

General Disclaimer

One or more of the Following Statements may affect this Document

- This document has been reproduced from the best copy furnished by the organizational source. It is being released in the interest of making available as much information as possible.
- This document may contain data, which exceeds the sheet parameters. It was furnished in this condition by the organizational source and is the best copy available.
- This document may contain tone-on-tone or color graphs, charts and/or pictures, which have been reproduced in black and white.
- This document is paginated as submitted by the original source.
- Portions of this document are not fully legible due to the historical nature of some of the material. However, it is the best reproduction available from the original submission.

ELEVENTH QUARTERLY PROGRESS REPORT

DEVELOPMENT OF A METHOD FOR FABRICATING
METALLIC MATRIX COMPOSITE SHAPES BY A
CONTINUOUS MECHANICAL PROCESS

Submitted to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA
Contract NAS8-27010

Technical Program Director: Mr. Felix Lalacona, S&E-ASTN-MM

Prepared by

A. P. DIVECHA

Covering Period From
October 15th, 1973
March 15, 1974

COMMONWEALTH SCIENTIFIC CORPORATION
500 Pendleton Street
Alexandria, Virginia



**COMMONWEALTH SCIENTIFIC
CORPORATION**

(NASA-CR-120248) DEVELOPMENT OF A METHOD
FOR FABRICATING METALLIC MATRIX COMPOSITE
SHAPES BY A CONTINUOUS MECHANICAL PROCESS
QUARTERLY (Commonwealth Scientific Corp.)
46 p HC \$5.50

CSC 13H

G3/15

Unclass
16457

N74-29784

ELEVENTH QUARTERLY PROGRESS REPORT

DEVELOPMENT OF A METHOD FOR FABRICATING
METALLIC MATRIX COMPOSITE SHAPES BY A
CONTINUOUS MECHANICAL PROCESS

Submitted to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA
Contract NAS8-27010

Technical Program Director: Mr. Felix Lalacona, S&E-ASTN-MM

Prepared by

A. P. DIVECHA

Covering Period From
October 15th, 1973
March 15, 1974

COMMONWEALTH SCIENTIFIC CORPORATION
500 Pendleton Street
Alexandria, Virginia



FOREWORD

This Quarterly Progress Report covers the work performed under contract NAS8-27010 from October 15, 1973 to March 15, 1974. The contract is being performed under the technical direction of Mr. Felix LaLacona of National Aeronautics and Space Administration, George C. Marshall Space Flight Center, Huntsville, Alabama.



ABSTRACT

2" (.508m) Al/B tube fabrication attempts continued. Despite relatively superior temperature control, the consolidation characteristics did not improve as expected. Inadequate binder removal was positively identified as the cause largely responsible because the graphite residue generated during heating acted as a diffusion barrier. A new method to align the filaments without mat making was employed. Flat strips or modules were prepared by drawing aluminum clad boron ends encased in an aluminum jacket. The gradual reduction in area and consequent gentle squeezing aligned the binder free matrix-clad-filament (MCF) ends well. The MCF count and die sequence are described here in detail.

Aluminum-graphite (Al/c) composite fabrication received major emphasis. Approximately 150 infiltration experiments were conducted to examine fabricability of wires, rods and tubes via the electron beam. Concurrently the electron beam apparatus was gradually improved by incorporation of motors for traversing of Al/c preforms more reliably, higher voltage feed-thrus, ceramic stands for preform support, etc. It was demonstrated that electron beam heating is capable of forming aluminum-graphite composites. In particular, there is convincing



evidence that tubular composites are easier to fabricate than other shapes such as rods and wires. This is largely due to the core support in the tubular preform and its symmetry. These experiments are described in detail along with metallographic evidence of infiltration



TABLE OF CONTENTS

		<u>PAGE</u>
	ABSTRACT	
1.0	INTRODUCTION	1
2.0	Aluminum-Boron (Al/B) Composites	5
	2.1 2" (0.0508m) Diameter Tube Fabrication	5
	2.2 Modular Preform Preparation	8
2.2.1	Tubular Preforms	13
3.0	Aluminum-graphite (Al/c) Composites	15
3.1	Wire, Rod and Tube Fabrication Experiments	20
4.0	Future Work	35



