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FORMATION OF BORON FIBER-ALUMINUM COMPOSITES
BY DRAWING PROCESSES

by

Clifford D. Estes, LT.,USN

S.M. & NAV.ENG.

COURSE XIII A

May 1966

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University
of California Postgraduate School
Berkeley, California

FORMATION OF BORON FIBER-ALUMINUM COMPOSITES
BY DRAWING PROCESSES

by

CLIFFORD D. ESTES, LT.,USN
//

S.B. U.S. Naval Academy (1959)

Submitted in Partial Fulfillment of the Requirements for the

Master of Science Degree

in Naval Architecture and Marine Engineering

and the Professional Degree, Naval Engineer

at the

Massachusetts Institute of Technology

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FORMATION OF BORON FIBER-ALUMINUM COMPOSITES
BY DRAWING PROCESSES

by

CLIFFORD D. ESTES, LT., USN

Submitted to the Department of Naval Architecture and Marine Engineering on 20 May 1966 in partial fulfillment of the requirements for the Master of Science Degree in Naval Architecture and Marine Engineering and the Professional Degree, Naval Engineer

ABSTRACT

This work was undertaken to examine and develop a method of producing a boron fiber-aluminum composite by a drawing process. A method of placing boron fibers in aluminum tubes, joining the tubes by brazing, and hot drawing to produce 100% dense specimens was derived. Stress-strain data showed an increase in tensile strength of 100%. A cold drawing process consisting of passing a bundle of aluminum tubes with fibers inside was proven to be unfeasible, because the fibers broke before full density was reached. Basic hot drawing through consecutive dies using an outer tube of stainless steel produced dense specimens. However, it was found that there was little bonding among the aluminum tubes and only a minimal increase in strength resulted. The hot drawing process, preceded by dip brazing the aluminum tubes together, is a satisfactory process for composite preparation.

Thesis Supervisor: J. W. Mar

Title: Professor of Aeronautics and Astronautics

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I. INTRODUCTION

The increased requirements of performance in material applications is taxing conventional materials to the limit. Composites hold the potential of providing, in a single materials system, the combination of properties being sought for in many applications. A composite is a materials system composed of two or more dissimilar materials that are essentially insoluble in one another. The constituent materials work together to achieve a new material whose properties are an optimum combination of the properties of the constituents. Forms used in the structuring of composite materials include fibers, whiskers, and unidirectionally solidified eutectic alloys. Use of whiskers in composites has a very high potential based on theoretical strengths. The effect of orientation and shape of the whiskers on overall strength, the reaction between the whiskers and matrix, and the individual properties achievable from whiskers are presently being investigated by many different research organizations. It has long been known that wire forms of materials exhibit higher strength than do bulk properties. Recently, the availability of fine wire or fibers of a wide variety of metals and other materials of high strength present a promising chance to develop composites capable of utilizing their properties. The fibers are combined properties. The unidirectionally solidified eutectic alloys result from a liquid to solid transformation involving a liquid of fixed composition transforming simultaneously to two solids at a constant temperature. When the reaction is carried out unidirectionally, an aligned microstructure of two solid phases results. If one of the phases

possesses whisker-like properties, then a practical reinforced composite will result. Investigation is being conducted on these alloys to overcome such problems as a fixed volume fraction and the high purity materials required.

A fiber composite was investigated in this study. Fiber reinforced composites gain their strength by transferring the applied load to the strong fibers. If the processing conditions are optimum, the volume fraction rule applies. By this rule, if the stress-strain relationships of each component are known, the stress on each can be computed for a given strain and their combined action can be determined according to the following relation¹:

$$\sigma_c = V_f \sigma_f + V_m \sigma_m \quad (1)$$

σ_c = tensile strength of composite

σ_f = tensile strength of fibers

σ_m = tensile strength of matrix

V_f = volume fraction of fibers

V_m = volume fraction of matrix

McDanel, Jech and Wheeton¹, investigated tungsten-filament-reinforced copper composites. It was found that the stress-strain behavior of composites of this type, showed that the volume function rule applied in the continuous fiber case.

Cratchley² studied a composite of stainless-steel fibers in an aluminum matrix to assess the effect of salient parameters on the ultimate tensile strength of the composite. The considerations for choosing these components were that the difference in modulus between the components should be of the right order to enable the fibers to carry the major share of the load on the composite and that the fibers should be considerably stronger than the matrix. Further, to simulate the behavior of brittle oxide fibers, the wires should be fully work hardened so that on loading they remain elastic up to, or near to, the point of fracture. Within the range studied, 0 - 20% reinforcement, the UTS of the composite was linearly proportional to the percentage reinforcement. This supports equation (1). At 20% volume of fibers and length/diameter ratio of 400, an UTS of about 44,000 psi was measured. The aluminum alone had a breaking stress of 13,400 psi and the wire 192,000 psi.

The system of steel wires in a silver matrix has been studied by Piehler³. It was found that the strain of the composite was about twice the strain of the wires alone at failure. The UTS was above that given by the volume fraction rule. The failure of the composite generally occurred as a result of consecutive failures of the wires.

Studies in fiber composites have demonstrated fatigue and creep improvement as well. A system of steel wires in an aluminum matrix was investigated by Forsyth⁴. The principal aim of the work was to reinforce the metal by strategically placing the wires to hinder fatigue crack propagation. Sheets of composite material were prepared by hot rolling

continuous wires between sheets of aluminum alloy DTD 687 (aluminum, zinc, magnesium, copper). The ability of 10% volume of steel wires to restrict the propagation of fatigue cracks was clearly demonstrated. Also, the fracture toughness of the material was increased. Wires laid at 45 degrees to the sheet axis gave more improvement than wires laid parallel. Creep and tensile properties were improved also.

The volume fraction rule is true only when specified conditions are met. These conditions are: (a) the fibers are aligned, (b) a satisfactory matrix interface bond is attained, and (c) there are no deleterious reactions between the fiber and the matrix. The following examples show instances where these conditions were not met and the volume fraction rule predictions were not attained. Petrasek and Weeton⁵ examined damage to tungsten fibers in metals including copper. They found that body-centered cubic (BCC) materials did less damage to tungsten fibers than did face-centered cubic (FCC) materials. FCC materials, which have low solubility with tungsten but have high diffusion rates, are detrimental. BCC materials have melting points closer to tungsten and are isomorphous with tungsten. In tests conducted, damage to the fibers resulted in strengths less than that predicted by the volume fraction rule. In other work, Jech, McDanel, and Weeton⁶ have shown that molybdenum metal fibers 10 mil. in diameter, cut to lengths 1/10 to 1/4 inch and added as a reinforcement phase to Ti-6Al-4V alloy, significantly increased the strength over unreinforced alloy at room temperature. The elastic modulus showed from 25% to 65% increase over unreinforced composition. These results are less than predicted by the volume fraction rule. Proper alignment

was one factor not achieved in these studies. Williams and O'Brien⁷ gravity die-cast aluminum in a stainless steel mold in which a number of steel wires were held under tension. The as-cast LM6 aluminum alloy (silicon, copper, iron, magnesium) had a breaking stress of about 370,000 psi and 6.8% elongation at fracture. The results obtained were close to those given by equation (1) at about 5% volume content but they were considerably below the predicted value above 10% volume content. The effect of heat treatment reduced the breaking stress of the steel wires. Also, the formation of an aluminum-iron intermetallic apparently led to a poor bond between the aluminum and steel.

Processing techniques affect the variables necessary for the volume fraction rule to apply. Several different techniques have been used. Powder metallurgical techniques were used by the Clevite Corporation⁸. The addition of 18.6 volume per cent tungsten wire to a cobalt metal matrix, processed by hot pressing, increased the 2000°F yield stress from 2400 psi to 21,000 psi. Pressing can break the fibers when this method is used and it is also often difficult to obtain proper interfacial bonding. The melting and casting of metals around high-strength fibers has been used most often by investigators as the process for the preparation of fiber-metal composites. McDanel, Jech, and Weeton¹ successfully reinforced copper with tungsten fibers by this procedure. In addition, this method has been successfully used by Koppenaal and Parikh⁹ on the strengthening of silver with steel fibers. Correct alignment and control of the reaction with the fiber are problems when this method is used. Drawing techniques were successfully employed by

Piehler³. The steel wires were placed in silver tubes and then bundles of 7 and 19 each were cold drawn with intermediate stress-relieving.

