methanol.

3.3 Lacquering - After solvent cleaning, both surfaces of each piece of foil shall be sprayed with styrene to protect the surfaces.

3.4. Final Cleaning - If the cleaned foil is subjected to excessive and improper handling, or storage times are in excess of the specified limits (see para. 4.4), it must be re-cleaned until it is free of all foreign matter and conforms to the limits of surface resistivity (para. 4.4).

3.5 Handling - Care must be exercised to prevent tearing, wrinkling and contamination of foil during all steps of preparation. For each step subsequent to chemical cleaning, clean white gloves must be worn when the foil is handled. The requirements of para. 5 apply to temporary storage as well as long time storage.

3.6. Quality Requirements - All prepared foil shall be free of obvious defects such as dirt, tears, holes, non-uniform color, etc., as determined by visual inspection.

Thickness variations shall not exceed 5% of the nominal foil thickness as determined by the method(s) designated in para. 4 of this specification.

Surface resistance shall not exceed 300 microhms as determined by the method(s) designated in para. 4 of this specification.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Purchased Foil Receiving Inspection - Before commercial aluminum foil is processed for composite production, it must be inspected for proper alloy, thickness, quality and size in accordance with MIL-STD-245C.

4.2. Thickness Measurements - Foil thickness measurements shall be made with a micrometer or other approved measuring instrument to assure conformance with drawings.

4.3. Surface Resistivity - Surface measurements shall be made in accordance with the requirements of H.A. Spec. XCQ-3 and quality shall conform to para. 3 of this specification.

4.4 Conformance Certification - Aluminum foil accepted for production of composites in accordance with the foregoing provisions of this specification shall be so certified by the responsible quality control personnel. The certificate of conformance shall contain the identification numbers for each piece along with all pertinent quality data.

5.0 IDENTIFICATION AND STORAGE

5.1 Identification - Aluminum foil prepared and accepted for production of composites shall be identified as to the following:

Aluminum alloy and nominal thickness
Procurement order number
Certification number and date
Job number

5.2 Storage - Aluminum foil prepared for production of aluminum-boron composites must be maintained in an environment which will protect it from mechanical damage and re-contamination. Such environment may consist of a "clean-room" and/or local protection such as a dust-free container. In the event that the foil becomes contaminated, it must be re-cleaned.

All foil stored more than 5 days before consolidation into a composite must be re-tested for surface resistivity. All re-cleaned foil must also be re-tested for surface resistivity.

PROCESS SPECIFICATION

NO. XCPA-2

PREPARATION OF BORON FILAMENTS FOR PRODUCTION OF DIFFUSION BONDED COMPOSITES

1.0 SCOPE

1.1 This specification establishes the requirements for preparation of boron filaments for production of metal matrix composites by diffusion bonding.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of production, unless otherwise specified, form a part of this specification to the extent specified herein.

2.1.1 MMA Specification XCM-2 - Boron Monofilament

3.0 REQUIREMENTS

3.1 Material- Boron filaments shall conform to the requirements of MMA Specification XCM-2.

3.2 Selection of Filaments- Each lot of spools of filaments accepted by Receiving Inspection shall be segregated by filament strength. Spools will be segregated into the following groups of average tensile strength as determined by the supplier:

Group 1 - 425 ± 25 ksi

Group 2 - 475 ± 25 ksi

Group 3 - 525 ± 25 ksi

Group 4 - 575 ± 25 ksi

3.3 Cleaning- If required on the job order drawing, filaments shall be cleaned by running the filament through a bath of methanol during production of boron filament mats.

3.4 Quality Requirements

3.4.1 Filament Strength- The average strength of the filaments on each spool shall exceed the minimum required by MMA Spec XCM-2.

3.4.2 Filament Length- No more than three splices per spool are permissible.

3.4.3 Filament Winding- Filament shall be sufficiently free of defects and possess sufficient transverse strength to permit satisfactory cleaning and winding.