LIST OF ILLUSTRATIONS

		<u>PAGE</u>
Figure 1	Modular Preform Preparation Via Drawing, A Schematic. See Text for details.	9
Figure 2	Electron Beam (EB) Apparatus for Fabrication of Al/C Composites, A Schematic.	16
Figure 3	An actual photo of the apparatus in Figure 2 with ceramic stand and a hollow quartz cone for positioning the preform at the electron beam emitter center. The tungsten filament loop is barely visible from this angle.	17
Figure 4	Schematic of the Electron Beam Apparatus with vertically moving preforms. Larger diameter and longer preforms will be fabricated in this system expected to be operational by May, 1974. The bell jar is omitted in this illustration for clarity.	19
Figure 5	A typical example of multiple matrix clad Thornel yarns to separate even though each yarn is well consolidated. As polished, Dark Field, 80x.	22
Figure 6	Large matrix excess in the form of cladding is advantageous in joining the infiltrated yarns. However, each yarn maintains its identity as shown in this micrograph. Etched (HF 1%), Bright Field, 200x.	24
Figure 7	Portion of an aluminum-12% Si matrix infiltrated yarn at higher magnification shows good filament distribution and absence of carbides. Etched (1% HF), Bright Field, 800 x.	25



LIST OF ILLUSTRATIONS

		<u>PAGE</u>
Figure 8	Irregularly shaped composite containing seven Thornel 50 yarns. As polished, Bright Field, 80x.	26
Figure 9	Electron beam apparatus showing the Ti-B ₂ -TiN rolls and preform in position prior to evacuation.	28
Figure 10	Basic arrangement of materials including filament and the matrix for tubular composites. A schematic.	30
Figure 11	Micrograph of a portion of a tube showing partial infiltration. The black areas show clusters of filaments not penetrated by the matrix. In well-infiltrated areas, the filament distribution is fairly satisfactory. As polished, Dark Field, 80x.	31



LIST OF TABLES

	<u>PAGE</u>
Die Sequence for 2" Al/B tube fabrication	7



1.0 INTRODUCTION

The object of this program is to develop processes capable of producing composites in structural shapes and sizes suitable for space applications. The processes to be explored must be continuous and promise to lower composite fabrication costs significantly. The composite system of prime interest is aluminum boron (Al/B).

Sound metal matrix composites have been produced by numerous procedures such as hot pressing, electro and chemical vapor deposition, plasma spraying, high energy rate forming, powder metallurgy and others. In each technique, careful handling and placement of the filaments precisely is mandatory to achieve uniform interfiber spacing in the finished product. Filament-filament contacts despite extensive care and sophistication, nevertheless occur and the composite integrity suffers.

The present program is based on the ability to mechanically clad the boron filament continuously and uniformly with the matrix. The cladding process requires only periodic checks and up to 5ft/min (1.53m) matrix clad filament (MCF) can be produced in one apparatus. Since the cladding thickness can be varied over a wide range, the filament volume concentration can be precisely controlled. Practically any mono or



or multi-filament species can be clad effectively. The process is particularly suited to boron filaments.

The program is divided into three phases. In the first phase, promising metal working processes, such as rolling, drawing and extrusion are to be explored. Once the processing parameters are established for simple and miniature shapes, the information generated is to be applied in the second phase to produce at least two structural shapes with particular emphasis on hot and tube sections. The third phase to be conducted concurrently with the second, is concerned with detailed evaluation of the full-scale composites.

Another system, namely, aluminum-graphite (Al/C) composites, was added to the current Al/B investigations. The primary objectives of this work are nearly identical to that of its Al/B counterpart. That is, the program is concerned with development of one or more continuous processes capable of producing tubular and other (hats, Z's, T's, etc.) shapes. Most important, the experience gained from Al/B investigations is to be incorporated and utilized to the fullest extent.

During this reporting period, the Al/B tube fabrication attempts continued. More elaborate binder removal methods and longer flushing time in solvents were not sufficient. Despite superior temperature control, the tubular preforms failed to consolidate satisfactorily.



a new approach which precluded mat making was examined. The matrix-clad-filaments (MCF) contained in a metal tube were drawn through round and rectangular dies to form flat strips. The drawing forces were found to be adequate in aligning the MCF ends without significant filament damage. Tubular preform perforation thus requires minimal time as compared to the stacking of binder held mats and their placement on to the steel core. The new procedures, described here in detail, are expected to alleviate consolidation problems encountered in large tubes (and possibly hat sections.)