Of the various strengthening materials considered, boron is one of the most promising. First, boron fibers of high strength can be produced. Boron has a high elastic modulus (approximately 55×10^6 psi) and a high tensile strength (approximately 350,000 psi). The modulus and tensile strength to density ratios are also very high. In certain technical areas today there exist circumstances where the cost is secondary to reliability or where the feasibility of achieving specific properties in materials influences the success of the whole program. A high modulus to density or tensile strength to density ratio is very significant in many of these areas. The interest in boron fibers is therefore significant.

Boron fibers in an aluminum matrix is one of the composites which has achieved only limited success. It is a very promising material, however, when one considers the high strength to density ratio. Only a minimal increase in strength properties has been developed. The reaction between the two materials has not been properly controlled and no favorable method of specimen fabrication has been developed. Hot pressing boron fibers in an aluminum matrix resulted in an essentially fully dense matrix. However, the interfacial bond between the boron and matrix remains to be evaluated. Preliminary investigations of the vacuum infiltration casting technique have been unsuccessful (not published).

The purpose of this paper is to examine and develop drawing methods of producing a boron-aluminum composite for controlled test purposes.

Continuous boron fibers will be used. Comparison will be made with the predictions given by the volume fraction rule. Processing techniques and their influence on the stress-strain relationships of the composite will be studied. Specimens will be tested to determine fracture properties.

II. EXPERIMENTAL PROCEDURES

Drawing processes were investigated to determine a method of preparing boron fiber-aluminum composite specimens for test purposes. In all cases, boron fibers were first placed in individual aluminum tubes. Seven such tubes were then placed in a larger aluminum tube. The following methods were examined: (1) cold drawing, (2) hot drawing, and (3) dip brazing the inner tubes first and then hot drawing. The details of these procedures and the methods of examination and testing of specimens will now be described.

A. Cold Drawing

Several boron fiber-aluminum composites were prepared by cold drawing through a series of dies. The boron fibers were first cleaned by rinsing and wiping with acetone to remove foreign particles and the oily film which covers the fibers when received. Properties of the fibers used in these experiments are listed in Table I. The fibers, cut in four inch lengths, were placed by hand in 3003 aluminum alloy tubes*. These tubes were .0105 inch O.D. and about .005 inch I.D. Seven of these tubes containing boron fibers were placed in an outer aluminum tube (.068 inch O.D., .037 inch I.D.). Specimens so prepared were then drawn through a series of dies either by hand or with the use of a die holding apparatus on an Instron testing machine. The rate of draw on

* 3003 aluminum alloy contains the following elements: 1.2% Mn, .6% Si, .7% Fe, .2% Cu, and .1% Zn. See Table II for list of properties.

TABLE I

Boron Fibers Used in Composite Preparation

Manufacturer	United Aircraft, Inc.
Diameter	.004" \pm .001"
Modulus of Elasticity	55 x 10 ⁶ psi
Tensile Strength (Manufacturer's)	350,000 psi, min.
Tensile Strength (Measured)	327,500 psi, avg. (excluding tests below 70,000 psi)
Substrate	.0005" Tungsten

TABLE II

Aluminum Alloy 3003 Used in Composite Preparation

Manufacturer	Uniform Tubes, Inc.
Diameter (1) Small Tubes	.016" O.D./ .0103" I.D.
(2) Large Tubes	.124" O.D./ .096" I.D.
Modulus of Elasticity	10.5 x 10 ⁶ psi
Tensile Strength	17,260 psi

the Instron was two inches per minute. The specimens were drawn through two dies to .0619 inch O.D. The fibers were heard breaking when passing through the .0619 inch die.

Some specimens prepared by cold drawing were zone melted. Zone melting was accomplished with specimens two inches in length mounted vertically in an electron beam melting apparatus. In this equipment, a small localized melted zone can be formed on the mounted piece by a focused electron beam. The process takes place in a vacuum about 5×10^{-6} mm Hg. The mechanically driven beam travels along the piece, at a preselected rate, and melts a small length at a time. The purpose for using this equipment on the composites was to fill voids in the specimen by allowing the molten aluminum to flow. The fibers in the specimen were held in place by the solid aluminum above and below the molten zone. The zone formed was about one-quarter to one-half inch in length. The zone was passed over the length of the specimen either one, two, or three times to determine the effect of multiple passes.

B. Hot Drawing

Composites were hot drawn through successive dies by using a furnace attached to an Instron testing machine. Preliminary experiments were conducted to determine an adequate lubricant at various temperatures. Molybdenum-disulfide paste and silicone oil were examined for their lubricating properties. A relative comparison of the effectiveness of these lubricants is given in Table III. Molybdenum-disulfide had the better lubricating properties at the temperatures used in the final drawing process. The furnace used consisted of a dual wound coil heater with the top and bottom sections having individual temperature control. A holder was machined in the top section of the furnace to

TABLE III
Lubricant Comparison at Various Temperatures

Temp.	Moly-disulfide		Silicone Oil	
	Max	Steady	Max	Steady
65°C	10.6*	9.0	-	-
147°C	8.2	6.5	6.0	5.6
240°C	6.7	6.0	8.0	8.0
325°C	6.1	3.5	7.6	5.3
400°C	5.5	3.5	10.1	4.7

* Relative numbers for the force required to pull a coated tube through a given die.

hold the die in place while drawing. The furnace was ten inches high and three inches in diameter.

Several variations in specimen preparation were tried in an attempt to achieve 100% dense specimens. In the first series of tests, the fibers, four inches in length, were individually placed in 3003 aluminum alloy tubes drawn to .0105" O.D. Seven of these tubes were put in a larger tube as described under Cold Drawing. In some specimens, the inner tubes were preannealed. The specimens were then covered with a molybdenum disulfide paste which was allowed to dry. They were heated uniformly in the drawing furnace and then hot drawn at a rate of two inches per minute. The temperature of the furnace was varied to determine the best

drawing temperature. The temperatures used were 400, 500, 550, and 590°C. The specimens were reduced from .0688 inch to .0619 inch by drawing through two dies. In the second series of tests, a tube of different material was fitted around the specimen described above. The work-hardening jacket was considered necessary as the specimens prepared with only the outer aluminum tube were not dense. This jacket was expected to absorb most of the stress during drawing and prevent longitudinal fiber breakage. Copper and stainless steel tubes were used. Three temperatures were used during hot drawing, 500, 550, and 590°C. When it was found that the aluminum tubes were not bonding together, the inner tubes were coated with a welding flux (AIRCO Napolitan Aluminum Welding Flux No. 40) before hot drawing. It was hoped that the welding flux would activate during hot drawing and cause the tubes to weld together. Some of the specimens prepared by hot drawing were zone melted to determine the effect on density and on tube bonding. The procedure used for zone melting was the same as described under Cold Drawing.

C. Dip Brazing Followed by Hot Drawing

Joining aluminum is difficult because of the aluminum oxide layer formed on aluminum surfaces. Dip brazing is a process in which the oxide layer is removed by a salt bath and a low melting eutectic of aluminum containing silicon joins the sections together. The eutectic, or brazing material, melts at a temperature below that of the aluminum to be joined. This brazing yields a strong weld. In these experiments, a bundle of seven aluminum alloy tubes with boron fibers inside, were

wrapped with a wire form brazing material (ALCOA 718). They were then preheated to 520°C and placed in a salt bath (Aluminum Dip Brazing Salt, D, Park Chemical Co.) at 590°C to accomplish the brazing. The brazed tubes were then put in an aluminum tube and an outer stainless steel tube. Hot drawing was accomplished as previously described except at a temperature of 500°C. This temperature is below the melting point of the brazing material.

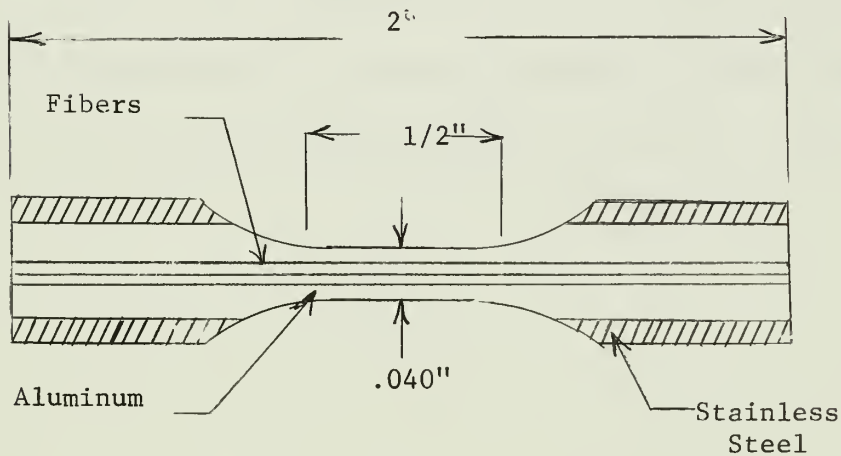
D. Methods of Examination

The specimens were examined to determine if they were dense (i.e., if the aluminum was packed tight against the boron fibers and if the spaces among the aluminum tubes were filled), and to determine if the fibers were broken. The following methods were used:

1. Specimens were examined microscopically. Transverse cross sections of the specimens were mounted in bakelite mounts. Polishing was done on silicon carbide paper and on diamond polishing wheels.
2. Photographs were taken of the cross sections and of fractured surfaces of tested specimens. A Reichert metallograph was used at magnifications of 100X and 500X. Several such photographs are contained in the results part of this report.
3. The state of the fibers were determined by microscopic examination and/or by dissolving the aluminum surrounding the fibers.