3.4.4 Cleanliness- Filaments shall be free of foreign matter such as grease, dirt, loose scale.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Purchased Filaments Receiving Inspection- Before filaments are processed for composite production, each spool must be inspected for proper diameter, strength, unspliced length, total length, net weight, spooling, etc., as required by MMA Spec. XCM-2. This information shall be tabulated and recorded for each shipment.

4.2 Cleaning- All boron filaments shall be checked at intervals sufficient to assure that the cleaned boron filament will be free of foreign matter, such as grease, dirt and loose scale.

4.3 Tensile Testing- If required on the composite work order drawing, samples of filaments shall be collected during production of mats (MMA Spec. XCPA-3) and tested by preparing specimens from the end of the mat and determining the bundle strength.

4.4 Certificate of Conformance- Boron filaments accepted for production of composites in accordance with the foregoing provisions of this specification shall be so certified by the responsible Quality Control personnel. The certificate of conformance shall contain the identification information in accordance with para. 5 and shall be initialed by the inspector.

5.0 IDENTIFICATION AND STORAGE

5.1 Identification- Each spool of boron filaments accepted for production of composites shall be identified as to the following:

- Filament type (boron, Borsic, etc)
- Filament diameter
- Supplier
- Procurement order number
- Job number
- Tensile strength group number

5.2 Storage- Boron filaments shall be maintained in an environment which will protect them from mechanical damage and contamination by foreign matter. Such environment may consist of a "clean-room" or local protection such as a dust-free container.

PROCESS SPECIFICATION

NO. XCPA-3

PRODUCTION OF BORON FILAMENT MATS FOR DIFFUSION BONDED COMPOSITES

1.0 SCOPE

1.1 This specification establishes the requirements for preparation of boron filament mats for production of metal matrix composites by diffusion bonding.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of production, unless otherwise specified, form a part of this specification to the extent specified herein.

2.1.1 XCPA-1 - Preparation of Aluminum Foil for Composite

2.1.2 XCPA-2 - Preparation of Boron Filaments for Composites

3.0 REQUIREMENTS

3.1 Materials- Only constituents specially prepared shall be used for production of mats. All constituent materials and supplies shall conform to applicable specifications listed in para. 2, unless otherwise specified.

3.2 Winding Equipment- Winding equipment shall be such that mats of proper length, width and filament spacing may be produced for the specified composite without splicing or piecing, unless otherwise specified. A backing such as teflon sheet shall be used so that the plastic binder may be easily stripped away after winding.

3.3 Filament Spacing- Filament spacing shall be that required on the composite work order drawing.

3.4 Mat Size- Mat size shall be that required on the composite work order drawing.

3.5 Filament Winding- Filament winding shall be performed so that filaments are uniformly spaced and parallel with no cross-overs or loose filaments. Adhesive tape may be used for temporarily securing the filaments, but must be removed prior to lay-up. Styrene shall be evenly applied to hold the filaments uniformly spaced and firmly in place until the diffusion bonding process is initiated.

3.6 Cutting, Handling and Storage of Mats- Mats shall be removed from the filament winding mandrel by cutting and handling in such a manner to avoid mechanical damage and contamination of the mat with grease, dirt or other foreign matter. Clean white gloves shall be worn when mats are handled, and mats shall be stored in a "clean room" or dust-free containers until ready for lay-up for diffusion bonding.

3.7 Quality Requirements- Filament mats shall be free of splits; stray, misaligned, crossed, broken or missing filaments; excessive or uneven build-up of plastic binder; dirt, grease and other foreign matter. Filaments shall be properly and uniformly spaced, and mat size shall be in accordance with requirements of the composite work order drawing.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Inspection of Prepared Constituents- All constituents shall have been inspected and approved in accordance with MMA Specifications XCPA-1 and XCPA-2.

4.2 Inspection of Filament Mats- Prior to lay-up for diffusion bonding of composites, filament mats shall be inspected for defects in accordance with para. 3.7. Defective or contaminated mats shall be rejected. Acceptable mats shall be certified by the responsible inspector.

