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# FABRICATION AND EVALUATION OF LOW FIBER CONTENT ALUMINA FIBER/ALUMINUM COMPOSITES

By: J. E. Hack and G. C. Strempek

## FINAL REPORT

Prepared for:

NASA-LEWIS RESEARCH CENTER
Contract NAS3-21371


SUBMITTED BY

FIBER MATERIALS, INC.
BIDDEFORD INDUSTRIAL PARK
BIDDEFORD, MAINE 04005

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16. Abstract

The mechanical fabrication of low volume percent fiber, polycrystalline alumina fiber reinforced aluminum composites was accomplished. Wire preform material was prepared by liquid-metal infiltration of alumina fiber bundles. The wires were subsequently encapsulated with aluminum foil and fabricated into bulk composite material by hot-drawing. Extensive mechanical, thermal and chemical testing was conducted on preform and bulk material to develop a process and material data base. In addition, a preliminary investigation of mechanical forming of bulk alumina fiber reinforced aluminum composite material was conducted.

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## ABSTRACT

The mechanical fabrication of low volume percent fiber, polycrystalline alumina fiber reinforced aluminum composites was accomplished. Wire preform material was prepared by liquid-metal infiltration of alumina fiber bundles. The wires were subsequently encapsulated with aluminum foil and fabricated into bulk composite material by hot-drawing. Extensive mechanical, thermal and chemical testing was conducted on preform and bulk material to develop a process and material data base. In addition, a preliminary investigation of mechanical forming of bulk alumina fiber reinforced aluminum composite material was conducted.

## FOREWORD

This work was performed by the Advanced Materials Development Laboratory of Fiber Materials, Inc., of Biddeford, Maine, for the NASA-Lewis Research Center, Cleveland, Ohio, under Contract No. NAS3-21371 from May 19, 1978 to August 19, 1979. The NASA Technical Monitor was Dr. R. G. Barrows. Mr. John E. Hack was the FMI program manager with G. C. Strempek assisting as program engineer. Other FMI personnel who assisted on the program are listed below:

Dr. Roger T. Pepper, Technical Advisor
Mr. Thomas C. Daley
Ms. Elizabeth N. Haydock
Mr. Michael Regan

## SUMMARY

The objective of this program was to study the fabrication of low volume percent, alumina/aluminum ($Al_2O_3$/Al) composite material in order to establish a process and property data base for the material. Specifically the work involved 1) developing a preform $Al_2O_3$/Al wire; 2) optimizing parameters for the primary mechanical fabrication of wire preforms into bulk form; 3) extensive mechanical, thermal and chemical testing of bulk composite material; and 4) preliminary trials on secondary fabrication processes.

Task I involved the optimization and fabrication of $Al_2O_3$/1100 Al composite wire at 20% $Al_2O_3$ fiber level. A Ti/B CVD coating process was used to induce wetting of the FP $Al_2O_3$ fiber bundles by the aluminum. It was found that the desired 20 v/o fiber level could not be achieved through the liquid-metal infiltration process alone and subsequent encapsulation of wire preforms with aluminum foil was necessary. In addition to wire development, parameters were established for the hot drawing of the wire preforms into consolidated composite materials during this task.

Task II was concerned with the mechanical testing of the material developed in Task I. The data obtained was used to form a process and material data base in conjunction with 30 and 40 v/o fiber material data generated under NASA contract NAS3-21013. Thermal and chemical analyses were also performed on the $Al_2O_3$/Al material. Testing was carried out on both as-drawn material and material which had been exposed to 300°C for 1000 hours in air. Some test data was generated at 300°C.

Task III was directed towards a very preliminary investigation of secondary mechanical fabrication processes for $Al_2O_3$/Al composites. Hot-rolling and upset forming of bulk material was performed. The method which showed the most promise was hot-rolling of the material at 90° to the fiber orientation.

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## 1.0 INTRODUCTION

Metal matrix composites offer the potential for excellent structural materials in a wide range of applications. Generally, they exhibit high specific strengths and moduli over a wide temperature range for use in such areas as substructures, truss members, stiffeners and pressure vessels. Metal matrix composites also enjoy property advantages over present organic matrix composites in temperature capabilities, environmental stability, off-axis properties and design flexibility in joining.

A review of the available polycrystalline $Al_2O_3$ fibers[1] showed that the only alumina fiber suitable for use in aluminum matrix composites is the DuPont FP continuous alumina yarn.[2] The FP fiber is pure polycrystalline aluminum oxide and, consequently, is chemically inert to metals such as aluminum, is highly stable at high temperatures, and suffers no oxidation damage on exposure to air. Table 1 gives the FP alumina fiber properties. The FP fiber retains essentially 100% of its strength and modulus at approximately 500°C, which is the probable maximum use temperature of aluminum matrix composites.

FP alumina fibers are not readily wetted by aluminum. Two main approaches have been taken for the liquid-metal infiltration of fiber bundles using special wetting techniques. In the first approach, Dhingra and co-workers[2] employed lithium additions to aluminum to form $LiAlO_2$. This compound promotes wetting by lowering the interfacial tension between the aluminum and aluminum oxide and yields strong fiber/matrix interfacial bonding. For a second approach, work at the Aerospace Corporation[1] has shown that the Ti/B wetting process[3] developed for graphite fiber reinforced aluminum is a viable process for making aluminum oxide fiber reinforced composites. The Ti/B wetting process has a significant advantage over the lithium addition technique since only very small additions of Ti and B are needed and commercial cast and wrought composition aluminum alloys can be used.

Dhingra et al[2] have shown that FP/magnesium matrix composites can be successfully hot worked by extrusion, hydrostatic extrusion,

TABLE 1

FP ALUMINA FIBER PROPERTIES

| | |
|---|---|
| Tensile Strength (Type I) | 200 Ksi |
| Tensile Modulus | $50 \times 10^6$ Psi |
| Melting Point | $2045^{\circ}C$ |
| Density | 3.95 $g/cm^3$ (0.143 $lb/in.^3$) |
| Fiber Diameter | 15-25 microns |
| Projected Cost | $25/lb. |

rolling and forging. Hydrostatic extrusion results in minimum surface shearing of billets during extrusion, thus reducing the chances of fiber fragmentation due to non-axial deformation. Extruded FP/magnesium composites showed a three times increase in tensile elongation and a 25% loss in tensile strength due to 30% of the fibers being broken to less than the critical length for efficient fiber reinforcement. Forging reductions up to 60% were performed with no significant losses in strength and modulus. Composites were also clad with stainless steel and reduced in thickness by rolling up to 70% through a six step reduction process. No fiber breakage was observed for rolling reductions up to 50%.

Under previous NASA contract (NAS3-21013), the Ti/B wetting process was adapted to produce continuous wire of FP alumina fibers in an 1100 aluminum matrix. It was found that a small amount of Mg (0.3% by weight) added to the melt greatly increased wetting between the fiber and matrix phases. Most Al alloys contain at least this amount of Mg. Prior to work with A201 aluminum, which does contain 0.3% Mg, yielded very consistent and complete wetting of $Al_2O_3$ fibers in conjunction with the Ti/B wetting process. Wire was produced at nominal volume percents of 30 and 40 v/o fibers on contract NAS3-21013.

Development of a hot drawing process for the consolidation of preform wires into bulk material was also accomplished under contract NAS3-21013. Parameters were also established for the adaption of FMI's pultrusion process for graphite-aluminum composites to aluminum oxide aluminum material, and extensive mechanical testing was performed on the material fabricated by pultrusion. In addition, some secondary forming processes were analyzed. Creep forming and rolling studies were performed with limited success being achieved with hot rolling.

The results presented in this report deal with a follow-on program to that just outlined where low volume percent $Al_2O_3$/Al

wire was developed, fabricated into bar stock and subsequently tested. The work was broken down into three major tasks.

During Task I, the Ti/B wetting process adapted under contract NAS3-21013 to produce continuous wire of FP alumina fiber was modified to achieve low volume percent fiber. This was achieved through the simultaneous infiltration of seven fiber tows forming one large diameter wire. As before, a magnesium modified 1100 aluminum matrix was used to aid wetting. Wire was produced at a nominal volume percent of 20 v/o fiber.

The preform wire was then consolidated into bulk material via the hot drawing process. This process was adapted for use with $Al_2O_3$ during the earlier program and has been found to be highly successful in such applications. Sufficient material was processed during this task to allow for completion of Tasks II and III, and for delivery of material to NASA for independent testing.

Task II was involved with establishing a data base for the low volume percent $Al_2O_3$/1100 Al material developed and fabricated in Task I. Extensive mechanical testing at room and elevated temperature (300°C) was performed. The effect of long time exposure at 300°C was also determined. Mechanical property evaluations conducted include both longitudinal and transverse tensile, compression, flexure and fatigue testing at room temperature. Stress rupture, longitudinal tensile and transverse tensile testing was performed at elevated temperatures. In addition, chemical analysis was performed on preform material and thermal expansion behavior was characterized.

Task III was concerned with a preliminary analysis of secondary forming processes on 20 v/o fiber material. Both upset forming and rolling were performed on test coupons at both room temperature and elevated temperatures. Upset forming in such a way that the fibers were perpendicular to the applied force showed no promise even at slow forming rates. Rolling studies showed that hot-rolling transverse the fiber direction yielded suprisingly good results.

In reporting the results of fabricating and testing the $Al_2O_3$/1100 Al material, data generated during the earlier program, NASA contract NAS3-21013, has been included. Hence a more complete picture for the $Al_2O_3$/1100 Al material's response to mechanical deformation can be presented.

## 2.0 TASK I - PRIMARY FABRICATION DEVELOPMENT FOR LOW VOLUME PERCENT $Al_2O_3$/1100 Al MATERIAL

Task I involved the development of primary fabrication techniques for low volume percent $Al_2O_3$/1100 Al composite material. These techniques included a chemical processing technique which produced wetting between the FP fiber bundles and the 1100 aluminum matrix alloy to produce a continuous composite wire, and a mechanical hot drawing process for the consolidation of the preform wires into bulk material. After both techniques were optimized, sufficient $Al_2O_3$/1100Al material was fabricated at a nominal level of 20 v/o fiber to permit extensive testing and preliminary secondary fabrication studies. A 1000 hour heat treatment was performed on selected test material to determine the effects of long term thermal exposure.

### 2.1 Composite Wire Development

The Ti/B infiltraiton unit developed during the previous program for the fabrication of aluminum oxide/aluminum material was used as a starting point for this work. The coating process involves the chemical vapor deposition (CVD) of the titanium/boron coating on the $Al_2O_3$ fibers by the reduction of titanium tetrachloride ($TiCl_4$) and boron trichloride ($BCl_3$) with zinc vapor. The Ti/B coated yarn is then pulled through an in-line melt to yield a composite wire.

The liquid metal infiltration unit, shown schematically in Figure 1, was modified to allow for the simultaneous collimation and infiltration of multiple $Al_2O_3$ fiber tows. This involved expanding the yarn creel assembly to accomodate up to nine spools at one time. The round-bottom pulleys developed on NASA contract NAS3-21013 were used in the window box and melt pulley assemblies to minimize fiber damage from the handling system.

The FP $Al_2O_3$ fiber used throughout this program was characterized at DuPont and a copy of the data is included in Table 2. As indicated in the First Four-Month Technical Narrative on NASA-Lewis contract NAS3-21013, actual fiber bundle strengths are approximately 140,000 Psi, as opposed to the single filament test values

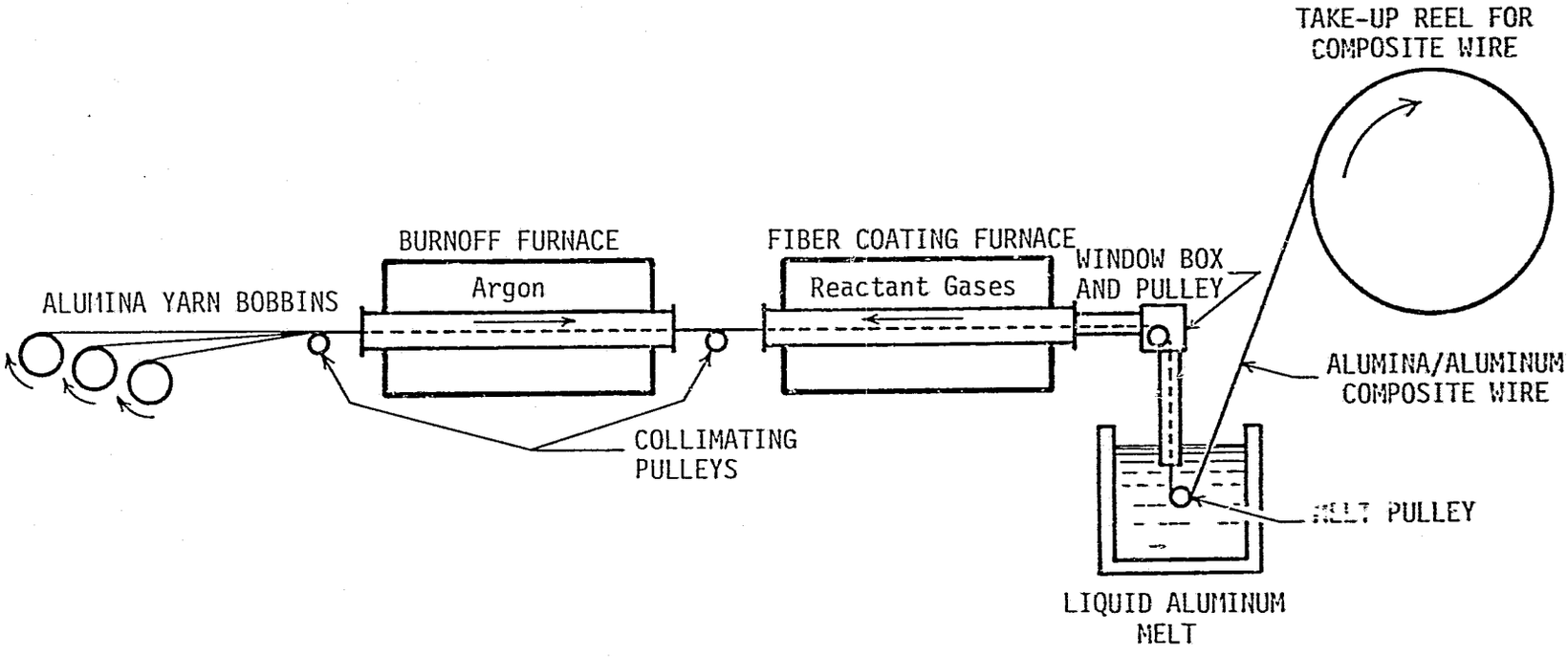



FIGURE 1

SCHEMATIC OF ALUMINUM INFILTRATION UNIT

TABLE 2

CHARACTERIZATION OF BOBBINS OF FP ALUMINA YARN

(Finish Applied)

| Bobbin | Tensile (1/4") (kpsi) | Std. Dev. | Modulus (Mpsi) | Fil. Diameter (mils) | Std. Dev. | Wt. (lbs) |
|---|---|---|---|---|---|---|
| P236-2 | 251 | 30 | 58.3 | 0.74 | 0.05 | 0.99 |
| P236-4 | 238 | 47 | 62.5 | 0.75 | 0.05 | 1.24 |
| P236-5 | 242 | 54 | 59.8 | 0.76 | 0.04 | 1.15 |
| P236-10 | 252 | 25 | 58.3 | 0.76 | 0.03 | 1.41 |
| P236-12 | 243 | 37 | 60.0 | 0.74 | 0.03 | 0.99 |
| P236-17 | 258 | 57 | 63.2 | 0.74 | 0.06 | 1.35 |
| P236-19 | 227 | 22 | 57.4 | 0.76 | 0.04 | 1.43 |
| P236-22 | 231 | 34 | 59.2 | 0.76 | 0.02 | 1.41 |
| P236-25 | 212 | 40 | 55.1 | 0.75 | 0.03 | 1.33 |
| P236-29 | 226 | 50 | 57.4 | 0.77 | 0.03 | 0.94 |
| P236-30 | 221 | 53 | 58.3 | 0.75 | 0.04 | 1.00 |
| P236-31 | 219 | 18 | 55.3 | 0.76 | 0.04 | 1.46 |
| P236-32 | 261 | 37 | 59.1 | 0.74 | 0.04 | 1.44 |
| P236-34 | 245 | 51 | 61.5 | 0.74 | 0.04 | 0.89 |
| P236-36 | 234 | 51 | 56.9 | 0.76 | 0.03 | 1.04 |
| P236-37 | 211 | 24 | 54.8 | 0.77 | 0.05 | 1.42 |
| P236-39 | 249 | 53 | 58.6 | 0.75 | 0.03 | 1.23 |
| P236-41-2 | 269 | 30 | 58.2 | 0.74 | 0.03 | 0.92 |
| P236-42 | 227 | 50 | 55.0 | 0.75 | 0.03 | 1.40 |
| P237-2 | 216 | 54 | 61.6 | 0.73 | 0.06 | 1.29 |
| P237-3 | 252 | 22 | 55.5 | 0.76 | 0.04 | 1.27 |
| P237-4 | 256 | 34 | 61.7 | 0.74 | 0.04 | 1.36 |
| P237-5 | 245 | 38 | 55.2 | 0.78 | 0.05 | 1.16 |
| P237-7 | 266 | 35 | 54.3 | 0.79 | 0.05 | 1.20 |
| P237-19-2 | 227 | 36 | 57.1 | 0.75 | 0.04 | 0.89 |
| | | | | | | 30.2 lbs |

Average Precursor Tensile (1/4") = 220 kpsi ± 23

reported by DuPont. This is probably due to interaction and crossing over of fibers during the bundle testing.