E. Methods of Testing

Test specimens were made by dissolving, machining, or sanding off the excess material (aluminum or stainless steel depending on the method of preparation). The specimens were two inches long and had a one-half inch gage length. The diameter of the gage length was reduced to give an outside wall thickness approximately equal to twice the distance between fibers. This couldn't be done in all cases, however, because of the geometry of the specimen. The specimens were then tested on an Instron testing machine. Below is a sketch of a typical test specimen.



TENSILE TEST SPECIMEN

Twelve boron fibers were tensile tested. One inch and three inch lengths were used. The ends of the fibers were placed in copper tubing

holders filled with epoxy resin which was allowed to harden. This holder was used to prevent fiber breakage in the grips.

Properties of 3003 aluminum alloy were determined from tests on 1/4 inch rolled sheet stock. Two methods were used for preparing the aluminum for testing. One piece cut with the direction of roll (longitudinal) and one against the rolling direction (transverse) were machined into four inch tensile specimens having a 2.2 inch gage length of .147" in diameter. These were sintered at 550°C for one hour and were then tested on the Instron. Longitudinal and transverse specimens were also machined into bars .135 inch in diameter. These were cold drawn to .130 inch and hot drawn through two dies at 550°C to .117 inch in diameter. These were then machined into four inch tensile specimens having a two inch gage length of .096 inch diameter. An extensometer was used for strain data when these specimens were tested on the Instron.

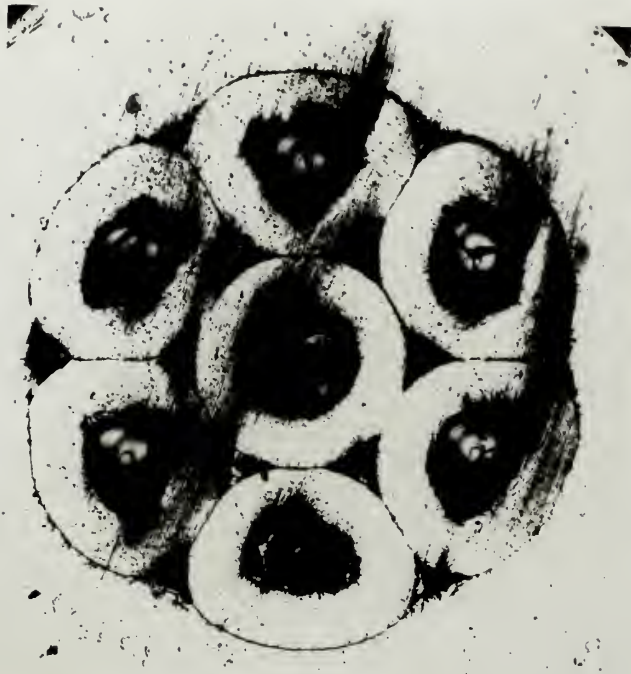
III. EXPERIMENTAL RESULTS

Experimental results are listed in the following sections. The first section contains results relating to the various process techniques. The second section contains results of the tests and analysis made on the composite specimens.

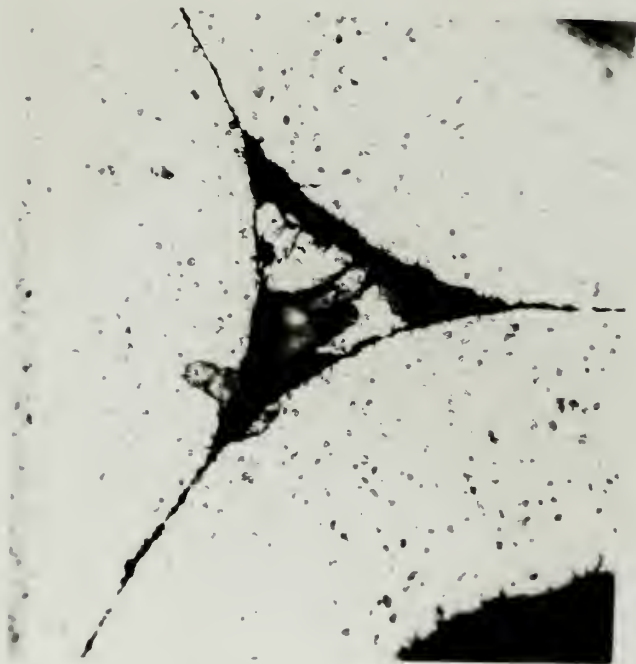
A. Processes

1. Cold Drawing.

In the cold drawing process, the boron fibers were determined to break in a longitudinal direction considerably before densification of the composite. Figure 1 and 2 show a cross section of a typical specimen prepared by this method. The fibers appear shattered (see Figure 2B). The composite was not 100% dense as is shown in Figure 1 and thus the aluminum was not forced tightly against the fibers. The fibers are then able to move slightly during polishing or cutting and the vibrations can cause the shattering of the fibers. Voids always existed at the interstices of the composites prepared by cold drawing. Cold drawn specimens which were zone melted were 100% dense. However, the geometry of the fibers in the aluminum was altered during this melting process. The microstructure of such a specimen is given in Figure 3. Zone melting was not satisfactory. The exact state of the aluminum could not be determined during melting. Sometimes the aluminum overheated and ran down the specimen rather than being held by viscous forces. The fibers would be exposed when this happened.