5.0 IDENTIFICATION AND STORAGE

5.1 Identification- Boron filament mats accepted for production of composites shall be identified as to the following:

Filament type and supplier
Part number
Job number
Certification number and date

5.2 Storage- Mats approved for production must be stored in an environment that will protect them from mechanical damage and contamination by foreign matter. Such environment may consist of a "clean room" and/or local protection such as a dust-free container.

PROCESS SPECIFICATION

XCPA-4

LAY-UP AND PACKAGING OF CONSTITUENTS FOR DIFFUSION BONDED COMPOSITE

1.0 SCOPE

1.1. This specification establishes the requirements for lay-up of boron filament mats and prepared aluminum foil, and for packaging this lay-up for vacuum diffusion bonding.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on the date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1. Composite Constituents

2.1.1.1 XCPA-1, Preparation of Aluminum Foil for Composites

2.1.1.2 XCPA-3, Production of Boron Filament Mats for Composites

2.1.2 Package Components

2.1.2.1. Federal Spec. QQ-A-250, Aluminum Alloy (Bumper) Plate

2.1.2.2 ASTM Spec. A-167, Stainless Steel (Glide) Sheet (half-hard or better)

2.1.2.3 ASTM Spec. A-283, Carbon Steel (Cover) Plate

2.1.2.4 ASTM Spec. A-366, Carbon Steel (Seal) Sheet

2.1.3 Commercial

2.1.3.1 Graphite Parting Compound (Aquadag #8)

2.1.4 MMA Specification XCP-7 - Welding Steel Packages for Diffusion Bonding Composites.

3.0 REQUIREMENTS

3.1 Preparation of Steel Covers and Seal - Covers and seals conforming to ASTM Spec. A-283 and A-366 shall be fabricated in accordance with the applicable drawing designated on the composite work order drawing. Before each use for diffusion bonding, all components shall be inspected. Dimensions shall conform to the drawing. All interior and bearing surfaces of package components shall be free of grease, dirt and other foreign matter.

3.2 Preparation of Aluminum Bumper Sheets - Aluminum bumper sheets of any alloy conforming to QQA-250 shall be used in each package so that both covers are separated from the composite by a 1/8-in thick bumper sheet and so that at least one side of each composite sheet in a multiple lay-up is adjacent to 1/16-in. thick aluminum bumper sheet assembly. The bumper sheets shall be the same size as composite sheet. Prior to lay-up, each bumper sheet shall be inspected for defects and cleaned as required to insure that it is free of grease, dirt, and other foreign matter.

3.3. Preparation of Stainless Steel Glide Sheets - Unless otherwise specified, stainless steel glide sheets conforming to ASTM Spec. A-167 (0.025" max. thickness) shall be used to separate all aluminum surfaces except multiple layer composite sheet, within the package. The glide sheets shall be the same size as the bumper sheets. Prior to lay-up each glide sheet shall be inspected for defects and cleaned as required to insure that it is free of grease, dirt and other foreign matter. Both sides of each glide sheet shall then be sprayed with a light uniform coat of parting compound (Aquedag No. 8, unless otherwise specified).

3.4 Lay-up of Constituents and Package Components - Prepared aluminum foil and boron filament mats shall be laid up with bumper sheets and glide mats in the prepared bottom cover of the package in accordance with the composite work order drawing. In the event that the dimensions of the composite constituents are smaller than the inside dimensions of the cover, spacers of the required size and thickness shall be used to position the composite constituents for diffusion bonding. The proper sequence for lay-up is as follows:

- a. Spray-coat inside surface of package covers with graphite.
- b. Pre-place spacers in bottom cover.
- c. Lay first outside bumper sheet.