Bundles of five, seven and nine strands of $Al_2O_3$ fiber were infiltrated with a modified 1100 aluminum (0.3% Mg added) alloy. Due to the thickness of the fiber bundles, infiltration start-up times were longer and maximum operating speeds were slightly slower than those experienced with single strand $Al_2O_3$. Infiltration, however, was very consistent. Seven-strand wire was continuously produced for up to seven hours with no loss in wire integrity.

Examples of the wire produced are presented in Figures 2-5 Full infiltration was observed in all cases. Volume percent fiber for each condition was calculated based on planimeter traces across the micrographs. The results indicate that the volume percent fiber for the five-strand wire produced at 24 inches per minute was approximately 27.5 v/o, the seven-strand wire produced at 12 and 24 inches per minute was approximately 25.2 v/o, and the nine-strand wire produced at 24 inches per minute was approximately 30.1 v/o. Thus, the seven-strand wire yields the lowest volume percent values.

In order to achieve lower volume percents in the required range, precoating the fiber with borocarbon coating prior to CVD treatment and infiltration was explored. This method has been utilized at FMI to improve the wetting of graphite fibers. It was believed that the treatment would cause the $Al_2O_3$ fiber to pick up more aluminum as it is drawn through the melt.

The coating was applied in a CVD unit by the chemical decomposition of triethylborane, $(C_2H_5)_3B$, at 1100°C. Figures 6-8 represent seven-strand $Al_2O_3$ wire produced using the above precoating technique at 1, 2 and 3 feet/minute, respectively. It was found that as the wire speed was increased the volume percent also increased. The volume percents obtained were: 25.5 v/o fiber at 1 foot/minute; 26.5 v/o fiber at 2 feet/minute; and 29.5

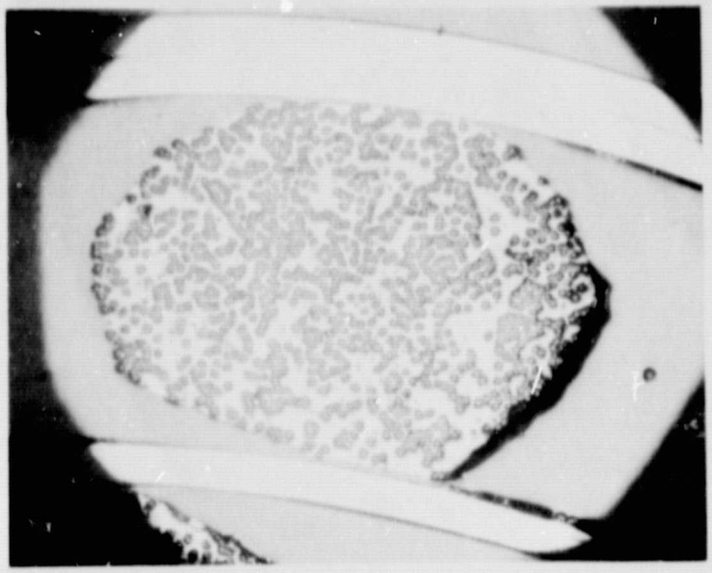


FIGURE 2. 5-STRAND $Al_2O_3$/Al COMPOSITE WIRE PRODUCED AT 24"/min. 50x.



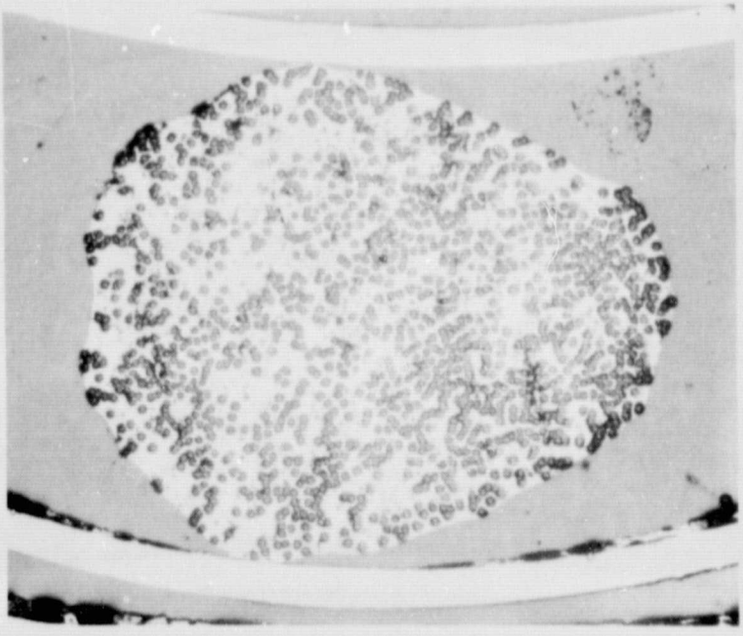


FIGURE 3. 7-STRAND $Al_2O_3$/Al COMPOSITE WIRE PRODUCED AT 12"/min. 50x.

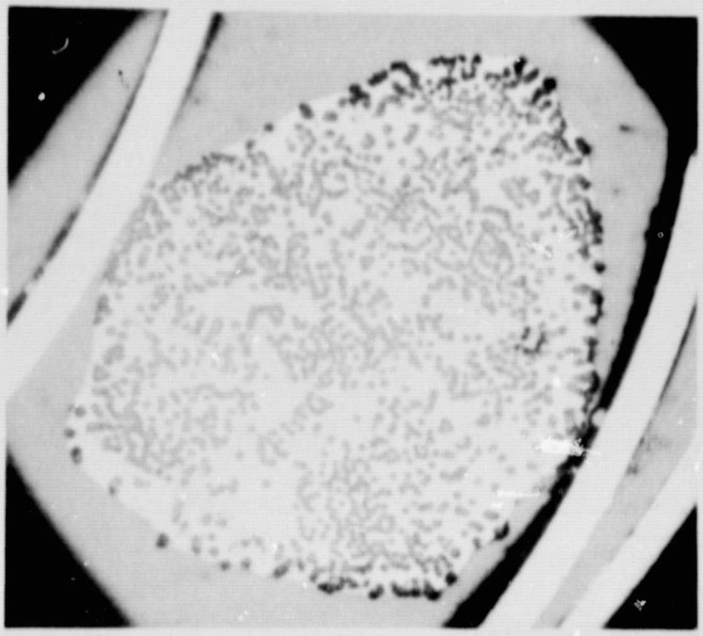


FIGURE 4. 7-STRAND $Al_2O_3$/Al COMPOSITE WIRE PRODUCED AT 24"/min. 50x.

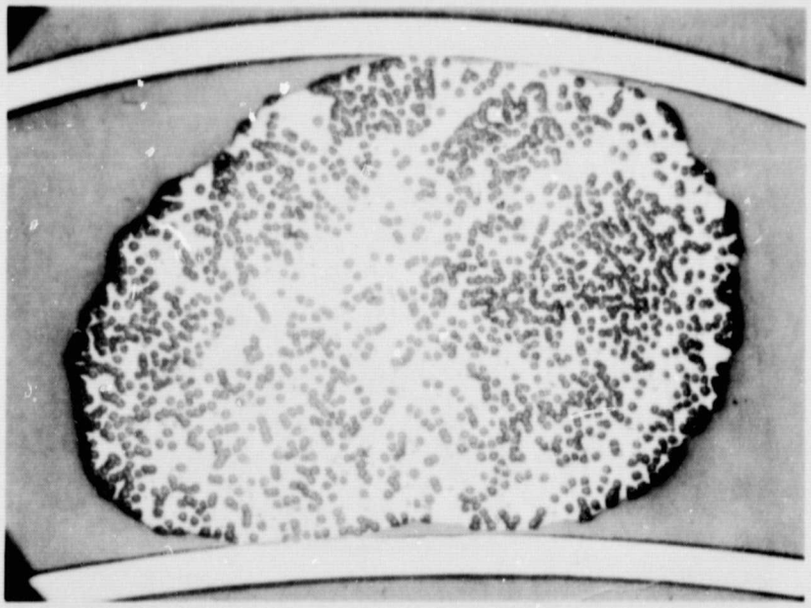


FIGURE 5. 9-STRAND $Al_2O_3$/Al COMPOSITE WIRE PRODUCED AT 24"/min. 50x.

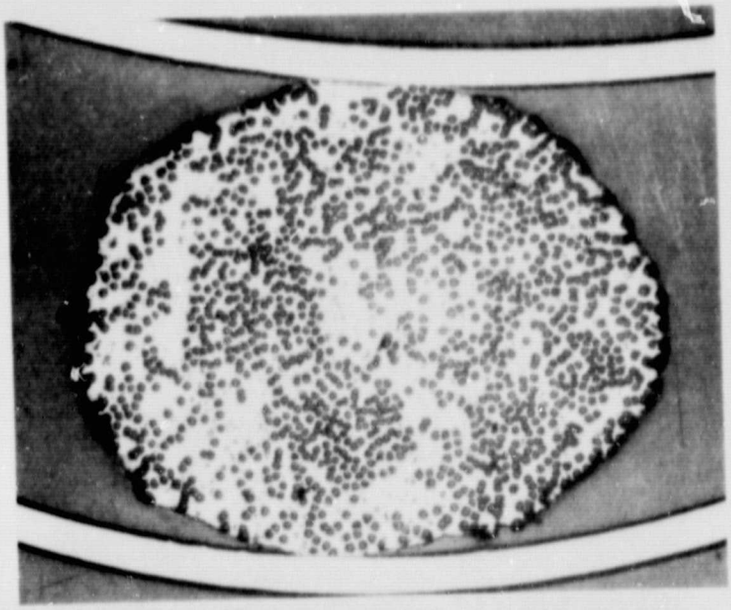


FIGURE 6. 7-STRAND $Al_2O_3$/1100 Al COMPOSITE WIRE PRODUCED WITH PRECOATING TREATMENT AT 1 FOOT/MINUTE. (50X)



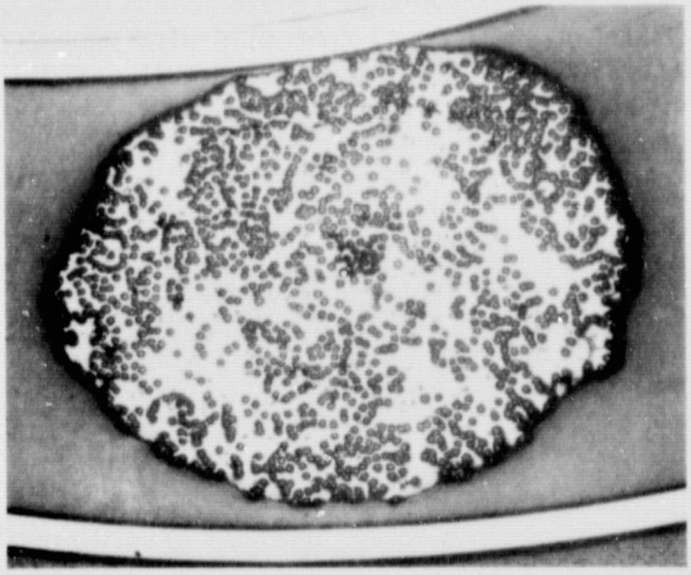


FIGURE 7. 7-STRAND $Al_2O_3$/1100 Al COMPOSITE WIRE PRODUCED WITH PRECOATING TREATMENT AT 2 FEET/MINUTE. (50X)

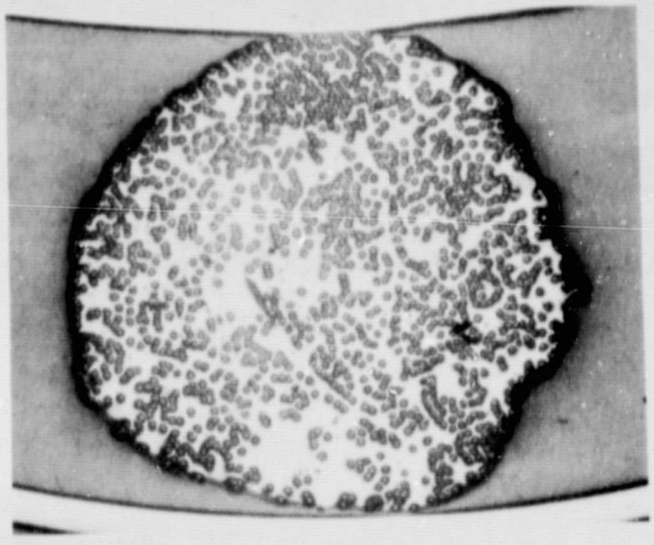


FIGURE 8. 7-STRAND $Al_2O_3$/1100 Al COMPOSITE WIRE PRODUCED WITH PRECOATING TREATMENT AT 3 FEET/MINUTE. (50X)

v/o fiber at 3 feet/minute. Thus, even at the lowest speed the goal of 15-20 v/o fiber was not achieved using the pre-coat process. However, infiltration start-up times were greatly decreased by the use of the pre-coat.

Tensile tests on the seven-strand wire produced at 2 feet/minute were performed and the data was compared to data for similar wire processed without the triethylborane. As can be seen from the results in Table 3, the U.T.S. of the wire processed with the pre-coating was significantly higher than that for the non pre-coated wire. The modulus and strain-to-failure values were not significantly affected. Also, the data for the pre-coated wire was very consistent, which is in sharp contrast to the wire made without the pre-coat. Due to the improved start-up times and mechanical properties, it was decided to use the pre-coating treatment and process wire at 2 feet/minute for the material to be used on this program. Enough wire for several trials and the majority of the bars required on the program were processed using these conditions.

Samples of the preform wire were submitted for quantitative chemical analysis of the major matrix alloy constituents and impurities. The results are presented in Table 4 along with the allowable levels of each constituent as specified in Volume 1 of ASM Metals Handbook. The data shows that the percentages of Fe, Cu and Si present are within specifications. The high Ti and B are most likely due to the Ti/B coating applied at the fiber/matrix interface to induce wetting.

Although wetting was improved by the triethylborane pre-coating, the volume percent fiber in the wire was still higher than required in the objectives of the program. It was decided to employ encapsulation techniques similar to those used in the preparation of Gr/Al panels[4]. This process of placing a layer of additional aluminum around each composite wire allows for control of volume percent fiber in bulk composite material

TABLE 3

COMPARISON OF $Al_2O_3$/1100 COMPOSITE WIRE PROPERTIES

PROCESSED WITH AND WITHOUT $(C_2H_5)_3B$ PRE-COATING

| Material | Sample# | U.T.S. (Ksi) | E (msi) | $\varepsilon_f$ (%) |
|---|---|---|---|---|
| with pre-coat | 1 | 49.6 | 14.9 | 0.55 |
| with pre-coat | 2 | 50.4 | 15.8 | 0.52 |
| with pre-coat | 3 | 41.4 | 15.2 | 0.54 |
| with pre-coat | 4 | 46.7 | 16.4 | 0.50 |
| with pre-coat | $\bar{X}$ | 47.0 | 15.6 | 0.53 |
| without pre-coat | 1 | 44.3 | 17.5 | 1.17 |
| without pre-coat | 2 | 19.3 | 9.8 | 0.84 |
| without pre-coat | 3 | 32.0 | 11.9 | 0.69 |
| without pre-coat | 4 | 33.6 | 24.1 | 0.34 |
| without pre-coat | $\bar{X}$ | 32.3 | 15.8 | 0.76 |

TABLE 4

CHEMICAL COMPOSITION OF 1100 Al MATRIX (WIRE)

| Material | Composition (w/o) | | | | |
|---|---|---|---|---|---|
| | Fe | Si | Cu | Ti | B |
| Preform Wire Matrix | 0.43 | 0.10 | 0.14 | 0.29 | 0.07 |
| 1100 Al Melt Material(1) | 1.0 Max. (with Si) | 1.0 Max. (with Fe) | 0.20 | -- | -- |

Note:
(1) Composition from ASM Metals Handbook, Volume 1.

by adjusting the amount of aluminum foil added, and additionally offers the potential advantages of less damage to fiber during mechanical fabrication and improved shear and transverse properties in the bulk composite.