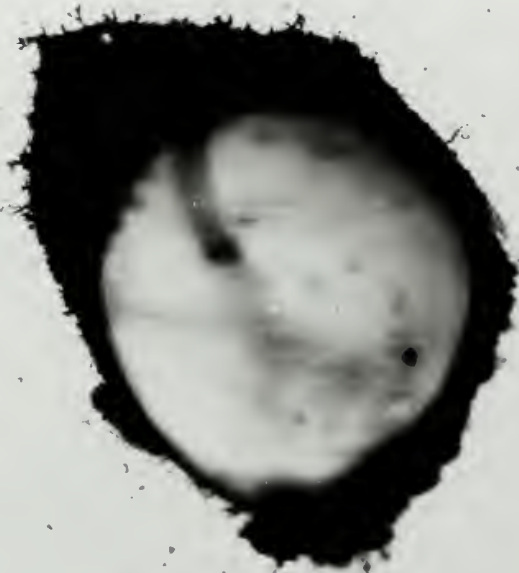


A. COMPLETE CROSS SECTION, 100X



B. INTERSTITIALS, 500X

FIGURE 1. MICROSTRUCTURE OF COLD DRAWN SPECIMEN



A. END VIEW OF FIBER IN MATRIX, 500X

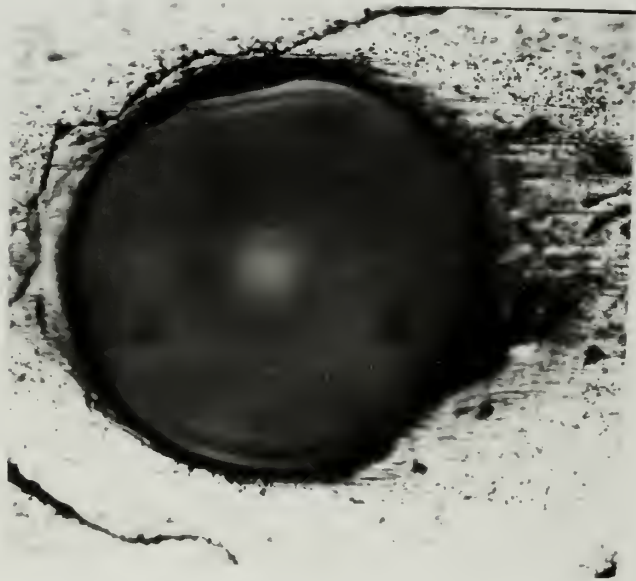


B. FIBER QUARTERED BY CUTTING OR POLISHING, 500X

FIGURE 2. MICROSTRUCTURE OF COLD DRAWN SPINNEY



A. COMPLETE CROSS SECTION, 100X



B. END VIEW OF FIBER IN MATRIX, 500X

PLATE 3. MIC. CHARACTER OF A ZONE HEATED GOLD-BROWN SPECIMEN

2. Hot Drawing.

Hot drawing was more satisfactory. Specimens were drawn at 550°C, as this was found to be the optimum temperature. Specimens with just the outer aluminum tube were hot drawn to the point of fiber breakage and the microstructure revealed that the composite was not dense. Spaces existed between the aluminum tubes and around the fibers. The specimens appeared similar to that represented in Figure 1, except that they were more dense than those prepared by cold drawing. The addition of a stainless steel tube surrounding the specimen to be drawn, resulted in 100% dense specimens. Figure 4 is the microstructure of a cross section of a specimen prepared in this manner. There are no spaces around the fibers or between the aluminum tubes. The shadows seen surrounding the fibers in Figure 4A are present because during polishing, the aluminum polishes faster than the boron and thus the boron extends above the surface of the aluminum. Figure 4B is the end view of one of the fibers. The fibers in these specimens were not broken. The microstructure of a composite prepared by hot drawing without an outer stainless steel tube and then zone melted is given in Figure 5. Figure 6 shows a specimen which was hot drawn with an outer stainless steel tube and then zone melted with this tube intact. An intermetallic compound was formed because of the reaction between the stainless steel and the aluminum. This compound is seen throughout the structure of Figure 6. The composites which were hot drawn with the welding flux, retained the flux in the structure. The specimens were usually not 100% dense. Figure 7 is a microstructure showing these features (the flux can be seen around the tubes).



A. COMPLETE CROSS SECTION, 100X

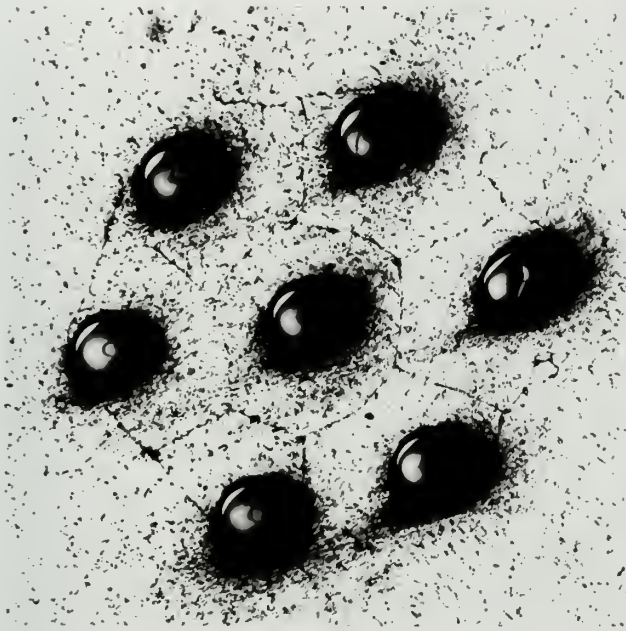


B. END VIEW OF FIELD IN MATRIX, 500X



C. BOUNDARIES OF ALUMINUM TUBING, 500X

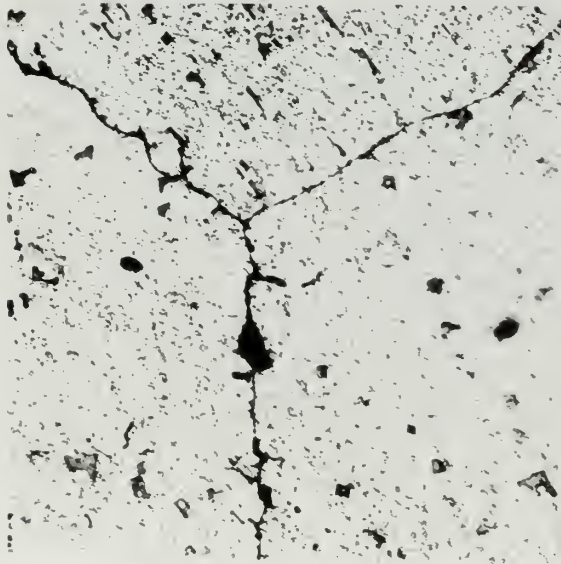
FIGURE 4. MICROSTRUCTURE OF A HOT DRAWN SPECIMEN WITH OUTFLOW STAINLESS STEEL TUBE