- d. Lay first sheet of prepared aluminum foil (cut to size)
- f. Lay sheet of filament mat (cut to size).
- g. Lay second sheet of prepared aluminum foil (cut to size)
- h. Repeat steps f and g for required number of layers of composite.
- i. Lay second sheet of coated glide sheet.
- j. Lay second sheet of bumper plate.
- k. Repeat steps d through i for required number of composite sheets per package.
- l. Lay second outside bumper sheet.
- m. Position top cover.
- n. Clamp package for welding.

3.5 Quality Requirements

3.5.1 Parallelism of Components - Package components shall be prepared and laid up in such a manner with the composite constituents that it will be possible to achieve the required composite sheet. Deviation from specified thickness and flatness of all components of a package shall not exceed the ability of the bumper sheets to accommodate such variation, so that pressure during diffusion bonding will be uniformly distributed on all parts of the composite constituents, thus producing a sound uniform composite sheet.

3.5.2 Filament Lay-up Pattern - The axis of filaments in each layer of mat shall be oriented in accordance with the composite work order drawing.

3.5.3. Glide Sheet Coating - The thickness and quality of the glide sheet coating shall be such that aluminum from either the foil or bumper sheets will not adhere during diffusion bonding.

3.5.4. Closure and Evacuation of Package - All packages must be sealed by welding and evacuated within one day after completion of lay-up. Welding shall be performed in accordance with MMA Specification XCP-7. Welded packages shall be tested for leaks.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Inspection of Package Components - All package components shall be examined before each use for damage during previous use, proper preparation and interior cleanliness.

4.2 Inspection of Composite Constituents - Foil and filament mats shall be examined before use for conformity to MMA. Specifications XCPA-1 and XCPA-2 respectively, and certification number shall be recorded on the package tag.

4.3 Inspection of Lay-up - During and after the lay-up procedure, checks shall be made to assure proper number of composite layers and sheets, proper sequence of lay-up of package components and composite constituents and proper assembly of package. A certificate of lay-up conformance shall be initialed by the responsible inspector.

4.4. Leak Test Certification - After the package is welded and leak tested, it shall be certified, if acceptable for diffusion bonding.

5.0 IDENTIFICATION AND STORAGE

5.1 Identification - All packages shall be tagged with the following information;

- Job Number
- Part Number
- Foil Certification Number
- Filament Mat Certification Number
- Lay-up Certification Number, Date and Initials
- Leak-test Certification Number, Date and Initials

5.2 Storage - Packages that have been certified shall be closed and evacuated within one day after certification. Between lay-up and evacuation, the package shall be stored in a "clean" room or a local dust-free container.

PROCESS SPECIFICATION

NO. XCPA-5

DIFFUSION BONDING OF ALUMINUM MATRIX COMPOSITES

1.0 SCOPE

1.1 This specification establishes the requirements for diffusion bonding filament reinforced aluminum matrix composites.

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on date of production, unless otherwise specified, form a part of this specification to the extent specified herein.

2.1.1 MMA. Spec. No. XCPA-4 - Lay-up and Packaging of Constituents for Diffusion Bonded Composites.

2.1.2 MMA. Spec. No. XCP-7 - Welding Steel Packages for Diffusion Bonded Composite.

3.0 REQUIREMENTS

3.1 Packages for Diffusion Bonding - Before diffusion bonding, a check shall be made to assure that the package has been certified for diffusion bonding.

3.2 Equipment

3.2.1 Presses - Presses used for diffusion bonding shall be capable of applying a minimum pressure of 6,000 psi uniformly and constantly over the pressing area of the package for a minimum of 30 minutes. Pressure variations throughout the diffusion bonding cycle shall not exceed 10%. The platens of the press shall be sufficiently parallel, flat and rigid so that the closure will remain constant within $\pm .02$ -in. throughout the entire pressing cycle.

3.2.2 Die Heaters - Die heaters shall be capable of heating the composite constituents uniformly and constantly to any selected temperature within the range of 500F to 1100F ± 20 F for any period within the range of 30 min. to 90 minutes.