1 mil thick 1100 Al foil was obtained and encapsulation studies were performed on wire preforms. The foil was cut into 1 inch by 13 inch strips and wrapped lengthwise around 13 inch wires. It was found that simple hand wrapping was an effective way of encapsulation due to the relatively large diameter of the wire. Although the method is slow and tedious when done by hand, it can be automated if large quantities of material are needed.

2.2 Composite Fabrication

The consolidation of wire preforms into bulk composite material was accomplished by a hot drawing or pultrusion process. A schematic of the unit is shown in Figure 9. In this process, preform wires are bundled uniaxially in the inconel containment and the entire assembly is heated to temperature and drawn through a die.

Hot drawing conditions established under NASA contract NAS3-21013 were used in fabricating low volume percent bar stock. Material was processed at a drawing temperature of 1125°F and 0% reduction.

Initial fabrication trials were performed to determine the effects of the encapsulant foil and drawing speed on the pultrusion process. The normal drawing speed is about 15 feet/minute. One bar with foils was drawn at that speed and was compared to a bar drawn at a similar speed in an earlier program (contract # NAS3-21013). One bar each with and without foils was drawn at a speed of 6.5 feet/minute. All three bars were submitted for radiography prior to mechanical testing. Radiography of these three bars revealed various degrees of cracking. The bar drawn at a speed of 15 ft./min. (18 v/o) possessed a significantly larger amount of cracking than the other bars drawn at 6.5 ft./min. The radiography was not precise enough to show a significant difference between the 18 v/o and 26 v/o bars drawn at the slower speed.

SECTIONAL VIEW OF PULTRUSION PROCESS SET-UP

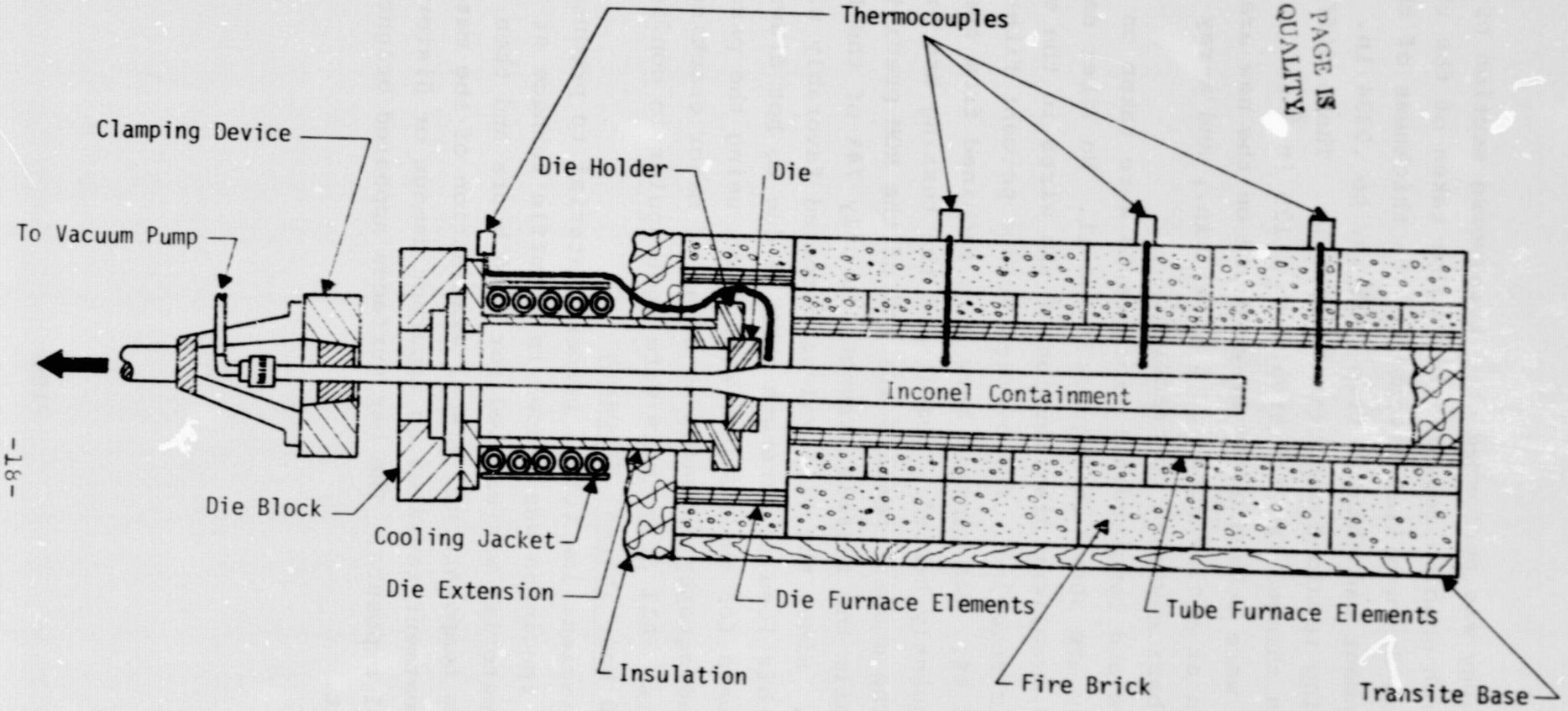



FIGURE 9

Metallography was performed on a transverse section of one of the bars which enabled measurements to be taken of the thicknesses of the as-drawn billet assembly. The thickness of the inconel containment had changed from .035 in. to .0334 in. The aluminum cladding remained constant at .028 in. These differences in measurements changed the bar area from .1125 $in.^2$ to .10857 $in.^2$ Six bars were subsequently drawn based on the new area. The bars were drawn at a slow speed of 5 ½ ft./min., and x-ray radiography of the bars revealed no cracking.

Transverse and longitudinal micrographs were taken on one of the bars. They are shown in Figures 10 and 11. No fiber damage is evident and excellent distribution of the wires in the encapsulated form is revealed. The average volume percent fiber was found to be 16.8%. Two tensile bars were machined from the same bar and subsequently tested. Results of the testing are presented in Table 5. The modulus results are 94% of the ROM prediction of 16.7 Msi, while the tensile strength is only 74% of the ROM value of 31,960 Ksi. Since the wire values compared favorably with ROM predictions, this loss in strength must be due to hot drawing.

All the bars for the program were drawn using the parameters set above. Radiography revealed no areas of major cracking in any of the bars. All drawn bars were rectangular in configuration.

2.3 1000 Hour Thermal Exposure

Sufficient low volume percent material to produce all required test specimens was placed in a muffle furnace at 300°C in air. The material was exposed for 1000 hours and then slowly cooled to room temperature. Visual examination of the material after heat treatment revealed no apparent damage or distortion due to thermal exposure. The bar surfaces appeared brighter after heat treatment.

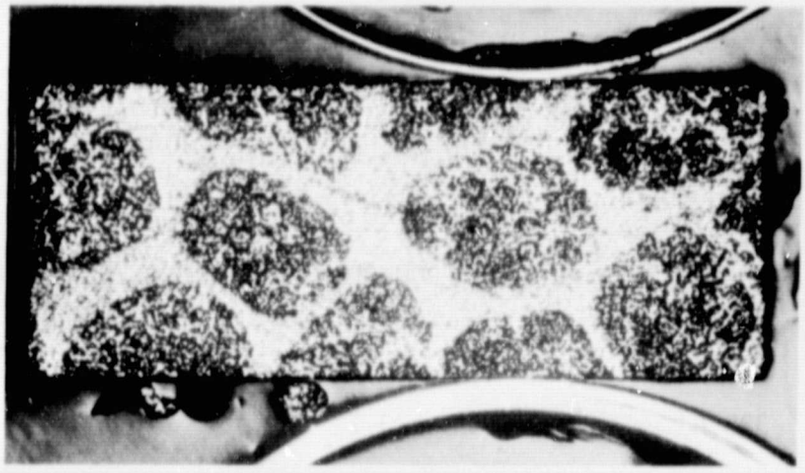


FIGURE 10 - TRANSVERSE MICROGRAPH OF 16.8% FIBER $Al_2O_3$/1100 Al COMPOSITE BAR (15X)



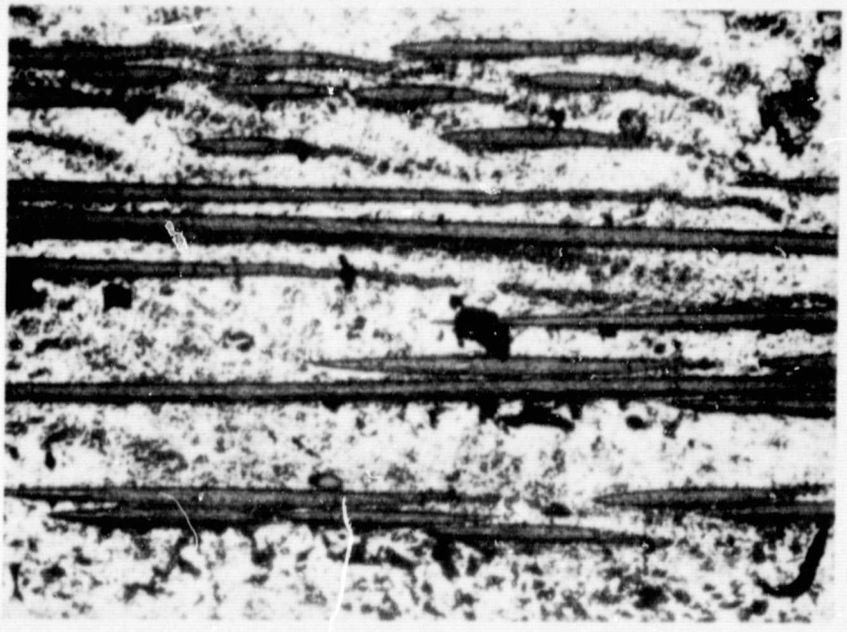


FIGURE 11 - LONGITUDINAL MICROGRAPH OF 16.8% FIBER $Al_2O_3$/1100 Al COMPOSITE BAR (125X)

TABLE 5

TENSILE TEST RESULTS FROM PRELIMINARY DRAWING TRIALS

| Sample | U.T.S.(ksi) | E (Msi) | $\varepsilon_f$ (%) |
|---|---|---|---|
| 328 A | 23.7 | 15.8 | 0.30 |
| 328 B | 23.7 | 15.6 | 0.26 |
| Average | 23.7 | 15.7 | 0.28 |

## 3.0 TASK II - TESTING AND DATA ANALYSIS OF $Al_2O_3$/1100 Al MATERIAL

Task II was concerned with the establishment of a data base for the low volume percent $Al_2O_3$/1100 Al composite material developed in Task I. Extensive mechanical, thermal and chemical testing was performed. Where appropriate, test data generated under NASA contract NAS3-21013 for nominal 30 and 40 v/o $Al_2O_3$/1100 Al material has been included in figures, tables and discussion. This gives an overall picture of the mechanical response of $Al_2O_3$/1100 Al composite material developed for NASA under the two programs. Tables 6-10 show cutting plans and test outlines for both as-drawn and heat-treated material.

### 3.1 Mechanical Testing Results

#### 3.1.1 Longitudinal Tensile Testing

Longitudinal tensile testing was conducted on the 20 v/o fiber $Al_2O_3$/Al material in both the as-drawn and heat-treated condition. Samples were pulled at room temperature and at 300°C in air. The sample size and configuration are shown in Figure 12.

Results of the tensile testing are given in Table 11 and Figure 13. COnsideration of the U.T.S. results indicates that values are lower than expected. At room temperature, values are 65% of ROM for the as-drawn, and 73% ROM for heat treated 20 v/o fiber material. These low values are probably due to fiber breakage during the drawing process.

Figure 14 is a graph of U.T.S. vs % fiber for as-drawn and heat-treated material. 30 and 40 v/o fiber values were taken from NASA contract NAS3-21013. As can be seen from the curves, there is a peak in the strength for the as-drawn material at around 30% fiber. The modulus of the as-drawn, 20 v/o fiber material tested at room temperature is approximately that of rule-of-mixtures.

Residual stresses occurring on cool down of the composites from the processing temperatures may also affect mechanical properties. Such stresses occur due to thermal expansion mismatch between the $Al_2O_3$ fiber and the aluminum matrix. The thermal expansion/contraction coefficient of $Al_2O_3$ is approximately $8 \times 10^{-6}$ in/in/°C and that of the aluminum approximately $25 \times 10^{-6}$ in/in/°C. On

TABLE 6

SPECIMEN LAYOUT, NASA LEWIS TEST PLAN

As-drawn Material Condition

| Bar # | Sample # | Description | Orientation | Dwg. # | Test Temp. |
|---|---|---|---|---|---|
| 341 | A.01 | Tensile | Long. | AMDL-105 | RT |
| | A.02 | Tensile | Long. | AMDL-105 | 300 |
| | A.03 | Flexure | Long. | Sketch 1-a | RT |
| | A.04 | Stress Rupture | Long. | AMDL-105 | 300 |
| | A.05 | Compression | Long. | Sketch 4-a | RT |
| | A.06 | Compression | Trans. | Sketch 4-b | RT |
| | A.07 | Tensile | Trans. | Sketch 2-a | RT |
| | A.08 | Tensile | Trans. | Sketch 2-a | 300 |
| | A.09 | Flexure | Trans. | Sketch 1-b | RT |
| | A.10 | Mechanical Fatigue | Trans. | Sketch 2-a | RT |
| 342 | A.11 | Tensile | Long. | AMDL-105 | RT |
| | A.12 | Tensile | Long. | AMDL-105 | 300 |
| | A.13 | Flexure | Long. | Sketch 1-a | RT |
| | A.14 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | A.15 | Compression | Long. | Sketch 4-a | RT |
| | A.16 | Compression | Trans. | Sketch 4-b | RT |
| | A.17 | Tensile | Trans. | Sketch 2-a | RT |
| | A.18 | Tensile | Trans. | Sketch 2-a | 300 |
| | A.19 | Flexure | Trans. | Sketch 1-b | RT |
| 343 | A.20 | Tensile | Long. | AMDL-105 | RT |
| | A.21 | Tensile | Long. | AMDL-105 | 300 |
| | A.22 | Stress Rupture | Long. | AMDL-105 | 300 |
| | A.23 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | A.24 | Compression | Long. | Sketch 4-a | RT |
| | A.25 | Compression | Trans. | Sketch 4-b | RT |
| | A.26 | Tensile | Trans. | Sketch 2-a | RT |
| | A.27 | Tensile | Trans. | Sketch 2-a | 300 |
| | A.28 | Flexure | Trans. | Sketch 1-b | RT |
| | A.29 | Mechanical Fatigue | Trans. | Sketch 2-a | RT |
| 344 | A.30 | Flexure | Long. | Sketch 1-a | RT |
| | A.31 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | A.32 | Stress Rupture | Long. | AMDL-105 | 300 |
| | A.33 | Stress Rupture | Long. | AMDL-105 | 300 |
| | A.34 | Thermal Expansion | Long. | Sketch 3-a | - |
| | A.35 | Thermal Expansion | Trans. | Sketch 3-b | - |
| | A.36 | Mechanical Fatigue | Trans. | Sketch 2-a | RT |