A. COMPLETE CROSS SECTION, SOCK



B. END VIEW OF FIBER IN SOCK

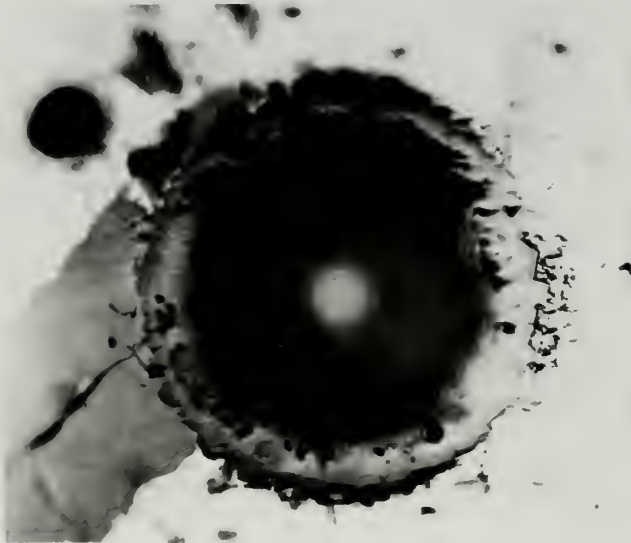


C. MICROSTRUCTURE OF ZONE TREATED FOR HEAVY SPINNING

FIGURE 3. MICROSTRUCTURE OF A ZONE TREATED FOR HEAVY SPINNING

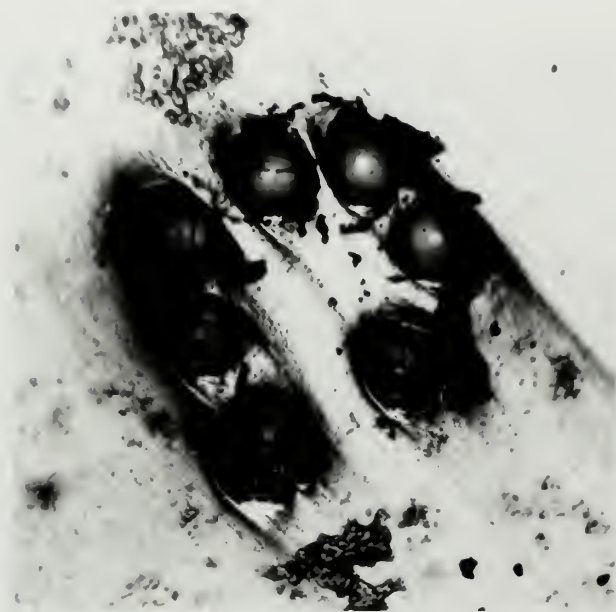


A. COMPLETE CROSS SECTION, 100X

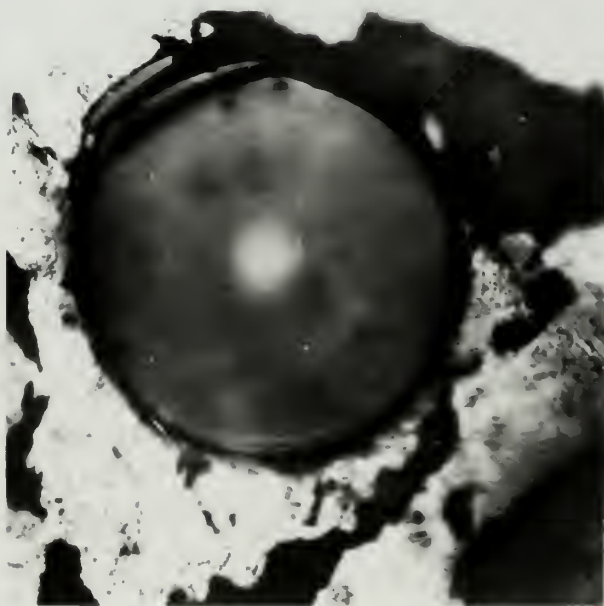


B. END VIEW OF FIBER, INTERMETALLIC, 500X

FIGURE 6. MIC. STRUCTURE OF A ZONE WELDED NOT DRAWN SPECIMEN WITH OUTER STAINLESS STEEL TUBE



A. COMPLETE CROSS SECTION, 10X



B. END VIEW, WITH TINT

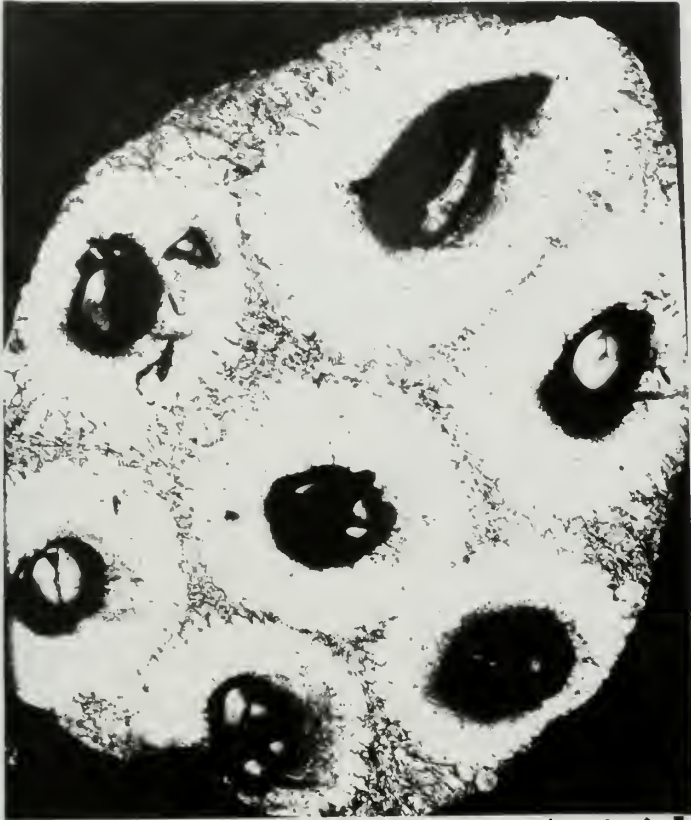
FIGURE 7. MICROSCOPIC OF A HOT DRAWN SPANDEX WITH TINT

3. Dip Brazing Followed by Hot Drawing.

The specimens prepared by dip brazing the bundle of aluminum tubes and then hot drawing with an outer stainless steel tube were 100% dense. The brazing of the tubes was not perfect. There were flaws where the specimen had rested against the steel basket used during dip brazing and only an estimated 90% of the tube surfaces were joined. Figure 8 shows a section after dip brazing. The out of roundness (Figure 8A) was caused by mishandling of the piece and did not exist in the actual piece as brazed. The microstructure of Figure 8A shows the aluminum tubes welded together with the eutectic welding alloy. The weld is shown at 500X in Figure 8B. Figures 9 and 10 are the microstructure of the brazed piece after it was hot drawn using an outer stainless steel tube. The eutectic brazing material has dispersed more than was shown in the previous figure. The composite formed was 100% dense and the tubes appeared to be welded together.

B. Test Results

Tensile tests on composites prepared by the basic hot drawing process and by zone melting showed only a minimal increase in strength (see Table IV). Figure 11 is the fracture surface of a tested hot drawn specimen. The individual aluminum tubes protrude above the fractured surface. Figure 12 presents the engineering stress-strain curves of these specimens. Elongation was measured by movement of the Instron cross-head. The composites prepared by the dip brazing followed by hot drawing process, gave significant increase in tensile



1. 37-111-31000
SECTION, 100X



37-111-31000
SECTION, 100X

37-111-31000 SECTION, 100X

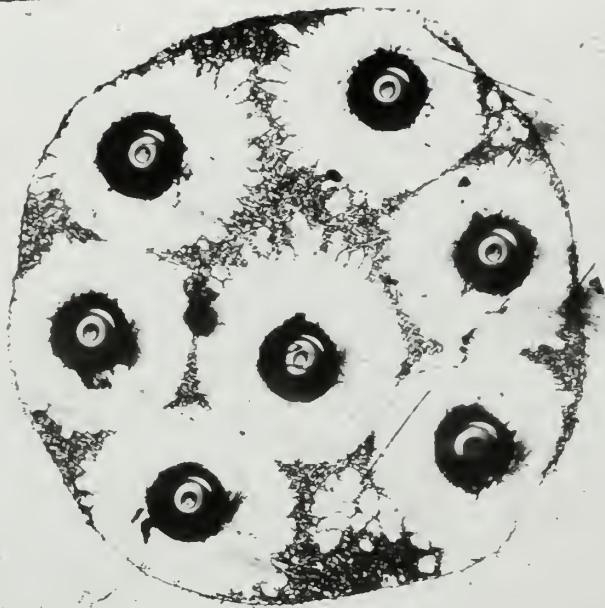


Fig. 1. *Clusia rosea* (Sw.) DC., 1907

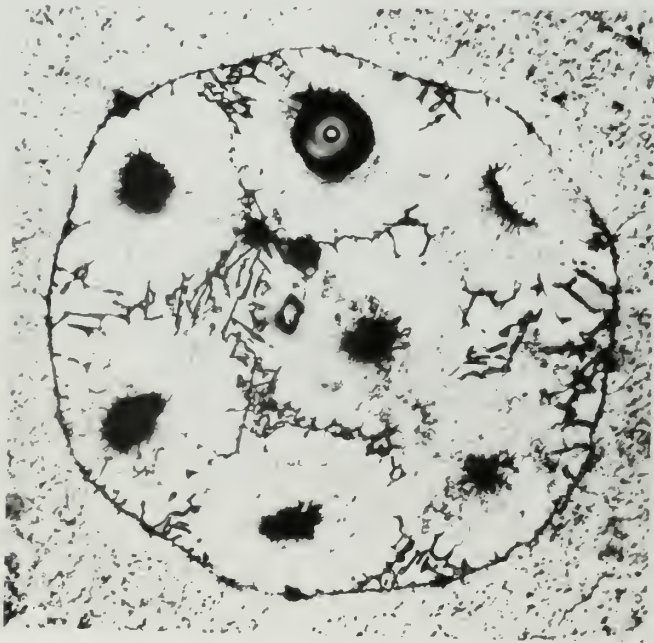
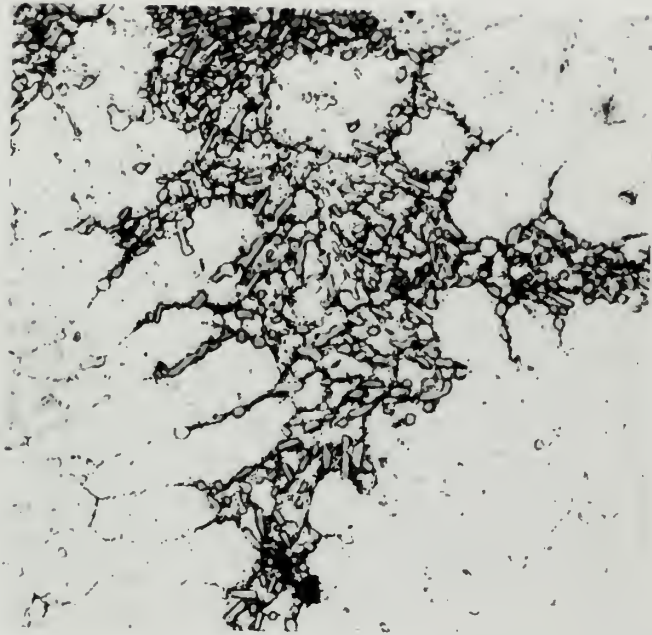