3.2.3 Pre-Heaters - Pre-heaters shall be capable of heating the package uniformly and constantly to any temperature within the range of 600F to 1000F $\pm$ 50F for any period with the range of 30 min. to 240 minutes.

3.2.4. Vacuum Equipment - Vacuum equipment shall be capable of maintaining a vacuum of less than 0.5 mm. of mercury in the package during the heating and pressing cycles.

3.2.5 Package Transfer Equipment - If required, the package transfer equipment shall be capable of moving the package in and out of the press without mechanical damage to the package or its contents.

3.3 Tooling

3.3.1 Dies - Dies shall be fabricated in accordance with the applicable drawings. Existing dies selected for diffusion bonding shall be sufficiently large to apply uniform pressure and heat at the specified level over the pressing surface of the composite package. Dies shall be checked before each usage for damage from previous usage or storage.

3.4. Instrumentation

3.4.1. Pressure. - Pressure applied to the composite constituents during pre-heat and diffusion bonding shall be maintained with an accuracy of not less than $\pm$ 10% for outgassing and $\pm$ 10% for diffusion bonding. Pressure measurements shall be taken from press dials.

3.4.2 Temperature- Temperature of the composite constituents during pre-heat and diffusion bonding shall be maintained with an accuracy of not less than $\pm$ 50F for preheat and not less than $\pm$ 20F for diffusion bonding. Temperature measurements shall be made with iron-constantin thermocouples with appropriate read-out.

3.4.3 Vacuum - The vacuum maintained in the package shall be determined from a gauge with an accuracy of not less than .01 mm of mercury.

3.5 Diffusion Bonding Procedure

3.5.1 Outgassing. - The package shall be heated to a temperature of 800F $\pm$ 50F for a period sufficiently long to complete outgassing of the package components and composite constituents. A vacuum pump will be in operation during all phases of outgassing, sufficient to rapidly remove all volatilized matter. An external pressure of 14 psi $\pm$ 10% will be maintained on the package during all phases of outgassing to assure that all contents of the package, especially boron filaments, remain in their proper position in the package until the composite constituents are consolidated during the diffusion bonding operation. Outgassing will be considered complete

when a vacuum of less than .5 mm of Hg can be maintained for a period of at least 30 minutes.

3.5.2 Diffusion Bonding - After the package is completely outgassed, the temperature of the composite constituents shall be raised to 50F below the desired bonding temperature. (The bonding temperature shall be specified on the composite work order drawing) When the temperature has stabilized at this level, the pressure shall be increased to 6,000 psi $\pm$ 10% and the temperature shall be stabilized at the bonding temperature. This pressure and temperature shall be maintained for a minimum of 30 minutes.

3.5.3 Step Pressing - In the event that step pressing is required, the portion of the package which extends beyond the dies and which has not yet been pressed shall be pre-heated to a temperature 100F $\pm$ 50F below the diffusion bonding temperature. After pressing the initial increment, the package shall be indexed into the dies for pressing of the next increment. Overlap of increments for pressing should be a minimum of 2 inches. Diffusion bonding shall be accomplished for each increment in accordance with para. 3.5.2.

3.6 Removal of Composite from Package. - Care shall be exercised in removing the composite from the package to avoid mechanical damage. The seal weld shall be trimmed off as near to the weld bead as possible so that the package may be re-used. Composite sheet must be handled with care to avoid splitting, tearing, etc.

3.7 Records - Records of the diffusion bonding operation shall be maintained. These records shall contain the following:

Job Number
Part Number
Press No:
Die No:
Outgassing temperature, time, final vacuum
Diffusion bond temperature, time and pressure for each increment.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Certification of Equipment, Tooling and Instrumentation - Prior to diffusion bonding each package, all equipment, tooling and instrumentation shall be checked by responsible quality control personnel and certified satisfactory for diffusion bonding composites.