TABLE 7

SPECIMEN CUTTING PLAN

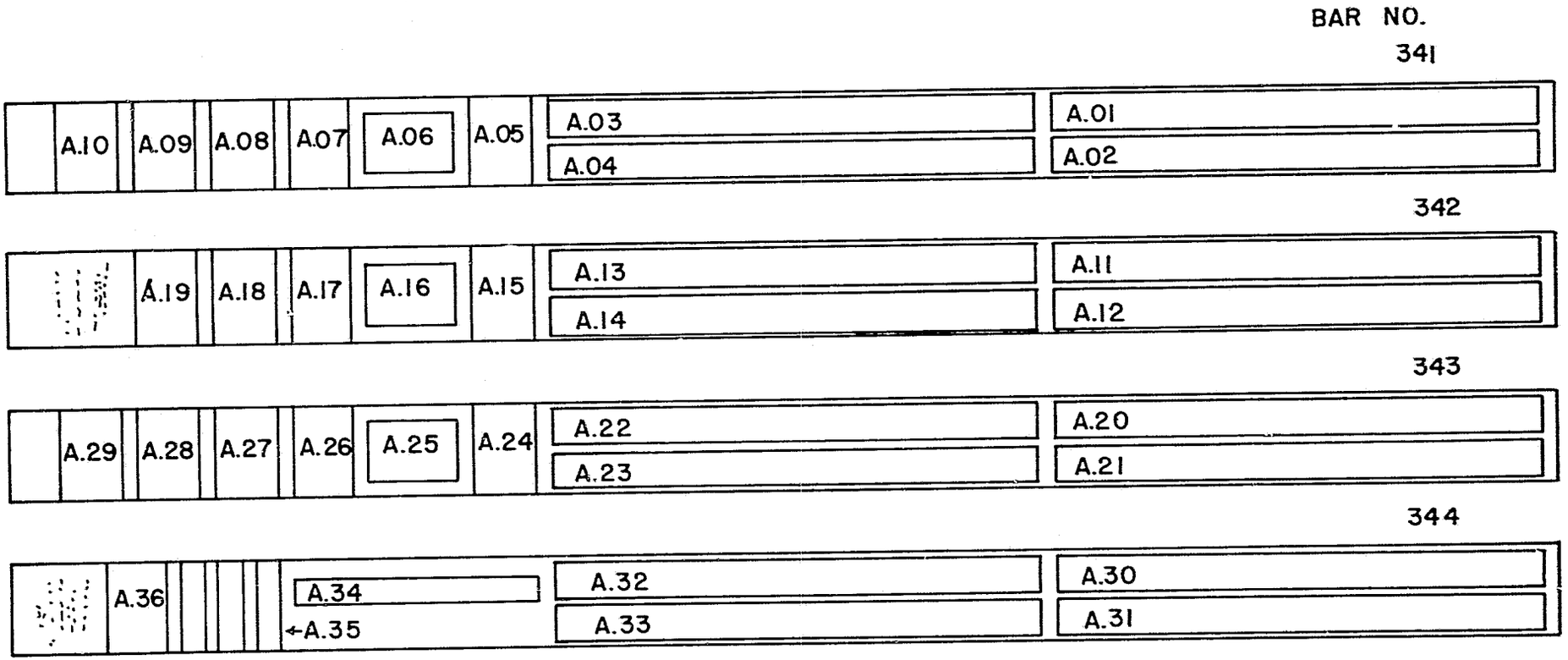


TABLE 8

SPECIMEN LAYOUT, NASA LEWIS TEST PLAN

Heatreated Material Condition

| Bar # | Sample# | Description | Orientation | Dwg.# | Test Temp. |
|---|---|---|---|---|---|
| 336 | H.01 | Tensile | Long. | AMDL-105 | RT |
| | H.02 | Tensile | Long. | AMDL-105 | 300 |
| | H.03 | Flexure | Long. | Sketch 1-a | RT |
| | H.04 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | H.05 | Compression | Long. | Sketch 4-a | RT |
| | H.06 | Compression | Trans. | Sketch 4-b | RT |
| | H.07 | Tensile | Trans. | Sketch 2-a | RT |
| | H.08 | Tensile | Trans. | Sketch 2-a | 300 |
| | H.09 | Flexure | Trans. | Sketch 1-b | RT |
| | H.10 | Mechanical Fatigue | Trans. | Sketch 2-a | RT |
| 337 | H.11 | Tensile | Long. | AMDL-105 | RT |
| | H.12 | Tensile | Long. | AMDL-105 | 300 |
| | H.13 | Flexure | Long. | Sketch 1-a | RT |
| | H.14 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | H.15 | Compression | Long. | Sketch 4-a | RT |
| | H.16 | Compression | Trans. | Sketch 4-b | RT |
| | H.17 | Tensile | Trans. | Sketch 2-a | RT |
| | H.18 | Tensile | Trans. | Sketch 2-a | 300 |
| | H.19 | Flexure | Trans. | Sketch 1-b | RT |
| | H.20 | Mech. Fatigue | Trans. | Sketch 2-a | RT |
| 339 | H.21 | Tensile | Long. | AMDL-105 | RT |
| | H.22 | Tensile | Long. | AMDL-105 | 300 |
| | H.23 | Flexure | Long. | Sketch 1-a | RT |
| | H.24 | Mechanical Fatigue | Long. | AMDL-105 | RT |
| | H.25 | Compression | Long. | Sketch 4-a | RT |
| | H.26 | Compression | Trans. | Sketch 4-b | RT |
| | H.27 | Tensile | Trans. | Sketch 2-a | RT |
| | H.28 | Tensile | Trans. | Sketch 2-a | 300 |
| | H.29 | Flexure | Trans. | Sketch 1-b | RT |
| | H.30 | Mech. Fatigue | Trans. | Sketch 2-a | RT |
| 340 | H.31 | Thermal Expansion | Trans. | Sketch 3-b | - |
| | H.32 | Thermal Expansion | Long. | Sketch 3-a | - |

TABLE 9

SPECIMEN CUTTING PLAN

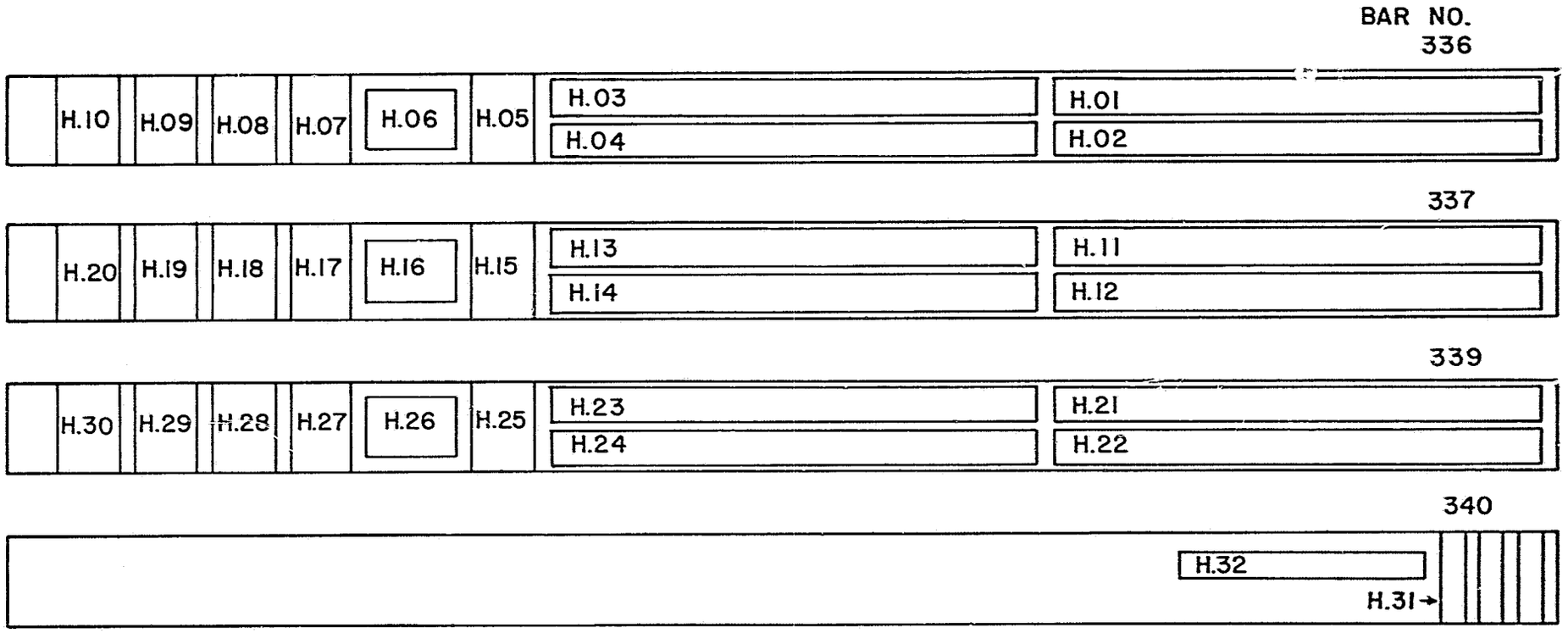
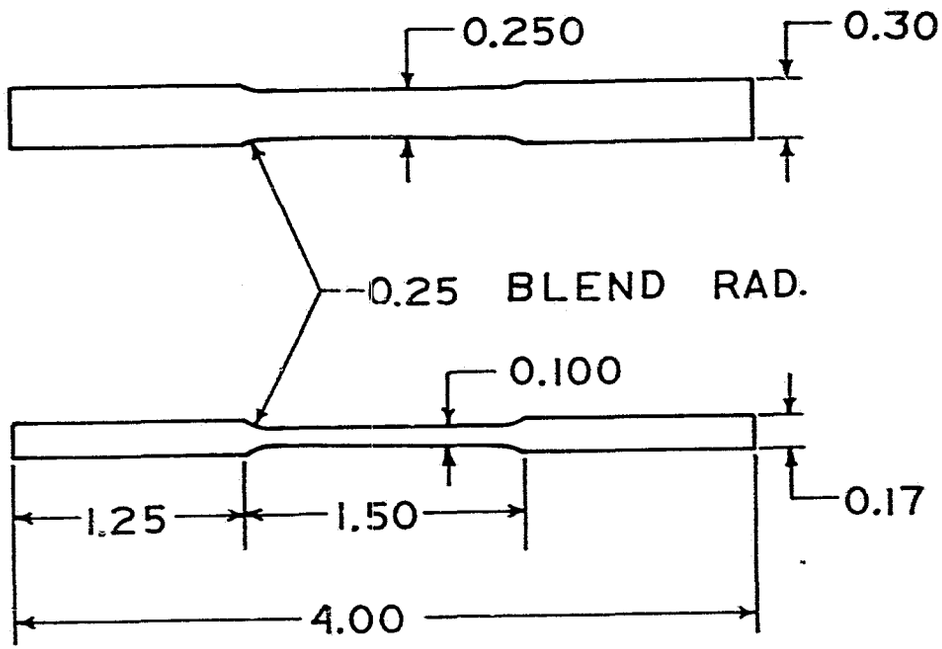


TABLE 10

TEST OUTLINE FOR $Al_2O_3$/Al MATERIAL

| Test Type | 20 V/O FIBER | |
|---|---|---|
| | As-Drawn | Heat-Treated |
| Longitudinal Tensile | 3 @ R.T.<br>3 @ 300°C | 3 @ R.T.<br>3 @ 300°C |
| Transverse Tensile | 3 @ R.T.<br>3 @ 300°C | 3 @ R.T.<br>3 @ 300°C |
| Longitudinal Compression | 3 @ R.T. | 3 @ R.T. |
| Transverse Compression | 3 @ R.T. | 3 @ R.T. |
| Longitudinal Flexure | 3 @ R.T. | 3 @ R.T. |
| Transverse Flexure | 3 @ R.T. | 3 @ R.T. |
| Longitudinal Stress-Rupture | 4 @ 300°C | |
| Longitudinal Mechanical Fatigue | 3 @ R.T. | 3 @ R.T. |
| Transverse Mechanical Fatigue | 3 @ R.T. | 3 @ R.T. |

FIGURE 12 - LONGITUDINAL TENSILE SPECIMEN CONFIGURATION




ALL DIMENSIONS
IN INCHES
TOLERANCES:
.XX = ±.05 .XXX = ±.005

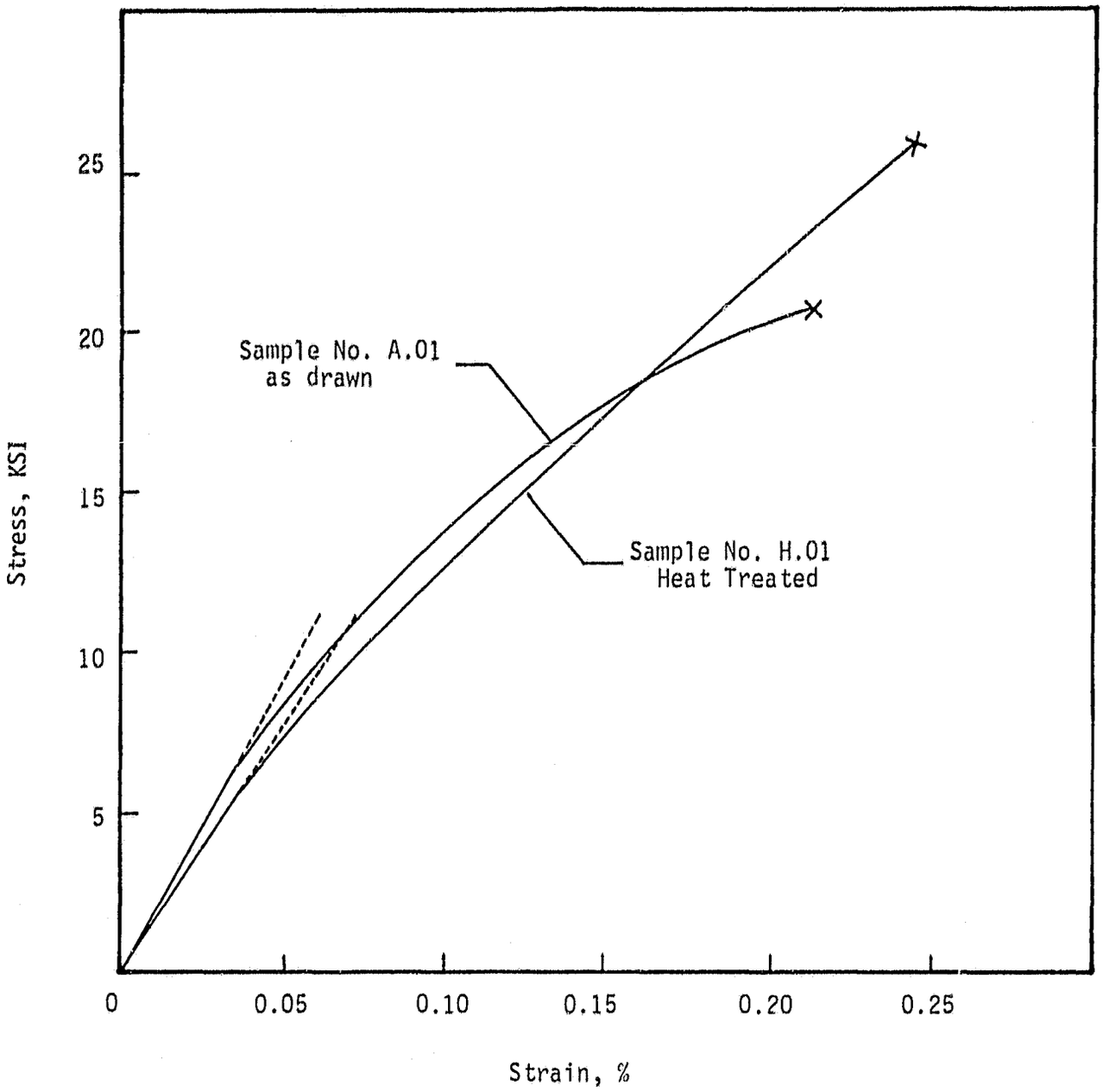
TABLE 11

LONGITUDINAL TENSILE TEST RESULTS

| Sample# | Condition | Test Temperature | U.T.S. (Ksi) | $E_1$ (Msi) | $\varepsilon$ (%) |
|---|---|---|---|---|---|
| A.01 | As-Drawn | R.T. | 20.8 | 18.9 | 0.21 |
| A.11 | As-Drawn | R.T. | 19.2 | 17.1 | 0.21 |
| A.20 | As-Drawn | R.T. | 24.0 | 15.4 | 0.33 |
| Average | | | 21.3 | 17.1 | 0.25 |
| H.01 | Heat-Treated | R.T. | 25.9 | 16.5 | 0.25 |
| H.11 | Heat-Treated | R.T. | 27.2 | 17.2 | 0.26 |
| H.21 | Heat-Treated | R.T. | 25.6 | 16.5 | 0.27 |
| Average | | | 26.2 | 16.7 | 0.26 |
| A.02 | As-Drawn | 300°C | 15.2 | 16.8 | 0.28 |
| A.12 | As-Drawn | 300°C | 11.8 | 15.4 | 0.21 |
| A.21 | As-Drawn | 300°C | 13.8 | 17.5 | 0.15 |
| Average | | | 13.6 | 16.6 | 0.21 |
| H.02 | Heat-Treated | 300°C | 10.8 | 16.8 | 0.32 |
| H.12 | Heat-Treated | 300°C | 11.4 | 13.2 | 0.22 |
| H.22 | Heat-Treated | 300°C | 15.2 | 14.4 | 0.23 |
| Average | | | 12.5 | 14.8 | 0.26 |