Fig. 2. *Clusia rosea* (Sw.) DC., 1907

Fig. 3. *Clusia rosea* (Sw.) DC., 1907



1. EUTECTIC, WELDING ALUMINUM TUBES TOGETHER, 500X



2. END VIEW OF PIPE IN MATRIX, 500X

FIGURE 10. MICROSTRUCTURE OF DIF BEARED AND DRAWN SPECIMEN

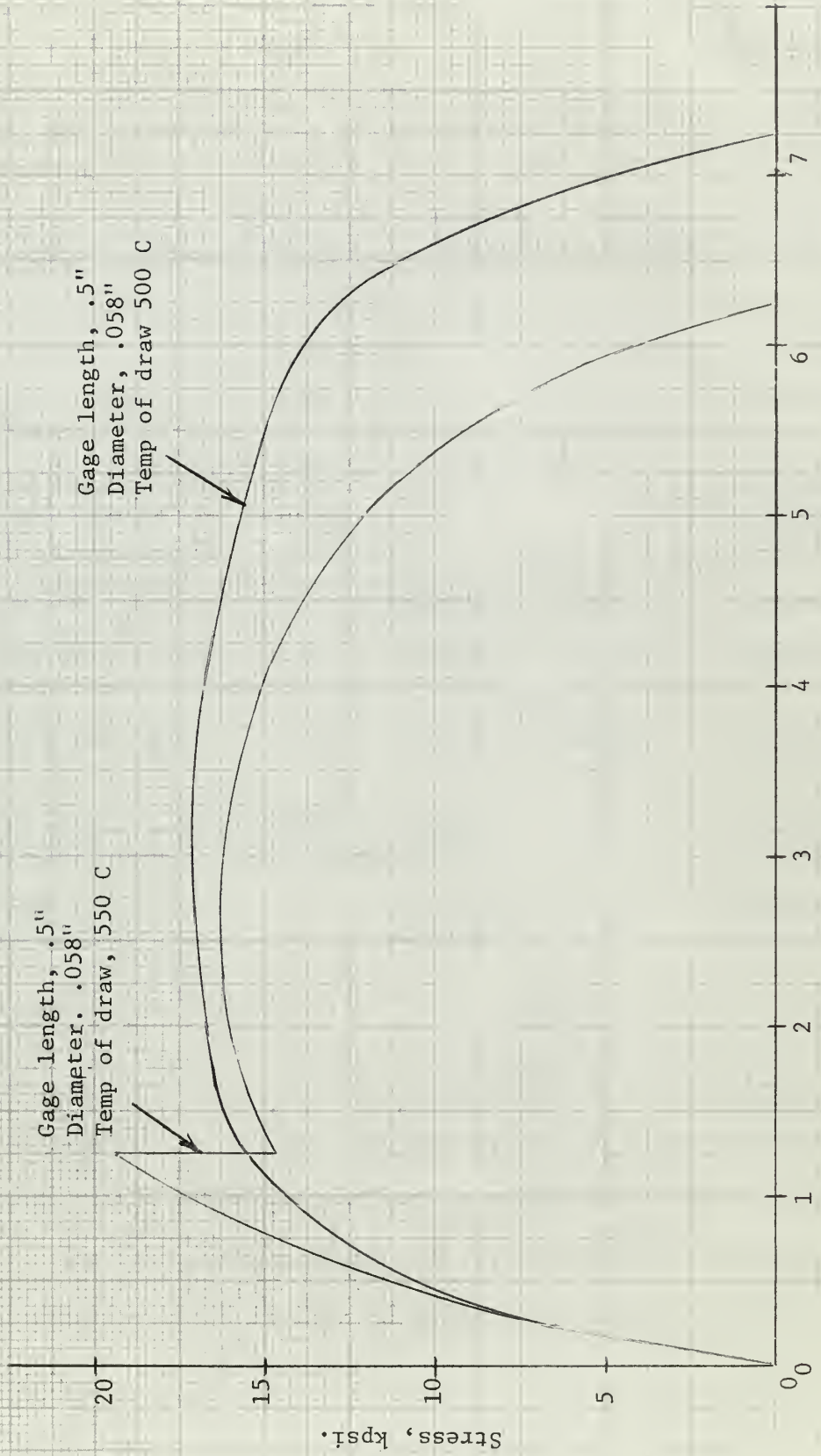


A. ALUMINUM TUBES NOT BONDED , VIEW A



B. ALUMINUM TUBES NOT BONDED , VIEW B

FIGURE 13. GRAFITE D'IMPACT ET DE BRAS SPECTRE



Gage length, .5"
Diameter, .058"
Temp of draw, 550 C

Gage length, .5"
Diameter, .058"
Temp of draw 500 C

FIGURE 12 Stress-Strain Curve for Hot Drawn Specimens

TABLE IV

Tensile Data on Selected Composite Test Specimens
(All Hot Drawn with Outer Stainless Steel Tube)

Specimen	Preparation	Boron Vol. Frac., %	Tensile Strength	Predict Strength	Calc. Stress on Fibers
2S	As drawn	3.81	17,000 psi	27,800 psi	42,000 psi
7S	With welding flux	3.81	19,600	27,800	110,000
8S	Zone melt., with weld. flux	3.72	23,000	27,500	Intermetallic formed properties
11S	Zone melt.	6.7	20,600	36,900	Unknown
12S	Zone melt., with weld. flux	6.7	19,300	36,900	Unknown
15S	Dip brazed and hot drawn	7.95	26,000	40,400	142,000
16S	Dip brazed and hot drawn	7.95	23,400	40,400	111,000
17S	Dip brazed and hot drawn	9.9	32,200	46,800	180,000

strength. Figure 13 presents the stress-strain curves of two specimens prepared in this manner. The fracture surface of some of these specimens revealed that some of the fibers had pulled out of the matrix at fracture (see Figure 14). The fiber extends above the fracture surface. Data on tests of these composites is summarized in Table IV.

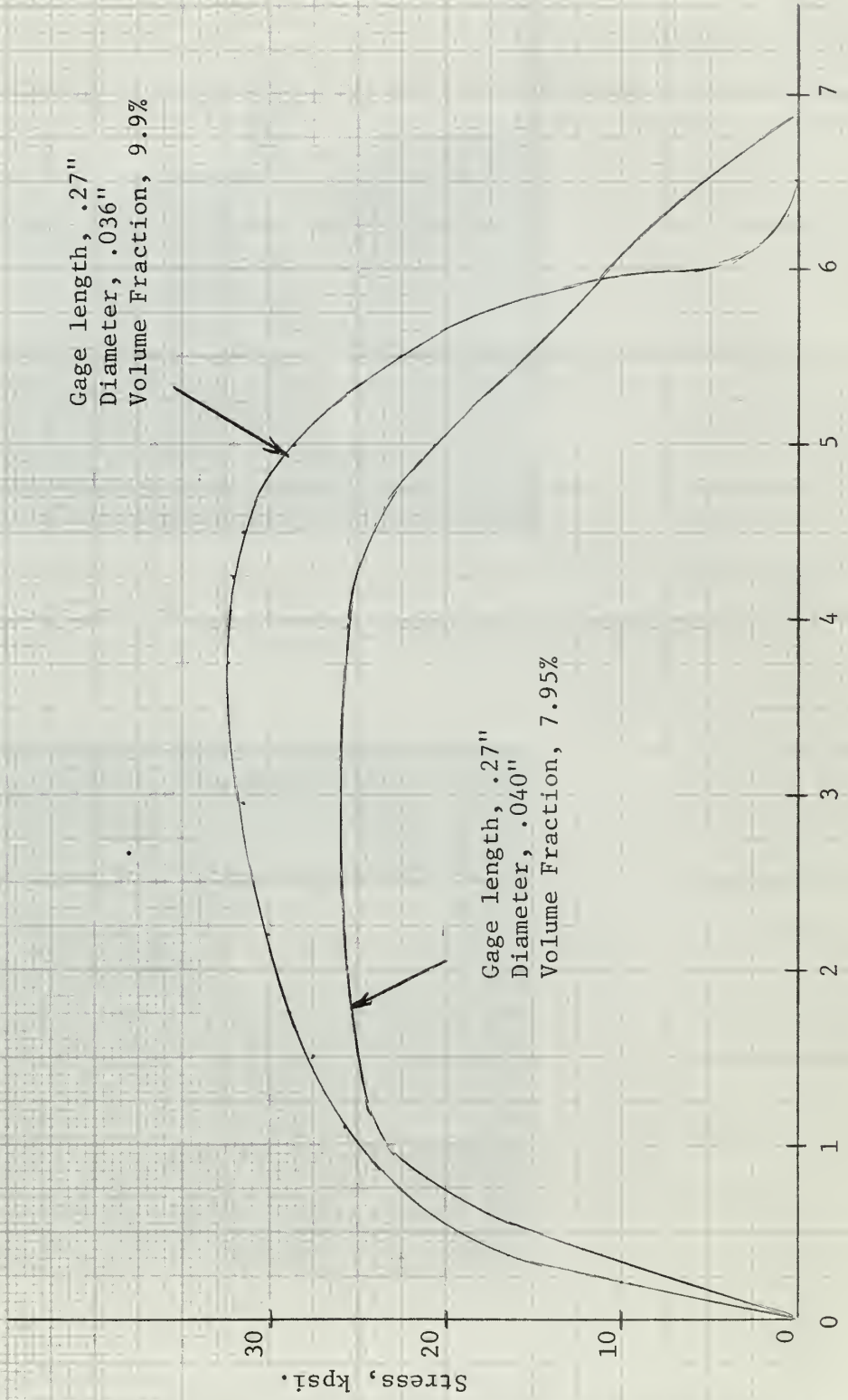
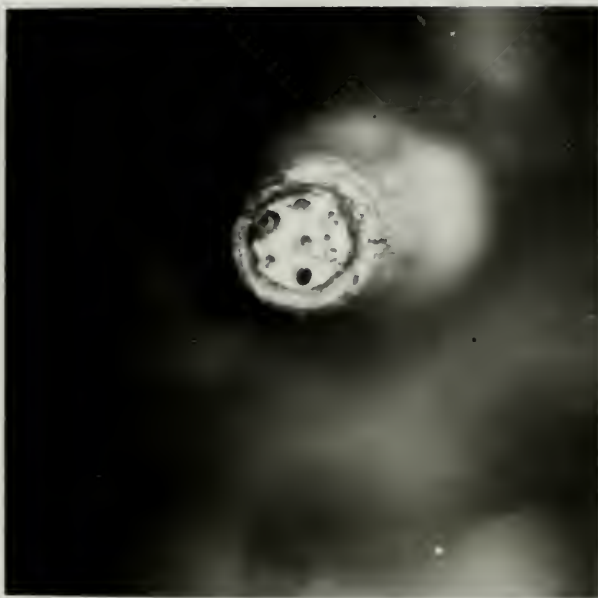