4.2 Certification of Processing Parameters - Prior to diffusion bonding the processing parameters shall be certified by the responsible quality control personnel, and periodic checks shall be made to verify all actual processing parameters. The quality control personnel shall initial the records of the diffusion bonding operation.

5.0 IDENTIFICATION AND STORAGE

5.1 Identification - All package components shall be identified by drawing number before storing. Composite sheet shall be marked along one or more edges to show the job number and item number.

5.2 Storage - Package components, equipment and tooling shall be stored in such a manner as to avoid mechanical damage, corrosion and contamination with dirt, oil, etc.

Composite sheet shall be stored in a "clean" room or in a dust free container.

MATERIAL SPECIFICATION

No. XCMA-1

Boron-Aluminum Composite Foil and Sheet

1.0 SCOPE

1.1 This specification establishes the requirements for diffusion bonded boron filament reinforced aluminum alloy matrix composite in the form of monolayer (thickness 0.009-in. or less) and multilayer sheet (thickness between 0.009 and 1.000-in.).

2.0 APPLICABLE DOCUMENTS

2.1 The following documents (and subsidiaries thereof) of issue in effect on the date of manufacture, unless otherwise indicated, form a part of this specification to the extent specified herein.

2.1.1 MMA Spec XCM-2 - Boron Filaments

2.1.2 ASTM Spec to be issued - Tensile Testing of B-Al Composite Sheet

2.1.3 FED-SPEC QQ-A-250 - Aluminum Alloy Plate and Sheet

2.1.4 FED-STD-245 - Tolerances for Aluminum Alloy and
Magnesium Alloy Wrought Parts

2.1.5 ASTM Spec. E94-68 - Recommended Practice for Radiographic
Testing

2.1.6 ASTM Spec. E214-68 - Recommended Practice for Immersed
Ultrasonic Testing by the Reflection Method Using
Pulsed Longitudinal Waves.

3.0 REQUIREMENTS

3.1 Filament

Boron filaments shall conform to MMA Spec. CM-2 or equivalent

3.2 Matrix Alloy

Aluminum matrix alloys shall conform to Federal Specification QQ-A-250 or equivalent.

3.3. Filament Percent by Volume

Filament content of the composite shall not vary from the specified content by more than $\pm 3\%$.

3.4 Filament Orientation

Deviation from the specified orientation of filament in any given layer shall not exceed one degree.

3.5 Dimensional Tolerances

Tolerances, unless otherwise specified shall conform to those in FED-STD-245.

3.6 Properties

The tensile properties of the composite shall meet the requirements specified in the purchase order.

3.7 Finish

Unless otherwise specified, the material will be furnished in the mill finish.

3.8 Surface Defects

The surface shall be free from cracks, scratches, folds, wrinkles, laps, indentations, edge delaminations, foreign objects, or other defects which would adversely affect the serviceability of the material.

3.9 Internal Defects

The material shall be free of deleterious voids, delaminations, stray filaments, broken filaments, filament and ply misalignment, and foreign matter.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Radiography

If required by the purchase order, composites will be examined by X-ray for misaligned, stray and broken filaments. X-ray testing will be performed in accordance with ASTM Spec. E94-68.

4.2 Ultrasonic C-scan

If required by the purchase order, composites will be examined by ultrasonic C-scan for delaminations. Ultrasonic C-scans will be made in accordance with ASTM Spec. E214-68

4.3 Tensile Testing

Tensile properties, as required by the purchase order, will be determined in accordance with ASTM Specification (To be issued).

5.0 MARKING, PACKING AND SHIPPING

5.1 Marking

The material will be legibly identified with the following information, unless otherwise specified:

- Purchase order number.
- Manufacturer's name
- Material description.
- Quantity and unit size.
- Lot number.
- Item number.

5.2. Packing

Unless otherwise specified the material will be coated with a light oil and packed in a wooden crate sufficient for protection against damage under normal shipping conditions.

5.3 Shipping

Unless otherwise specified, shipment will be made F.O.B. Torrance, California via United Parcel Delivery.