Figure 13

Typical Stress-Strain Curves for Pultruded $Al_2O_3$/Al Bar Stock
(20 volume percent $Al_2O_3$ fiber)



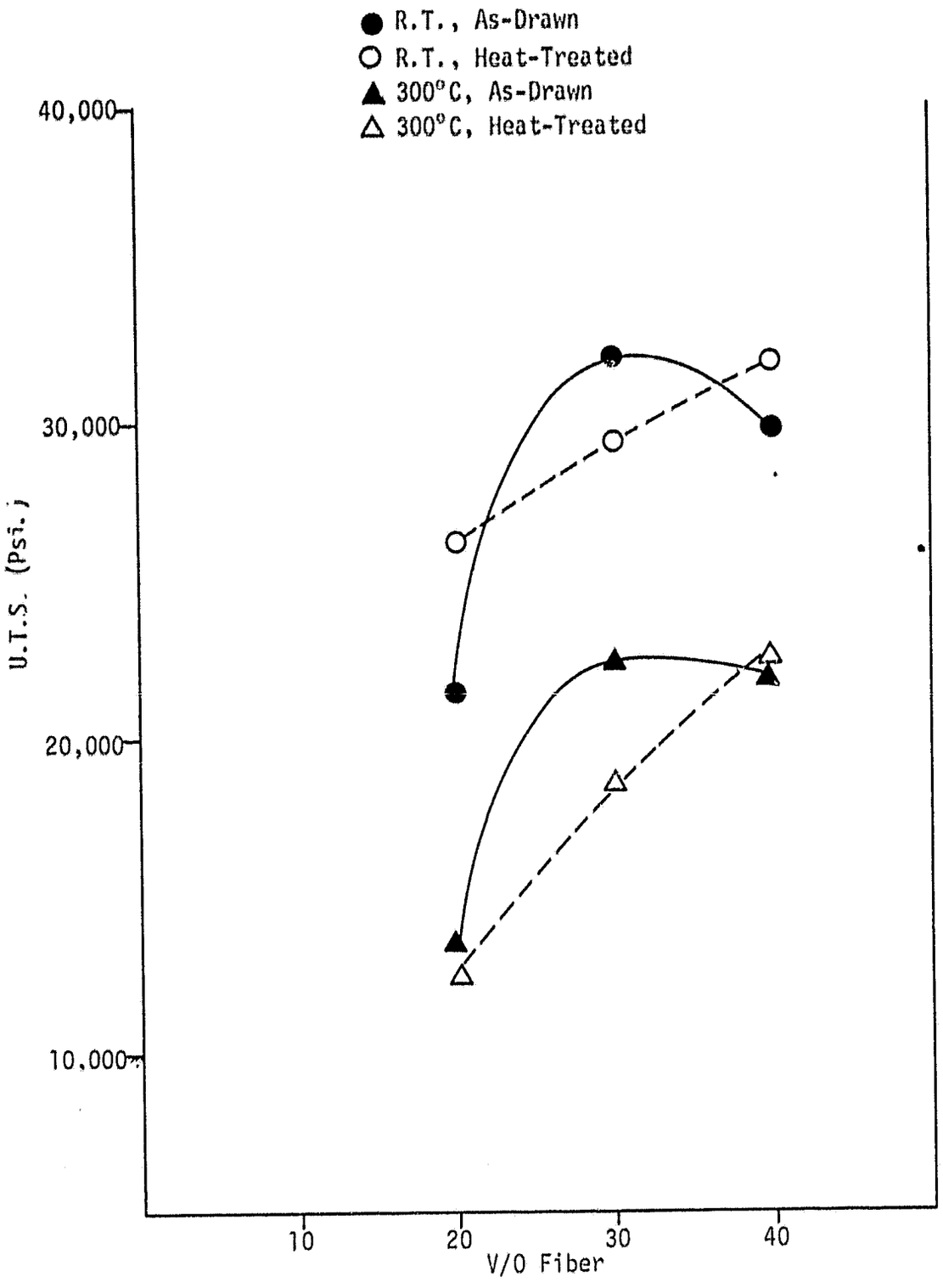



FIGURE 14 - LONGITUDINAL U.T.S. Vs. % FIBER FOR $Al_2O_3$/1100 Al BAR STOCK

cooling from the processing temperature, the greater longitudinal contraction of the matrix as compared to the fiber places the matrix in tension and, providing sufficient force is generated, places the fiber in compression. The room temperature strength data, Figures 13 and 14, show lower strength values on the as-drawn composites as compared to drawn and heat-treated composites. Premature failure of the matrix may occur during testing as-drawn composites due to the additive effect of the applied and residual tensile stresses present on the matrix. Heat treating of the composites after drawing may relieve this residual stress and higher strength values may be observed. The modulus of elasticity of the as-drawn composite samples at room temperature is higher than that of the heat-treated samples, and the effect becomes more pronounced as the fiber content of the composite increases, Figure 15. The presence of a residual compressive stress on the fibers in the as-drawn composites may affect the observed $E_1$ modulus values. For example, during the initial part of a tensile test unloading of the residual compressive stress may occur and a higher stress (the sum of the applied and residual stresses) may be recorded for a given strain, thus giving a high $E_1$ modulus value for the composite. Heat treating of the composite may relieve this residual compressive stress on the fibers through deformation of the aluminum matrix and a lower $E_1$ modulus value may subsequently be observed on testing.

3.1.2 Transverse Tensile Testing

Transverse tensile testing was performed on the 20 v/o fiber $Al_2O_3$/1100 Al material in the as-drawn and heat-treated condition. Samples were pulled at room temperature and at 300°C in air. The sample size and configuration are shown in Figure 16.

Results of the tensile testing are given in Table 12. The average ultimate tensile strength for the 20 v/o fiber material, along with 30 and 40 v/o fiber material values generated under contract NAS3-21013, are presented in curve form in Figure 17. As can be seen from the curve for the as-drawn material, the transverse strength drops off as the v/o fiber increases, as would be expected. In addition, the higher the v/o fiber the less effect elevated temperature has, as is evidenced by the 300°C test results, due to the larger percentage of reinforcement phase. Heat-treated

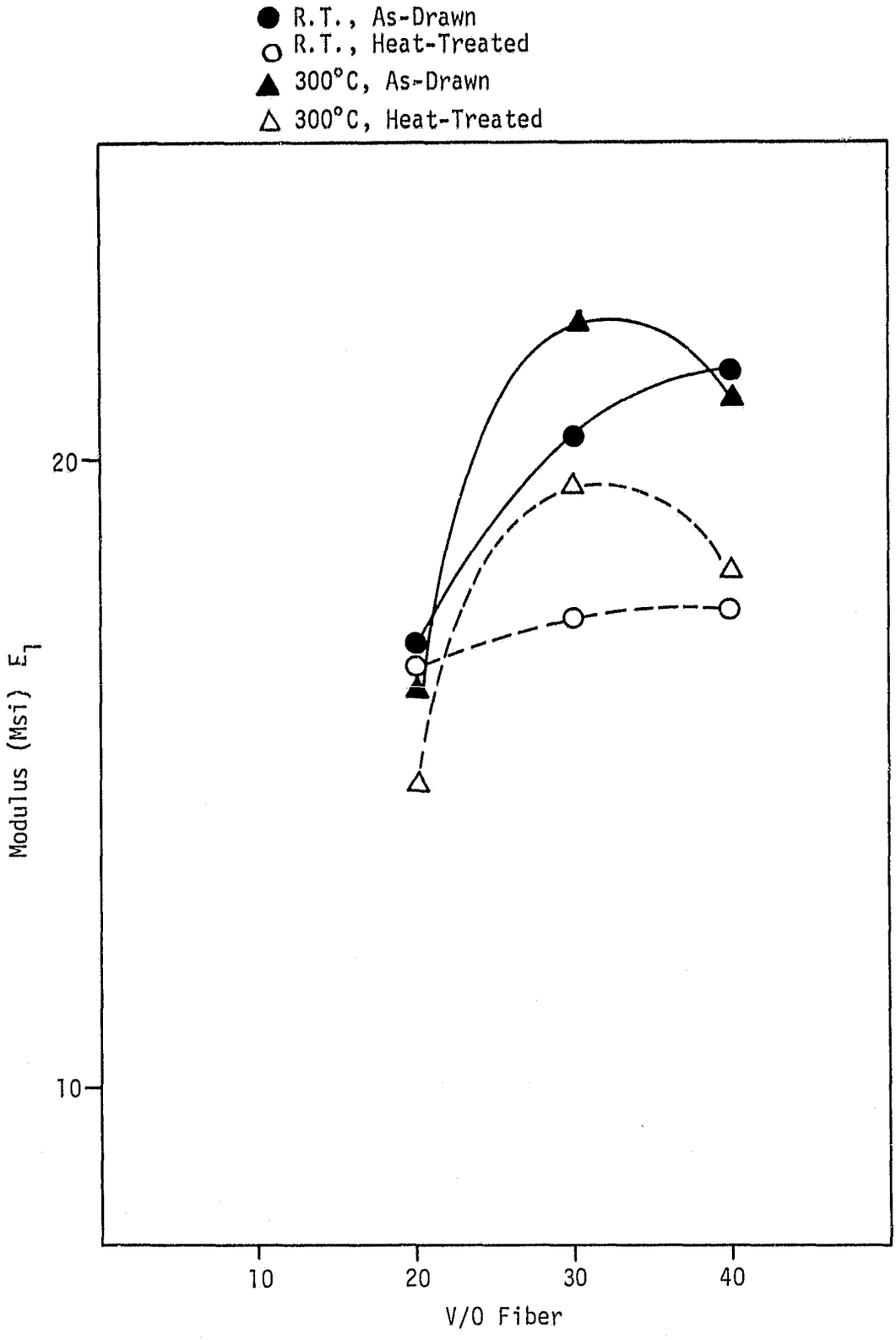



FIGURE 15 - LONGITUDINAL MODULUS Vs. % FIBER FOR $Al_2O_3$/1100 Al BAR STOCK

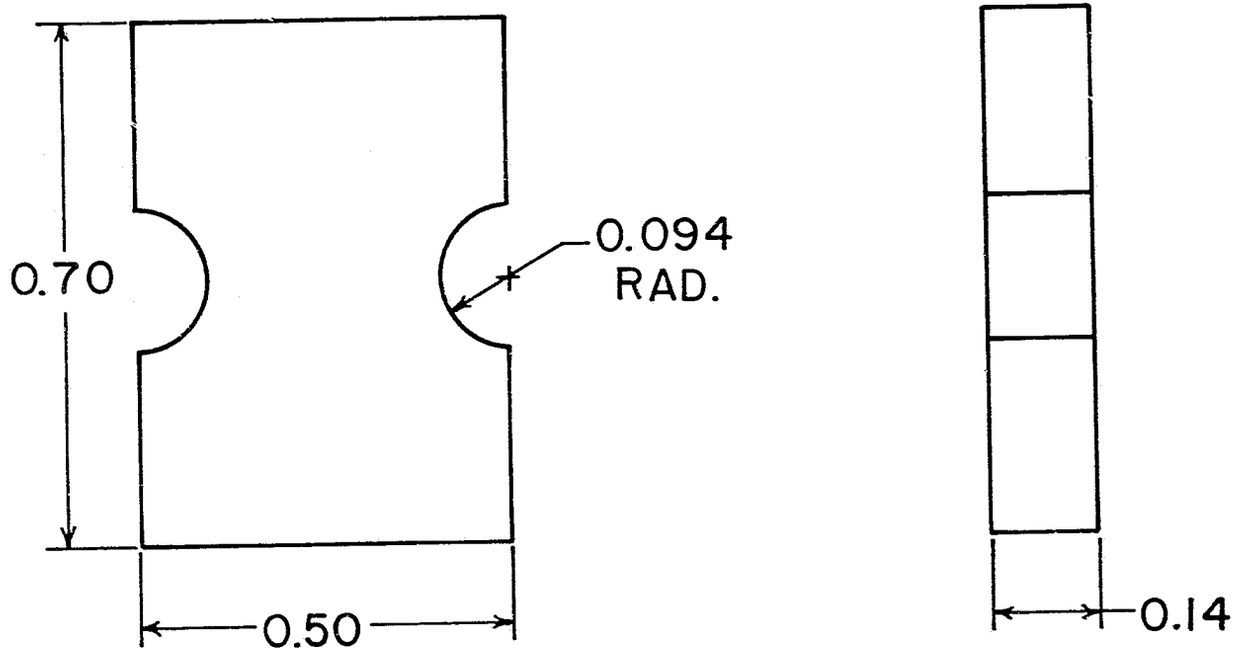



FIGURE 16 - TRANSVERSE TENSILE SPECIMEN CONFIGURATION

TABLE 12

TRANSVERSE TENSILE TEST RESULTS

| Sample# | Condition | Test Temperature | U.T.S. (Ksi) |
|---|---|---|---|
| A.07 | As-Drawn | R.T. | 8.2 |
| A.17 | As-Drawn | R.T. | 8.7 |
| A.26 | As-Drawn | R.T. | 7.5 |
| Average | | | 8.1 |
| H.07 | Heat-Treated | R.T. | 7.6 |
| H.17 | Heat-Treated | R.T. | 8.3 |
| H.27 | Heat-Treated | R.T. | 6.8 |
| Average | | | 7.6 |
| A.08 | As-Drawn | 300°C | 5.3 |
| A.18 | As-Drawn | 300°C | 5.1 |
| A.27 | As-Drawn | 300°C | 3.8 |
| Average | | | 4.7 |
| H.08 | Heat-Treated | 300°C | 4.4 |
| H.18 | Heat-Treated | 300°C | 4.6 |
| H.27 | Heat-Treated | 300°C | 4.2 |
| Average | | | 4.4 |

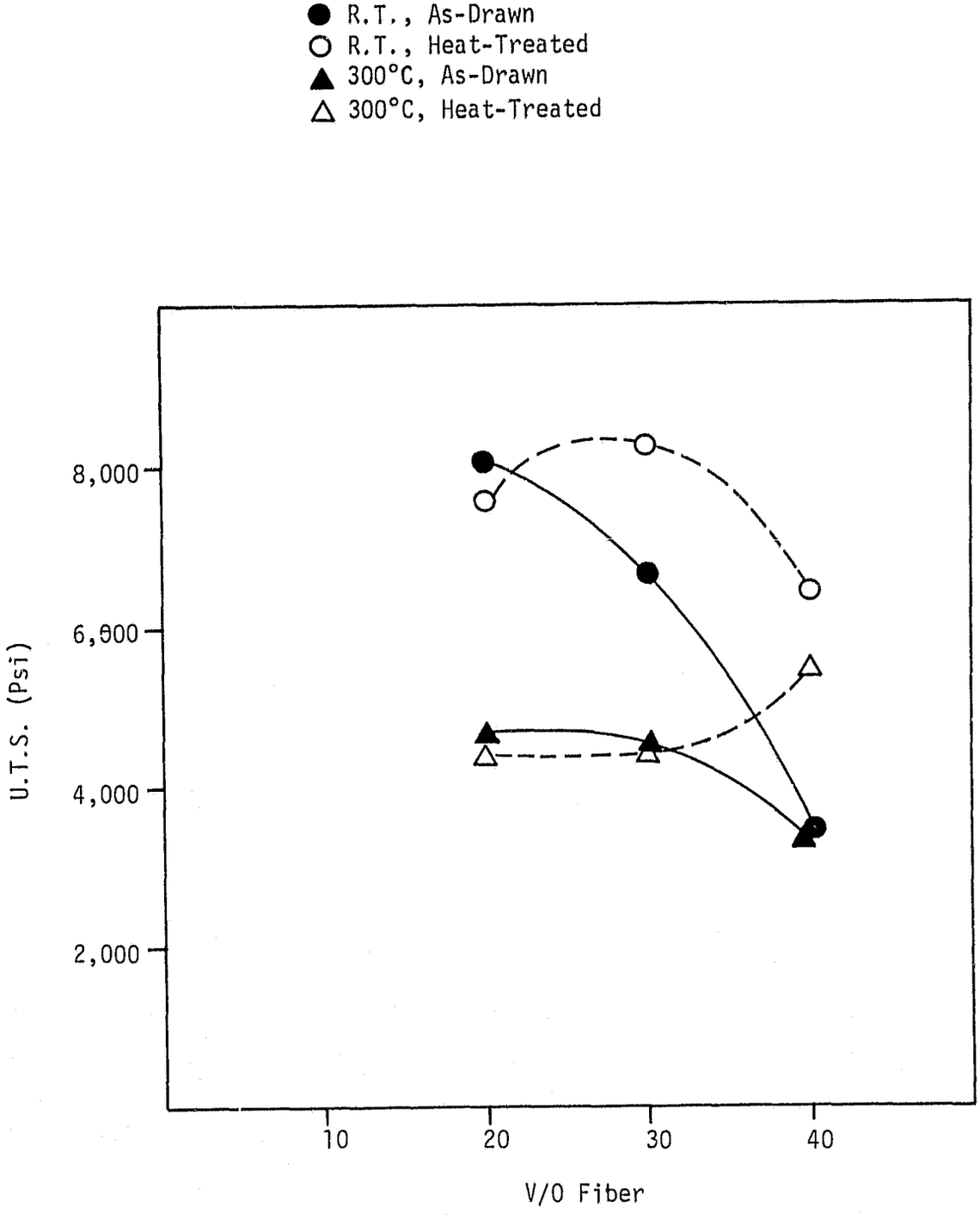



FIGURE 17 - TRANSVERSE U.T.S. Vs. % FIBER FOR $Al_2O_3$/1100 Al BAR STOCK

material appears to yield higher transverse properties than as-drawn material as v/o fiber increases. Obviously, the interfacial reactions which occur over long-time exposures are beneficial to the transverse properties as v/o fiber increases. This suggests compound formation (which is not visible) or an improvement in bonding.