FIGURE 13 Stress-Strain Curves for Dip Brazed Hot Drawn Specimen



A. FIBER EXTENDING ABOVE FRACTURE SURFACE



B. CROSS SECTION VIEW OF A FIBER

FIGURE 14. FRACTURE SURFACE OF 100 DENIER 60/40 JIR® SPECIMEN

One third of the twelve fibers tested individually, failed at a stress of less than 70,000 psi. The remaining failed at stresses between 250,000 psi and 409,000 psi, with the average being 327,500 psi. Typical stress-strain curves which show variations from test to test are given in Figure 15. The elongation calculation is based on movement of the Instron head.

Stress-strain curves for the four 3003 aluminum alloy test specimens are presented in Figure 16. An extensometer was used on these specimens to measure elongation.

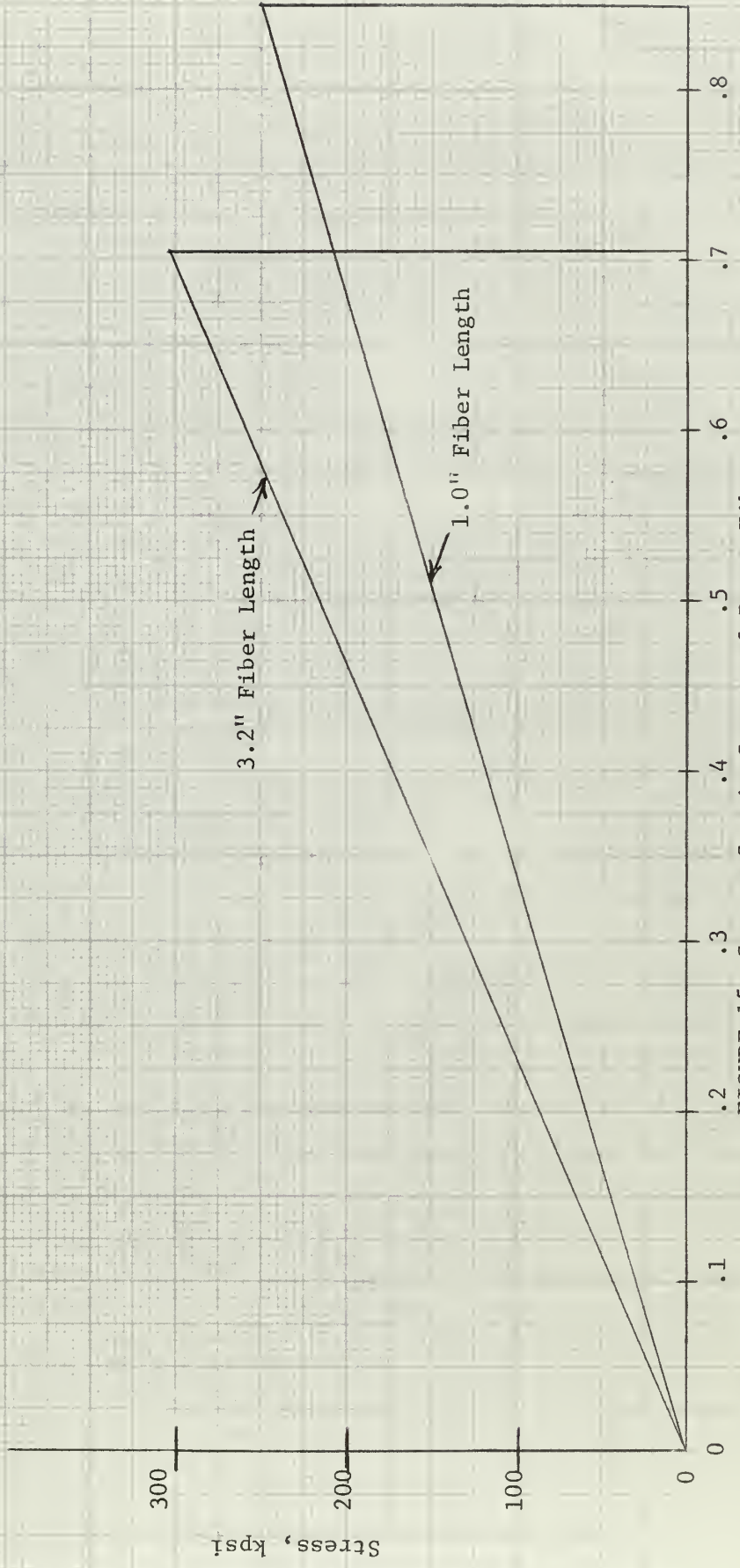


FIGURE 15 Stress-Strain Curves of Boron Fibers

- A, Longitudinal Direction, Furnace Treated, .147"
- B, Transverse Direction, Furnace Treated, .147"
- C, Longitudinal Direction, Hot Drawn, .096"
- D, Transverse Direction, Hot Drawn, .096"