3.1.3 Longitudinal Compression Testing

Room temperature longitudinal compression testing was performed on the 20 v/o fiber material in the as-drawn and heat-treated condition. The sample was 0.70 in. by 0.50 in. by 0.14 in. thick with the fiber orientation being along the 0.70 dimension.

Results of the longitudinal compression test are given in Table 13. The data shows that the compressive strength of the composite is roughly twice that of the tensile strength. Figure 18 is a graph of U.C.S. vs v/o fiber for the as-drawn and heat-treated material at 20, 30 and 40 v/o fiber levels. As can be seen from the curve, there is a marked increase in compressive strength for heat-treated material as the v/o fiber increases. This supports the theory of some interfacial reaction occurring during long-time exposure to elevated temperature suggested from transverse tensile testing.

3.1.4 Transverse Compression Testing

Room temperature transverse compression testing was performed on the 20 v/o fiber material in the as-drawn and heat-treated conditions. The sample was 0.70 in. by 0.50 in. by 0.14 in. thick with the fiber orientation along the 0.50 dimension.

As can be seen from the results shown in Table 14, the transverse compressive strength is again roughly twice that of the tensile strength. There is no 30 and 40 v/o fiber data available to compare due to sample design problems experienced in contract NAS3-21013. There is no apparent effect of long-term exposure to high temperature evident in the test results.

3.1.5 Longitudinal Flexure Testing

Room temperature three point flexure testing was conducted on 20 v/o fiber material in the as-drawn and

TABLE 13

LONGITUDINAL COMPRESSION TEST RESULTS

| Sample# | Condition | Test Temperature | U.C.S. (Ksi) | E (Msi) | $\varepsilon_f$ (%) |
|---|---|---|---|---|---|
| A.06 | As-Drawn | R.T. | 60.4 | 7.8 | 1.30 |
| A.16 | As-Drawn | R.T. | 60.4 | 12.5 | 1.06 |
| A.25 | As-Drawn | R.T. | 51.8 | 8.4 | 0.77 |
| Average | | | 57.5 | 9.6 | 1.04 |
| H.05 | Heat-Treated | R.T. | 55.8 | 15.9 | 0.94 |
| H.15 | Heat-Treated | R.T. | 45.7 | 9.9 | 1.31 |
| H.25 | Heat-Treated | R.T. | 51.3 | 13.6 | 0.315 |
| Average | | | 50.9 | 13.1 | 0.86 |

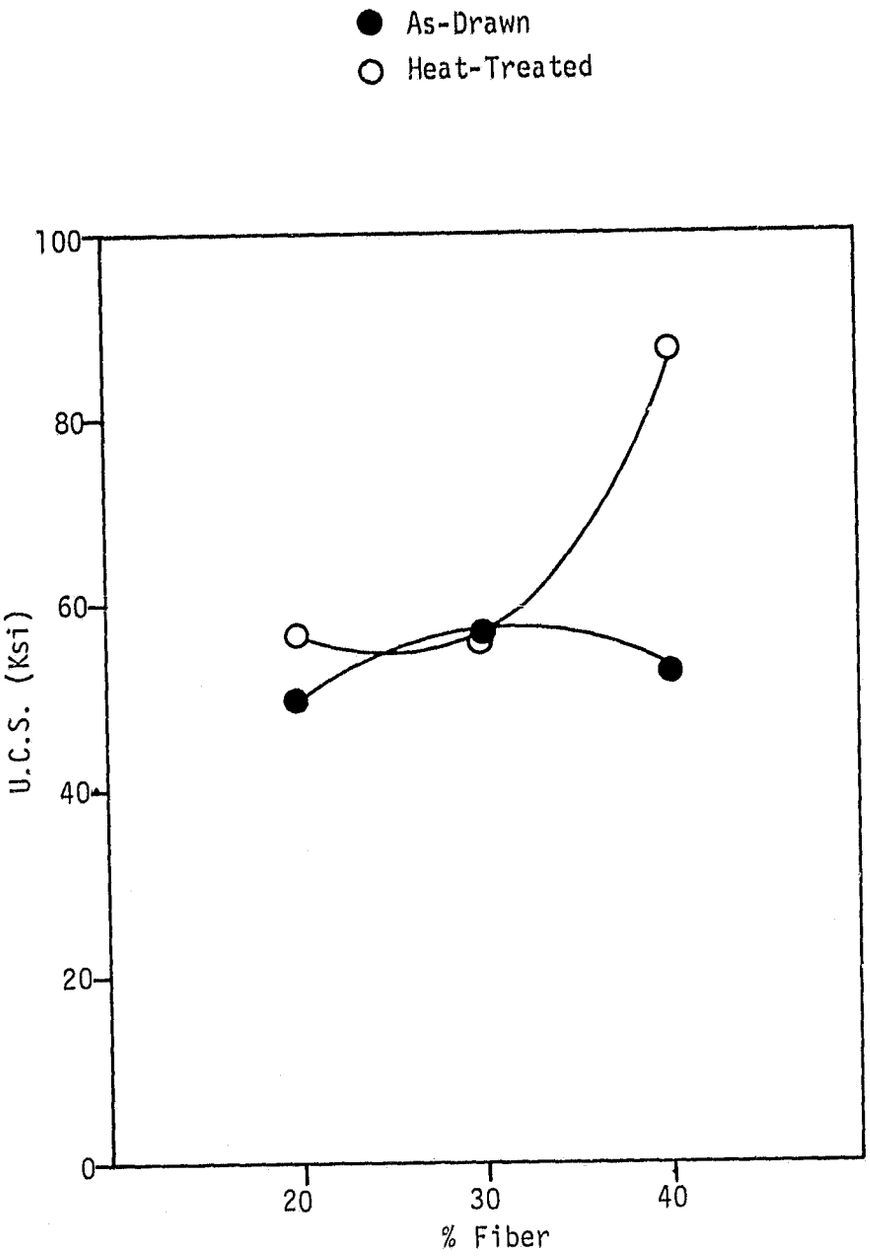



FIGURE 18 - LONGITUDINAL U.C.S. Vs. % FIBER FOR $Al_2O_3$/1100 Al BAR STOCK

TABLE 14

TRANSVERSE COMPRESSION TEST RESULTS

| Sample# | Condition | Test Temperature | U.C.S. (Ksi) | E (Msi) | $\varepsilon_f$ (%) |
|---|---|---|---|---|---|
| A.05 | As-Drawn | R.T. | 16.2 | 7.2 | 1.28 |
| A.15 | As-Drawn | R.T. | 19.0 | 7.2 | 1.30 |
| A.24 | As-Drawn | R.T. | 17.1 | 8.4 | 1.14 |
| Average | | | 17.4 | 7.6 | 1.24 |
| H.06 | Heat-Treated | R.T. | 17.7 | 7.5 | 1.18 |
| H.16 | Heat-Treated | R.T. | 18.3 | 7.7 | 1.22 |
| H.26 | Heat-Treated | R.T. | 17.8 | 6.2 | 1.27 |
| Average | | | 17.9 | 7.13 | 1.22 |

heat-treated conditions. The samples were rectangular in shape with dimensions 4 in. long, 0.25 in. wide and 0.18 in. thick.

Results of the longitudinal flexure tests are given in Table 15. Values for flexual strength are extremely low and test results questionable. Additional testing would have been desirable, but was not practical under the program scope and funding.

3.1.6 Transverse Flexure Testing

Room temperature three point flexure testing was conducted on 20 v/o fiber material in the as-drawn and heat-treated conditions. The samples were rectangular in shape with dimensions 0.70 in. long, 0.25 in. wide and 0.02 in. thick.

Results of the transverse testing are given in Table 16. Again, values for flexural strength were low but additional testing was not practical.

3.1.7 Longitudinal Mechanical Fatigue

Room temperature longitudinal mechanical fatigue testing was carried out on 20 v/o fiber material in the as-drawn and heat-treated conditions. The sample used was identical to that pictured in Figure 12 for the longitudinal tensile specimen. Specimens were cycled at 20 Hz in a tension-tension mode using 4,000 Psi as the value for $\sigma_{min.}$. $\sigma_{max.}$ was determined at various percentages of a $\sigma_{uts}$ value taken as 26,200 Psi.

Results of the longitudinal fatigue tests are presented in Table 17. Figure 19 is a graph of $\sigma_{max.}/\sigma_{uts}$ versus cycles-to-failure of all samples tested, as well as the 30 and 40 v/o fiber samples tested under NASA contract NAS3-21013. Due to material inconsistency and wide variations in tensile and fatigue behavior, the data in general is extremely scattered and consequenty is not suitable for quantitative comparison with the fatigue properties of aluminum alloys and other composites. However, indications are that $Al_2O_3$/Al composites have much greater fatigue life than conventional aluminum alloys.

3.1.8 Transverse Mechanical Fatigue

Room temperature transverse mechanical fatigue testing was performed on the 20 v/o fiber material in the as-drawn and heat-treted conditions. The sample was identical to that

TABLE 15

LONGITUDINAL FLEXURE TEST RESULTS

| Sample# | Condition | Test Temperature | U.T.S. (Ksi) | E (Msi) |
|---|---|---|---|---|
| A.03 | As-Drawn | R.T. | 1.3 | 14.5 |
| A.13 | As-Drawn | R.T. | 1.1 | 13.0 |
| A.30 | As-Drawn | R.T. | 1.2 | 13.2 |
| Average | | | 1.2 | 13.6 |
| H.03 | Heat-Treated | R.T. | 1.2 | 14.3 |
| H.13 | Heat-Treated | R.T. | 1.1 | 16.6 |
| H.23 | Heat-Treated | R.T. | 1.1 | 15.9 |
| Average | | | 1.13 | 15.6 |

TABLE 16

TRANSVERSE FLEXURE TEST RESULTS

| Sample# | Condition | Test Temperature | U.T.S. (Ksi) | E (Msi) |
|---|---|---|---|---|
| A.09 | As-Drawn | R.T. | 1.8 | 4.2 |
| A.19 | As-Drawn | R.T. | 2.1 | 5.0 |
| A.28 | As-Drawn | R.T. | 1.8 | 5.5 |
| Average | | | 1.9 | 4.9 |
| H.09 | Heat-Treated | R.T. | 1.4 | 4.3 |
| H.19 | Heat-Treated | R.T. | 1.7 | 5.2 |
| H.29 | Heat-Treated | R.T. | .875 | 5.2 |
| Average | | | 1.3 | 4.9 |

TABLE 17

LONGITUDINAL FATIGUE TEST RESULTS

| Sample# | Condition | Test Temperature | $\sigma_{max}/\sigma_{U.T.S.}$ | Cycles to Failure |
|---|---|---|---|---|
| A.14 | As-Drawn | R.T. | 0.67 | 94,000 |
| A.23 | As-Drawn | R.T. | 0.75 | 155,400 |
| A.31 | As-Drawn | R.T. | 0.85 | 181,500 |
| H.04 | Heat-Treated | R.T. | 0.61 | 1,985,400 |
| H.14 | Heat-Treated | R.T. | 0.75 | 12,700 |
| H.24 | Heat-Treated | R.T. | 0.85 | 200 |

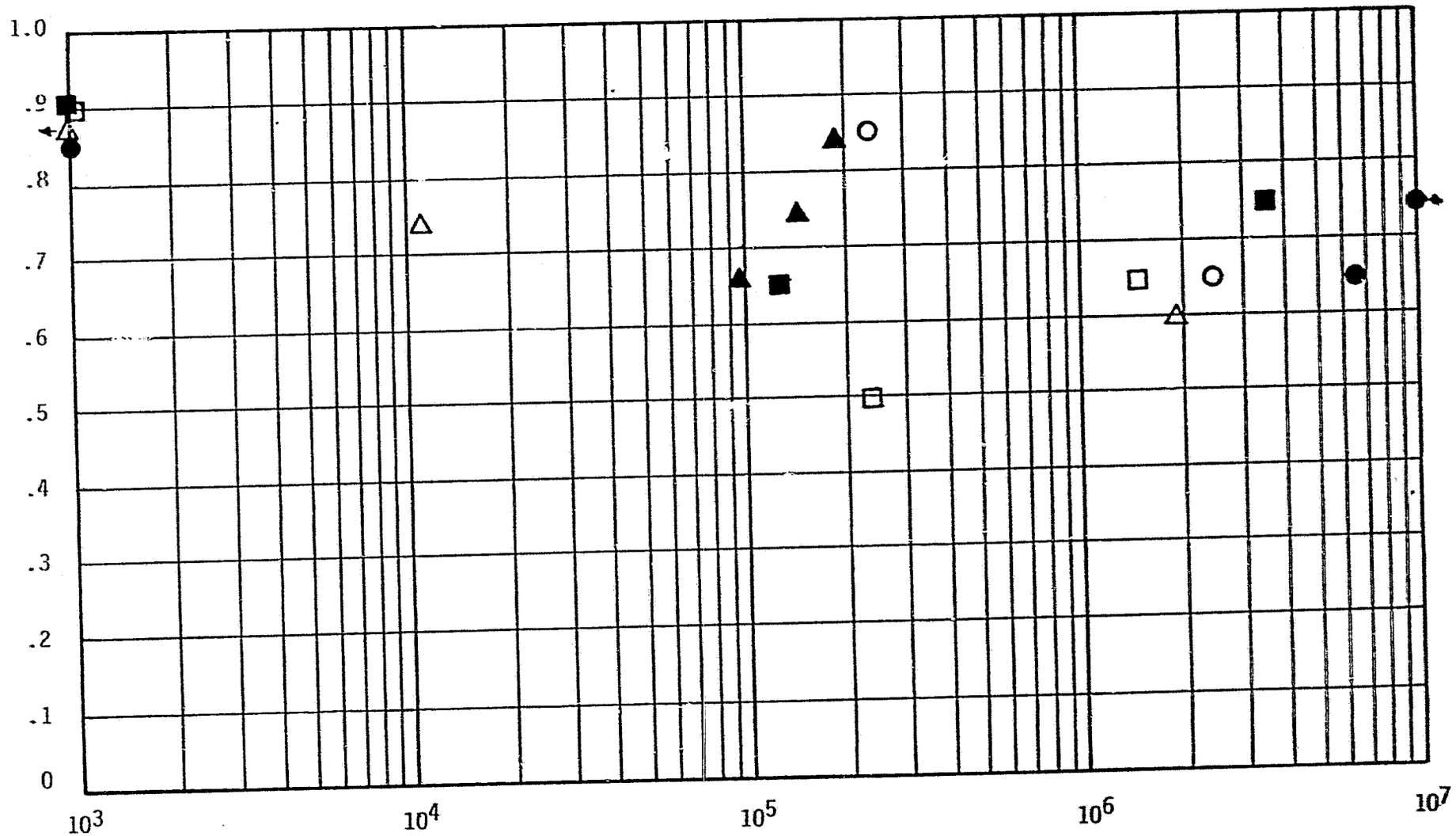



FIGURE 19_ LONGITUDINAL FATIGUE CURVE

pictured in Figure 16 for the transverse tensile specimens. Specimens were cycled at 20 Hz in a tension-tension mode using 1000 Psi as the value for $\sigma_{min.}$. $\sigma_{max.}$ represents various percentages of $\sigma_{uts}$ values for the different material conditions as determined from the transverse tensile tests.

Results of the transverse mechanical fatigue testing are presented in Table 18. Figure 20 is a graph of $\sigma_{max.}/\sigma_{uts}$ versus cycles-to-failure for all samples tested, as well as 30 and 40 v/o fiber material tested under NASA contract NAS3-21013. As can be seen from the figure the cycles-to-failure increase as v/o fiber decreases.

Some difficulty in obtaining data was encountered in both the transverse fatigue and transverse tensile testing due to the design of the specimen. The current pultruded bar stock limits the specimen to a very short length. A long specimen with a relatively long gauge length would be more appropriate and facilitate testing.

3.1.9 Longitudinal Stress Rupture

Longitudinal stress rupture testing was performed at 300°C on the 20 v/o fiber material in the as-drawn condition. The sample was identical to that pictured in Figure 12 for the longitudinal tensile specimen except the gauge width was reduced to 1/8 in. as opposed to the ¼ in. width shown in Figure 12.

Results of the stress rupture testing are presented in Table 19. Unlike the 30 and 40 v/o material tested under NASA contract NAS3-21013, where there appeared to be a critical point in stress where rupture occurred quite rapidly above the critical level, the 20 v/o material seems to have a more gradual transition.

3.2 Thermal Testing Results

Thermal expansion measurements for as-drawn material were preformed over a temperature range of 0-500°C for the 20 v/o fiber material in the longitudinal and transverse directions.

3.2.1 Longitudinal Thermal Expansion Behavior

Longitudinal thermal expansion specimens were 2.0 inches long by 0.14 inches square with all cladding removed.

TABLE 18

TRANSVERSE FATIGUE TEST RESULTS

| Sample# | Condition | Test Temperature | $\sigma_{max}/\sigma_{U.T.S.}$ | Cycles to Failure |
|---|---|---|---|---|
| A.10 | As-Drawn | R.T. | 0.67 | 90,370 |
| A.29 | As-Drawn | R.T. | 0.75 | 5,444,310* |
| A.36 | As-Drawn | R.T. | 0.85 | 1,694,220* |
| H.10 | Heat-Treated | R.T. | 0.61 | 4,524,390* |
| H.20 | Heat-Treated | R.T. | 0.75 | 2,118,600 |
| H.24 | Heat-Treated | R.T. | 0.85 | · |

* Did not fail.

· Did not fail - broke due to machine malfunction.

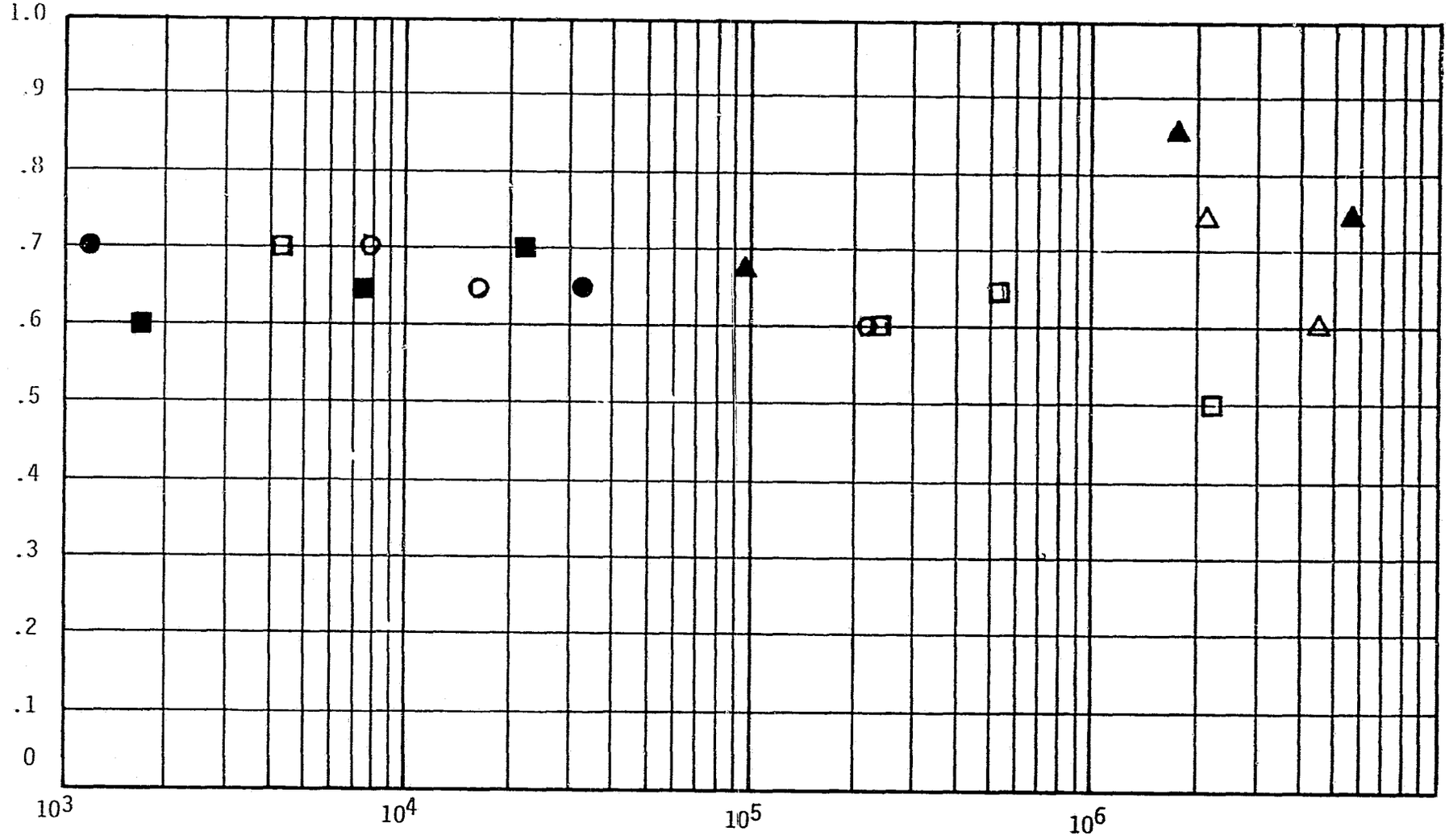



FIGURE 20 - TRANSVERSE FATIGUE CURVE

TABLE 19

STRESS RUPTURE TEST RESULTS

| Sample# | Condition | Test Temperature | Applied Stress (Ksi) | Hours to Failure |
|---|---|---|---|---|
| A.04 | As-Drawn | 300°C | * | -- |
| A.22 | As-Drawn | 300°C | 5.8 | 200.4 |
| A.32 | As-Drawn | 300°C | 9.7 | 1.5 |
| A.33 | As-Drawn | 300°C | 10.9 | 0.3 |

* Specimen failed during set-up.

Figure 21 shows curves of percent expansion vs temperature for the 20 v/o material in comparison with bulkaluminum and alumina. As can be seen from the curves, the longitudinal thermal expansion behavior of the composite, even at only 20 v/o fiber, is dominated by the fiber. This is especially true at elevated temperatures.

3.2.2 Transverse Thermal Expansion Behavior

Transverse thermal expansion specimens were in three equal seperate segments of 0.667 inches each, comprising a total specimen length when assembled together of 2.0 inches. The samples were 0.14 inches square with all cladding removed. Figure 22 shows curves of percent expansion in comparison with bulk aluminum and alumina. As can be seen from the curves, the transverse thermal expansion behavior is completely dominated by the aluminum matrix over the entire temperature range.

3.3 Chemical Analysis

As stated in 2.1, samples of preform wire were submitted for quantitative chemical analysis. Results were reported in Table 4. The data showed that the percentages of each major alloy constituent were within specifications. Ti and B were present in significant amounts due to the Ti/B coating process. Since the composite did not undergo any subsequent chemical processing, it is assumed that these levels remain the same for the bar material.

3.4 General Comments

Several comments about the material fabricated and tested on this program deserve special mention. The first is that the $Al_2O_3$/Al composite material at 20 v/o fiber level is extremely hard to machine, as was the 30 and 40 v/o material. This is due to a marked contrast in properties between the fiber and matrix phases. Grinding tools for ceramics, like $Al_2O_3$, load up with Al when machining the composite, and cutting tools for soft metal, like Al, cannot cut the FP fiber. A compacted diamond cutting tool was used throughout the program for the maching of tensile bars with excellent results.

Care must be taken when comparing the results presented in this report to current results for other metal matrix systems,

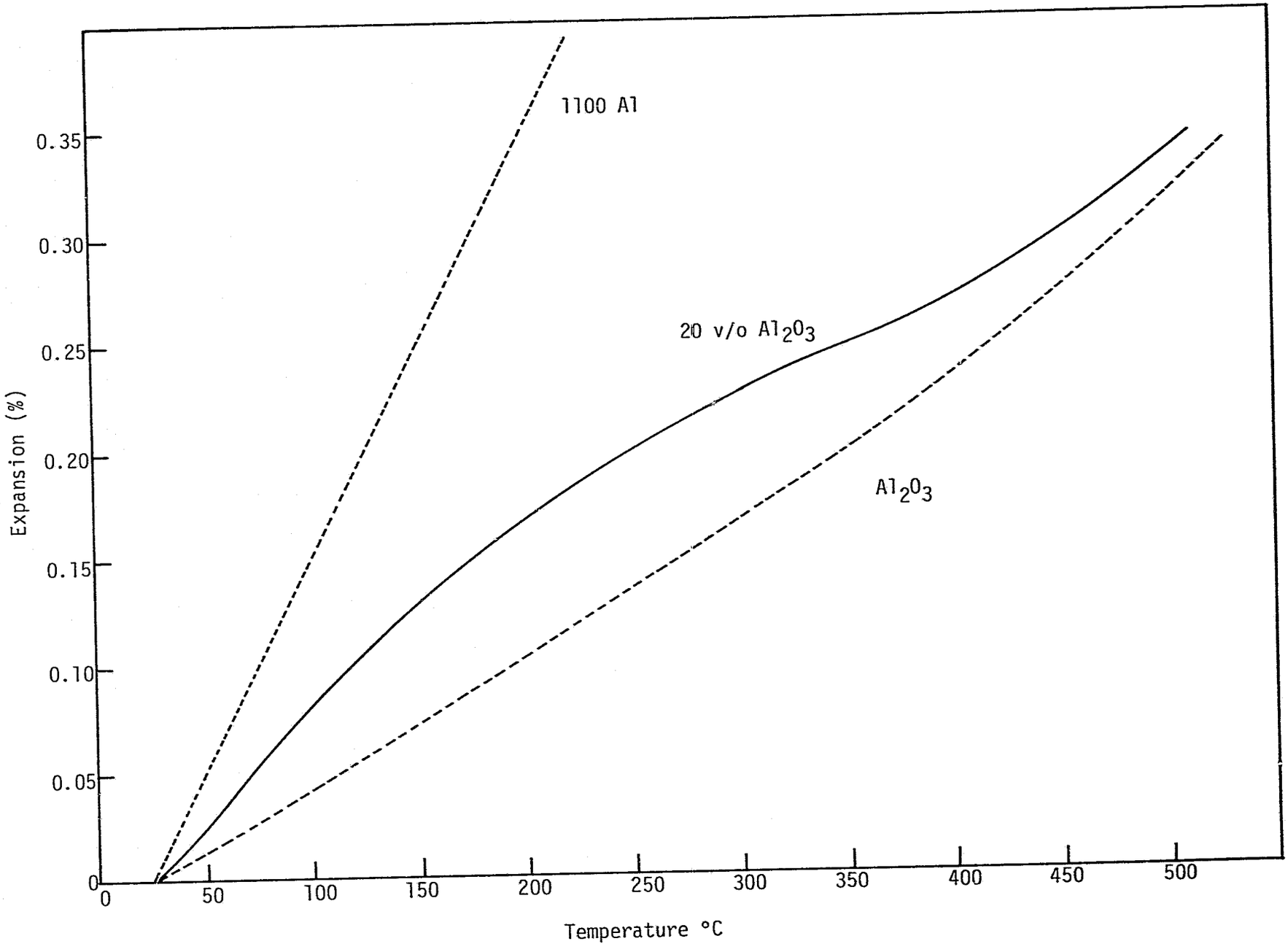



FIGURE 21 - LONGITUDINAL THERMAL EXPANSION BEHAVIOR

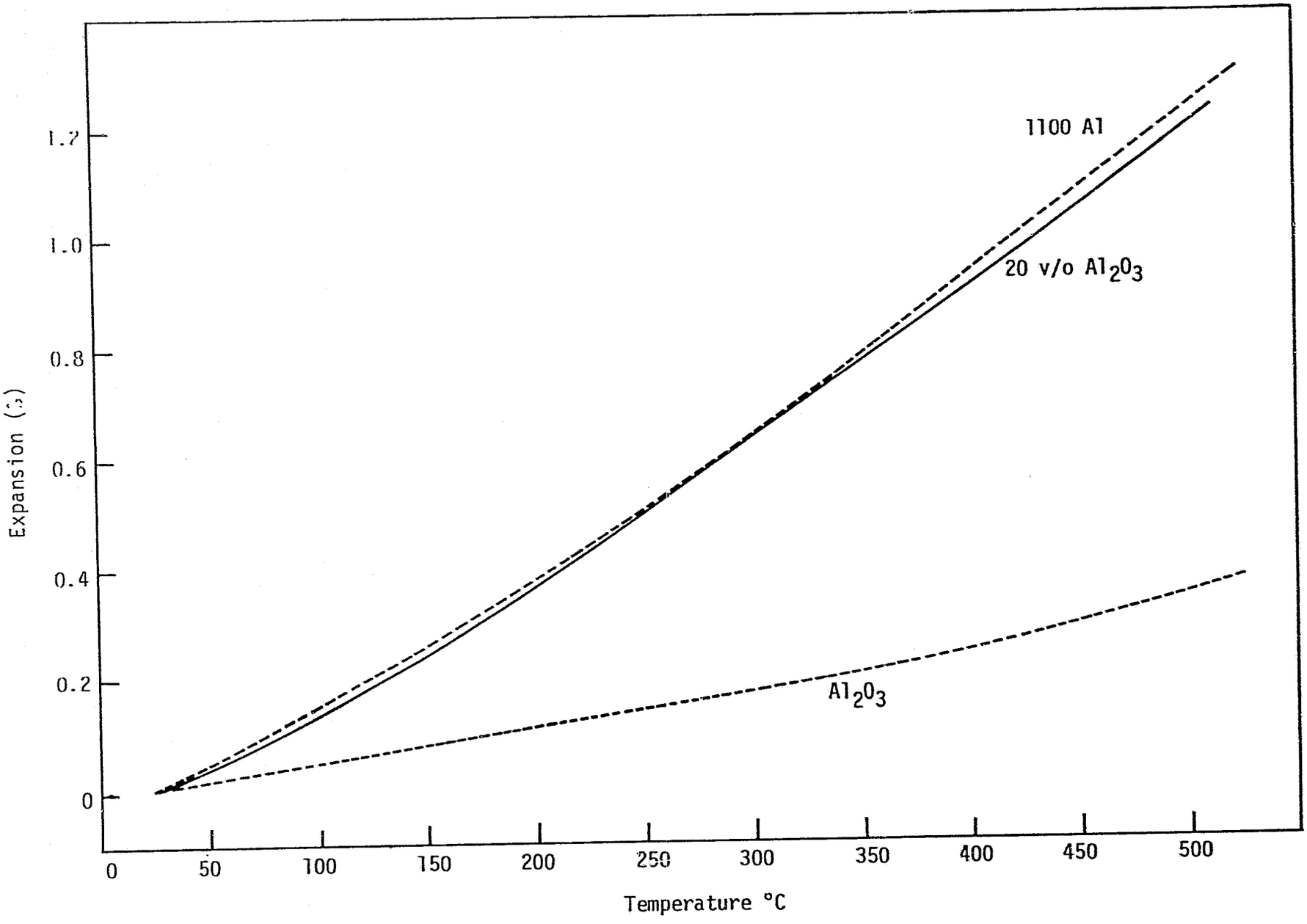



FIGURE 22 - TRANSVERSE THERMAL EXPANSION BEHAVIOR

such as Gr/Al. Results in the literature have generally been taken from material which has been cladded with surface foils of Al. Although material for this program was fabricated with a 3003 Al cladding, the cladding was fully removed from the gauge sections and surface of all test specimens. Thus, the data reported here is just on the bonded $Al_2O_3$/1100 Al composite wires with no Al rich areas present at the surface of the specimens. In addition, the 1100 Al matrix has been shown to yield poorer properties in metal matrix composites than other matrices[4], such as A201 aluminum, and much lower properties than an A201 matrix with 2024 surface and encapsulant foils.[4]

## 4.0 TASK III - SECONDARY FABRICATION TRIALS

Two forms of secondary fabrication were attempted with 20 v/o fiber $Al_2O_3$/Al bar stock. Rolling trials were conducted with the rolling direction perpendicular to the fiber direction. In addition, upset forming trials were performed with the load applied perpendicular to the fiber direction.