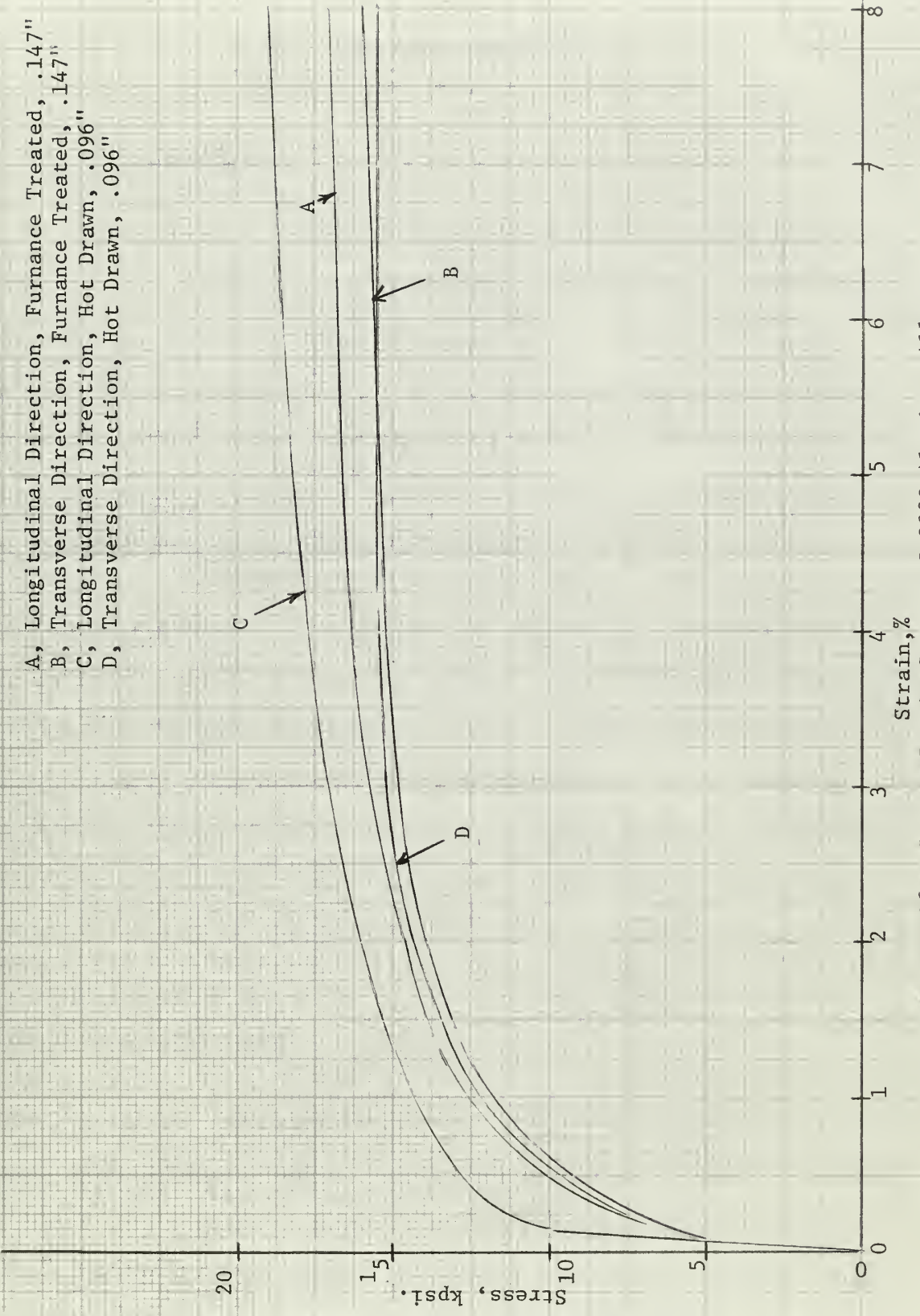


FIGURE 16. Stress-Strain Curves of 3003 Aluminum Alloy

IV. DISCUSSION OF RESULTS

A. Processing Techniques

The evaluation and interpretation of the three processing techniques is based on the following desired specimen characteristics. High density is required to provide meaningful results and is required for most practical material applications. All materials making up the composite must bond together. Fiber breakage is to be kept at a minimum during the preparation phase. A symmetric geometric arrangement of fibers with equal spacing between the fibers is desired.

The cold drawing process, with or without an outer stainless steel tube, is not a feasible process to produce boron fiber-aluminum composites. Other studies currently in progress at M.I.T., Cambridge, Massachusetts indicate that more ductile fibers can be incorporated in aluminum by this process without breaking the fibers. However, even in that situation, the aluminum oxide layer on the tube surfaces prevents bonding of the tubes. Cold drawn specimens which are not dense can be zone melted to achieve 100% density; however, the oxide layer will still prevent bonding between the tubes.

The hot drawing technique resulted in 100% dense specimens with only minimal fiber breakage, and in some cases, no fiber breakage. In order to achieve this result, an outer tube of a material such as stainless steel is required. This outer tube absorbs most of the tensile stresses during drawing, thereby preventing the stresses from acting on the fibers

and breaking them. At the same time, the specimen is squeezed as it passes through the die and densification occurs. A drawing temperature of about 550°C gave the best results. The aluminum is relatively soft at this temperature. Only a slight increase in tensile strength was obtained by hot drawn specimens because the aluminum tubes did not bond together. The introduction of a welding flux was unsatisfactory as the flux was retained in the specimen rather than being forced out by the squeezing action as was desired. Apparently this did join the tubes at certain points because the tensile strength did increase slightly. It was not determined if the fibers themselves were bonded to the aluminum because the aluminum tubes pulled apart. Zone melting of hot drawn specimens with only the aluminum outer tube gave 100% density. Satisfactory control of the zone cannot be obtained by the electron beam apparatus, however. Further, the formation of an intermetallic compound when a stainless steel outer tube was used rendered this procedure inadequate. During zone melting the aluminum tubes changed shape but the oxide layer remained intact and there was no significant bonding between the tubes.

The process of dip brazing followed by hot drawing is a successful technique. The desired geometry and density were obtained with little fiber breakage and it is indicated that refinement of the process would result in no fiber breakage. The specimen fracture surfaces showed that there was some fiber pullout at failure. Heat treatment after preparation or a preliminary coating on the fibers which would react with the aluminum might prevent this. This author working on other

research determined that boron fibers could be coated with such materials as cerrobend (a eutectic alloy of lead, bismuth, cadmium, and tin), in an air atmosphere (AVCO Corp. Disclosure of Invention dated February 24, 1966). The volume fraction of composites prepared by this process can be varied by using aluminum tubes of varying wall thicknesses. An advantage of the drawing process over other methods, is that the boron isn't exposed to molten aluminum. Thus, excessive reaction between the two materials does not occur.

Another method has been suggested by the studies conducted here. The inner aluminum tubes would be coated by an aluminum brazing paste and the specimen then hot drawn as before. A brazing paste has recently been developed by Handy and Harmond Inc., which contains the flux.

B. Test Results

The stress-strain curves in Figures 13 and 14 show the increased tensile strength of the prepared composites. The strain calculations are based on head travel of the Instron testing machine. The specimens were only two inches in length and prohibited the use of the available extensometer. More accurate strain data is required but could not be obtained in the time limitations for this study.

The specimen having a tensile strength of 32,200 psi (see Figure 13) had a 9.9% boron volume fraction. The corresponding value predicted by the volume-fraction rule is 46,800 psi.

$$\sigma_c = \sigma_f V_f + \sigma_m' V_m$$

$$\sigma_c = .099 (327,500) + .901 (1600) = 46,800 \text{ psi}$$

σ_m' = tensile strength of aluminum alloy 3003 at the elongation of the composite corresponding to the tensile strength of 32,200 psi.

The difference between the actual and predicted tensile strength is believed to be caused by: (1) the non-perfect tube brazing, and (2) lack of complete bonding between the boron and aluminum.

V. CONCLUSIONS

1. A boron fiber-aluminum composite of a high strength can be produced by dip brazing a bundle of aluminum tubes containing the fibers and hot drawing the bundle in a stainless steel tube through consecutive dies, at about 550°C.
2. Cold drawing of a bundle of aluminum tubes containing boron fibers through consecutive dies to form a composite is not feasible. The stresses on the fibers exceed their ultimate strength and break considerably before 100% density is attained.
3. Hot drawing a bundle of aluminum tubes containing boron fibers through consecutive dies at about 550°C, results in a 100% dense specimen with minimum fiber breakage. The specimen must be contained in a stainless steel or similar tube.
4. The aluminum oxide layer on the surface of the tubes prevents the aluminum tubes from bonding together significantly in any drawing or zone melting process.
5. In the hot drawing process, little or no chemical reaction occurs between the boron and the aluminum.

VI. RECOMMENDATIONS

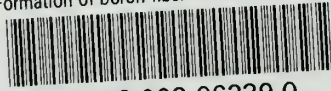
1. The dip brazing procedure should be investigated using a nineteen or larger bundle of aluminum tubes containing boron fibers. Volume fraction could be varied by using aluminum tubes of various wall thicknesses.
2. The feasibility and effect of pre-coating the boron fibers with some material which would form a strong bond between the boron and aluminum should continue to be investigated.
3. A drawing procedure, using a brazing paste containing the brazing material and the flux combined, should be explored. It is suggested that the inner tubes be coated with such a brazing paste and a bundle of tubes be hot drawn as previously described.

BIBLIOGRAPHY

1. McDanel, D. L., Jech, R. W., and Weeton, J. W., Stress-Strain Behavior of Tungsten-Fiber-Reinforced Copper Composites, 1963.
2. Cratchley, D., "Factors Affecting the UTS of a Metal/Metal Fibre Reinforced System", Powder Metallurgy, No. 11, 1963.
3. Piehler, H. R., "Plastic Deformation and Failure of Silver-Steel Filamentary Composites", Transactions of the Metallurgical Society of AIME, Vol. 233, Jan. 1965.
4. Forsyth, P. J. E., Ryder, R. A., and George, R. W., Applied Materials Research, 1964.
5. Petrasek, D. W., and Weeton, J. W., "Alloying Effects on W Fiber Reinforced Cu-Alloy or High Temperature Alloy Matrix Composites", NASA TN D-1568, 1963.
6. Jech, R. W., McDanel, D. L., and Weeton, J. W., Fiber Reinforced Metallic Composites, 1959.
7. Williams, R. V., and O'Brien, D. J., "The Reinforcement of Metals with Metal Fibers", Applied Materials Research, July, 1964.
8. Beskey, R. H., "Fiber Reinforcement of Metallic and Non-Metallic Composites", Clevite Corp., Report to AFSC, Aeronautical Systems Div., ASD Final Report TDR-63-619, 1963.
9. Koppenaar, T. J., and Parikh, N. M., Fiber Reinforced Metallic Composites, 1959.

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