### 4.1 Hot Rolling Trials

Rolling was performed at room temperature and after preheating the material at 800°F for 30 minutes. Starting coupons were 0.75 inches wide by 5 inches long. The material was only rolled in the direction perpendicular to the fiber direction since severe edge cracking resulted in rolling 30 and 40 v/o fiber material parallel to the fiber direction during the previous program. The 3003 Al cladding was not removed from the material used in the rolling trials.

Results of the trials are presented in Table 20. As had been expected based on the results from rolling in contract NAS3-21013, rolling perpendicular to the fiber direction yielded good results.

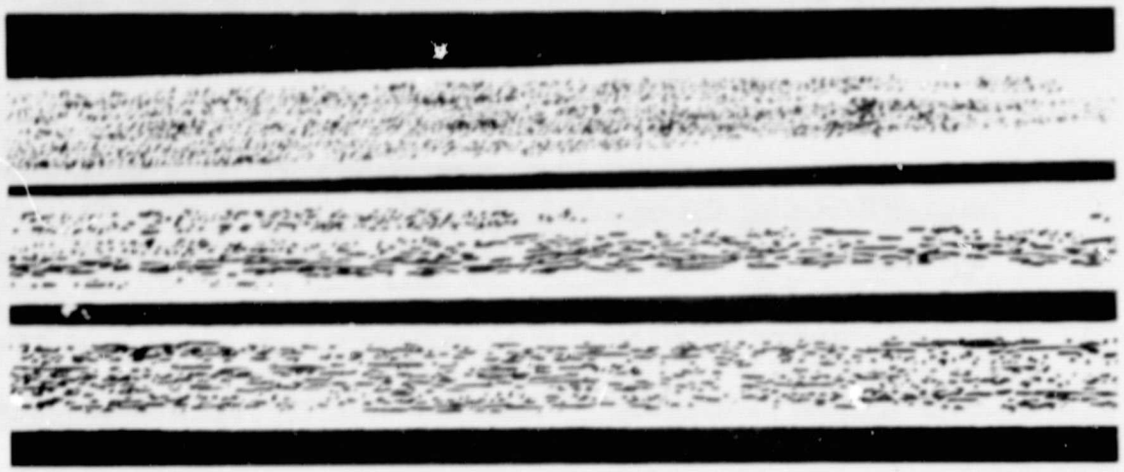
Reductions as high as 59% at room temperature and 69% at 800°F were achieved with only superficial edge cracking occurring. Samples were cut and examined under low magnification and no significant degree of macro cracking could be detected. Even at reductions as high as 80%, edge cracking was still minimal and did not extend more than 3/32 of an inch into the composite.

Figures 23 and 24 show representative sections of cold and hot rolled material at approximately 80% reduction. Again, no significant amount of macro cracking was detected. At high magnification, however, a considerable amount of fiber breakage is evident. In some cases broken fibers have separated, leaving a void in the matrix between. No quantitative analysis or mechanical testing was performed under the program scope. However, this method of secondary fabrication shows some promise and warrants further in-depth investigation.

TABLE 20

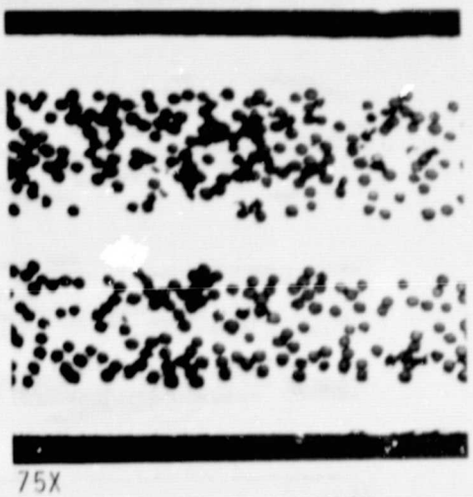
RESULTS OF HOT-ROLLING STUDIES

| Trial# | Test Temperature | % Reduction | # of Steps | Final Thickness (Inches) | Comments |
|---|---|---|---|---|---|
| 1 | R.T. | 41.5 | 2 | 0.110 | |
| 2 | R.T. | 58.7 | 3 | 0.077 | Superficial Edge Cracking |
| 3 | R.T. | 85.2 | 5 | 0.028 | Minor Edge Cracking |
| 4 | 800°F | 43.6 | 2 | 0.105 | |
| 5 | 800°F | 69.4 | 4 | 0.057 | Superficial Edge Cracking |
| 6 | 800°F | 83.3 | 6 | 0.031 | Minor Edge Cracking |



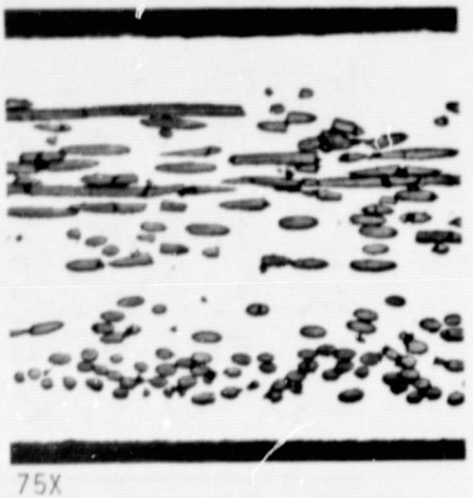
20X

LONGITUDINAL AND TRANSVERSE SECTIONS



75X

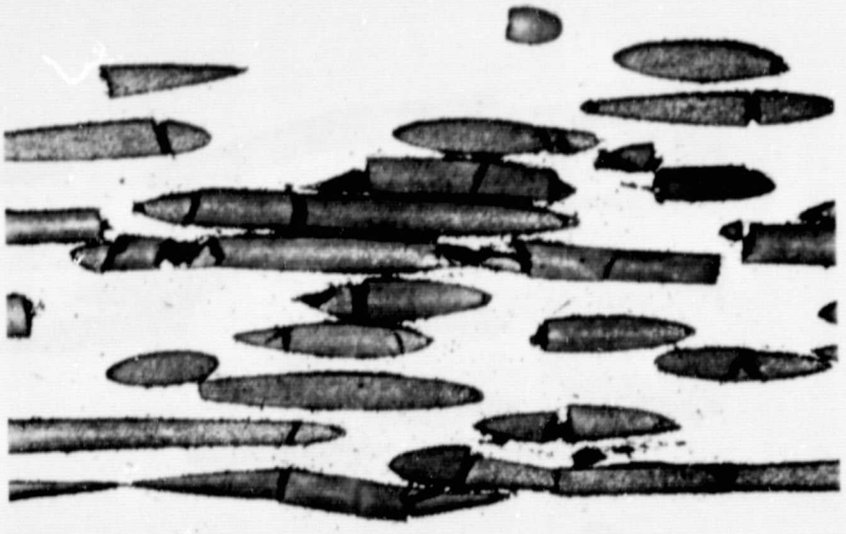
TRANSVERSE SECTION



75X

LONGITUDINAL SECTION





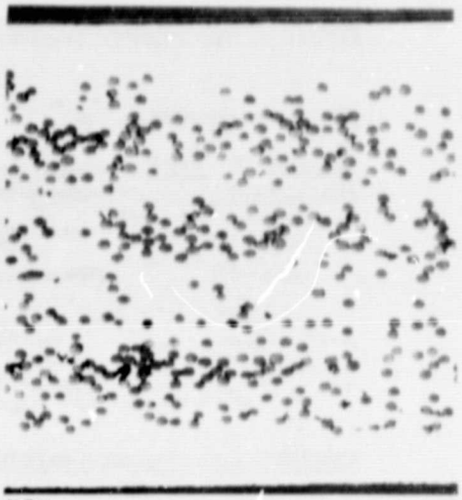
250X

LONGITUDINAL SECTION

FIGURE 23 SECTIONS FROM COLD ROLLED 20 V/O $Al_2O_3$/1100 ALUMINUM BAR STOCK, 85.2% REDUCTION @ R.T.

20X

LONGITUDINAL AND TRANSVERSE SECTIONS

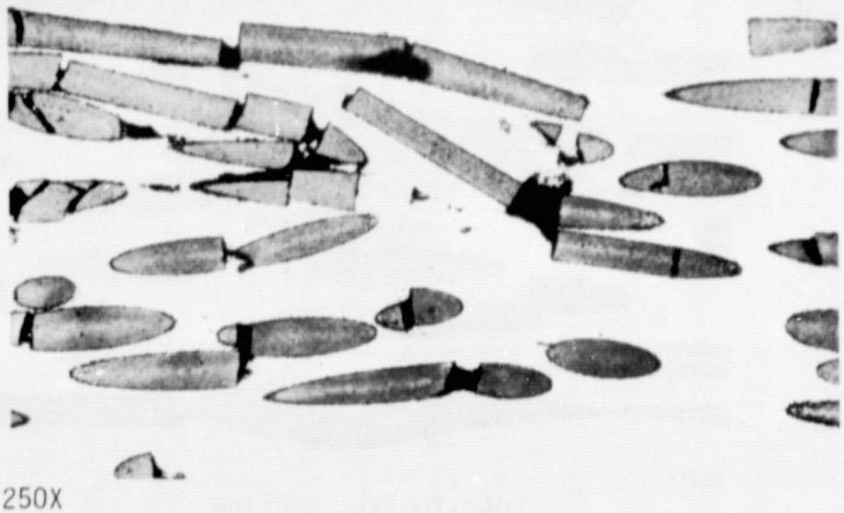


75X

TRANSVERSE SECTION

75X

LONGITUDINAL SECTION



250X

LONGITUDINAL SECTION

FIGURE 24 SECTIONS FROM HOT ROLLED 20 V/O $Al_2O_3$/1100 ALUMINUM BAR STOCK, 83.3% REDUCTION @ 800°F

## 4.2 Upset Forming

Upset forming was performed on the 20 v/o fiber material in such a way as to apply the load perpendicular to the fibers. It was anticipated that this method of loading would produce the least amount of fiber damage. The jig used is shown in Figure 25. Forming took place at a temperature of 800°F with the load being applied as slowly as control would allow. Fracture occurred in all specimens tested in the area where deformation was taking place.

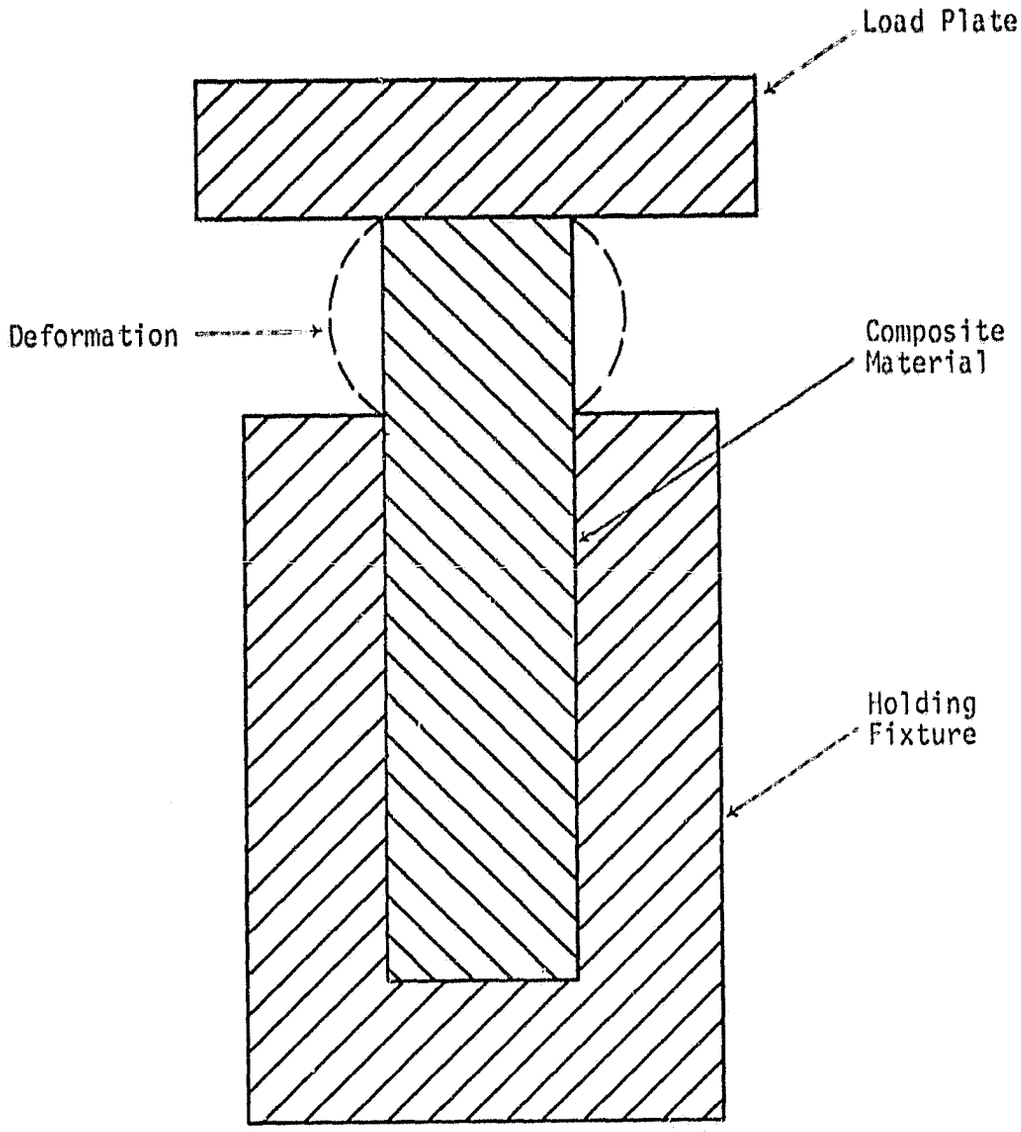



FIGURE 25 - UPSET FORMING JIG

## 5.0 CONCLUSIONS

1) Liquid-metal infiltation of FP $Al_2O_3$ fiber bundles using the Ti/B process to achieve wire preform in the 25 v/o fiber range has been demonstrated.

2) The preform wire exhibits Rule-of-Mixtures (ROM) strengths.

3) The Ti/B process permits liquid-metal infiltration with a wide range of aluminum matrix alloy compositions, allowing optimization of matrix-controlled composite properties by alloy selection.

4) Encapsulation of wire preform to lower the bulk v/o fiber in conjunction with the hot-drawing process is a viable method of producing FP/Al material.

5) The longitudinal composite strengths (about 75% ROM) are related to the low strain-to-failure of the alumina fiber and consequent fiber breakage during the hot-drawing consolidation process.

6) That chemical reaction or improved bonding may occur between the aluminum alloy matrix and the $Al_2O_3$ fibers during long term heat treatment at 300°C.

7) Fabrication of composite shapes may be possible by hot rolling in a direction perpendicular to the fibers.

8) Excellent potential for long fatigue life at high temperatures exists in the $Al_2O_3$/Al composite system.

## 6.0 RECOMMENDATIONS

1) Investigate ways of improving the fiber itself so that it is easier to get into the composite and will translate higher strengths to bulk composite maerial.

2) Investigate hot-rolling as a secondary fabrication technique to form thin sheets and curved sections using the concept of deformation in a direction perpendicular to the fiber surface.

3) Conduct a more extensive study of the fatigue behavior of $Al_2O_3$/Al composites.

4) Investigate methods for preform wire consolidation that will result in translation of the precursor ROM properties to the bulk composite.

5) Investigate methods to make near-net-shape composites by direct infiltration into Ti/B coated alumina fiber preforms, capitalizing on the capability of the process to utilize a wide range of aluminum matrix compositions.

## 7.0 REFERENCES

1) Amateau, M. F., Aerospace Corporation, Private Communication, March, 1977.

2) Dhingra, A. K. and Kreuger, W. H., "Fiber FP Continuous Alumina Yarn", Special Report, E. I. DuPont De Nemours & Co., Inc., Wilmington, Delaware, 1975.

3) Kendall, E. G. and Pepper, R. T., "Reinforced Metal Matrix Composite", U. S. Patent 4,082,864, April 4, 1978.

4) Webb, B. A. and Dolowy, J. F. Jr., "Graphite/Aluminum Composites and Hybrids for Launch Vehicle Applications", Final Report on Strategic Missile Materials Technology (SMMT) Program, Report No. 210-4, September, 1977.