Aluminum-graphite composite fabrication via electron beam has been given considerable emphasis. Approximately a total of 150 experiments have been performed in attempts to produce single infiltrated yarns, multiple yarns, and small tubes. Concurrent efforts have also been directed at increasing the versatility of the electron beam apparatus by incorporating better and higher voltage feed-thrus, motors for automatic traversing of the preforms in the electron beam, and enlarging the system capacity to enable production of larger composites.

Infiltration experiments conducted thus far show that as the aluminum melts in the electron beam, it also evaporates at a very rapid rate. The electron wattage, aluminum concentration, physical contact of the aluminum with the nickel coated filaments during aluminum melting



must be controlled accurately to achieve uniform consolidation.

Fortunately, tubular preforms exhibit superior consolidation to rod shaped composites because of the (glass) core support. The experiments are described in detail.



2.0 ALUMINUM-BORON (Al/B) COMPOSITES

The previous reports have described the fabrication procedure in considerable detail. This reporting period was largely expended at determining the causes for lack of uniform and complete consolidation. Some of these causes had been suspected and actions were initiated to correct them. Of particular significance was the fact that while thermal uniformity is important, the filament or MCF mat cleanliness is perhaps more critical. Near complete removal of the binder must be performed before adequate consolidation occurs. This aspect coupled with the difficulty in preparing wide (6", .1524m) mats prompted a new approach which would preclude the use of any binder. These are described in detail below.

2.1 2" (.0508m) DIAMETER TUBE FABRICATION

Four 3 feet (.9144m) long, preforms comprising of seven ply were prepared. The mats were obtained by using the level winder constructed during the past quarter. As usual, the preforms were washed in a solvent (toluene or dichloroethane) circulating pump. To assure adequate binder removal, the preform was washed in fresh solvent three times for two hours each.



After cold drawing according to a die sequence described in Table I, the preforms were hot drawn through two dies at 1000°F (538°C) at 13 ft/min (3.9120m/min). (The profile showed less than 75°F (23°C) variation on a dummy preform drawn at this rate).

Small sections of these tubes appeared well consolidated. However, when examined metallographically, the consolidation was not significantly improved when compared to similar small diameter tubular composites prepared earlier.*

Selected portions of these tubes were subjected to further examination to detect the cause for inadequate consolidation. Tube sections were secured in a vise and deliberately fractured. It was revealed that black (graphite) residue most likely to have been generated by pyrolysis of the binder must be responsible. It was clear now that even prolonged and repeated solvent flushing was not sufficient to remove the binder adequately. Furthermore, the very nature of the cladding was also likely to prevent complete removal because the binder trapped between the aluminum and the boron may not be easily reached by the solvent.

*4th. and 5th. Quarterly Reports, this contract



Table I

Die Sequence for 2" Al/B tube Fabrication

Die #	Die Diameter	# of Passes	Temp. °C
1.	2.240" (0.0568 m)	2	R. T.
2.	2.210" (0.0562 m)	2	R. T.
3.	2.190 (0.0556 m)	1	R. T.
4.	2.185 (0.0555 m)	2	R. T.
5. *	2.185" (0.0555 m)	1-3	R. T. and 538
6. *	2.175" (0.0552 m)	1-3	R. T. and 538

* Passage through these dies up to 3 times at 538° was attempted on short sections.



If, however, mat making or filament alignment were possible without the polymeric binder, the consolidation would be assuredly achieved. Considerable effort was, therefore, directed towards development of alternate methods. One method involving drawing of small sections was devised and evaluated as described below.

2.2 MODULAR PREFORM PREPARATION

It has been demonstrated that the matrix-clad-filament bundles when loosely inserted into a steel or aluminum tube align nearly perfectly after passage through round dies. If this round preform could be further drawn through rectangular dies without damaging the filaments or perturbing filament alignment, the consolidation characteristics would markedly improve. Unlike mat making which involves the unavoidable use of a polymeric binder, the Al/B clad material would be totally free from any contaminants at all stages of the preform preparation. The success of this approach would make tubular as well as flat (or hat) preform preparation easier, faster and more reliable. The preform cross-section would appear as shown in Figure 1* schematically.

* See Section 2.2.1 for details of preform preparation



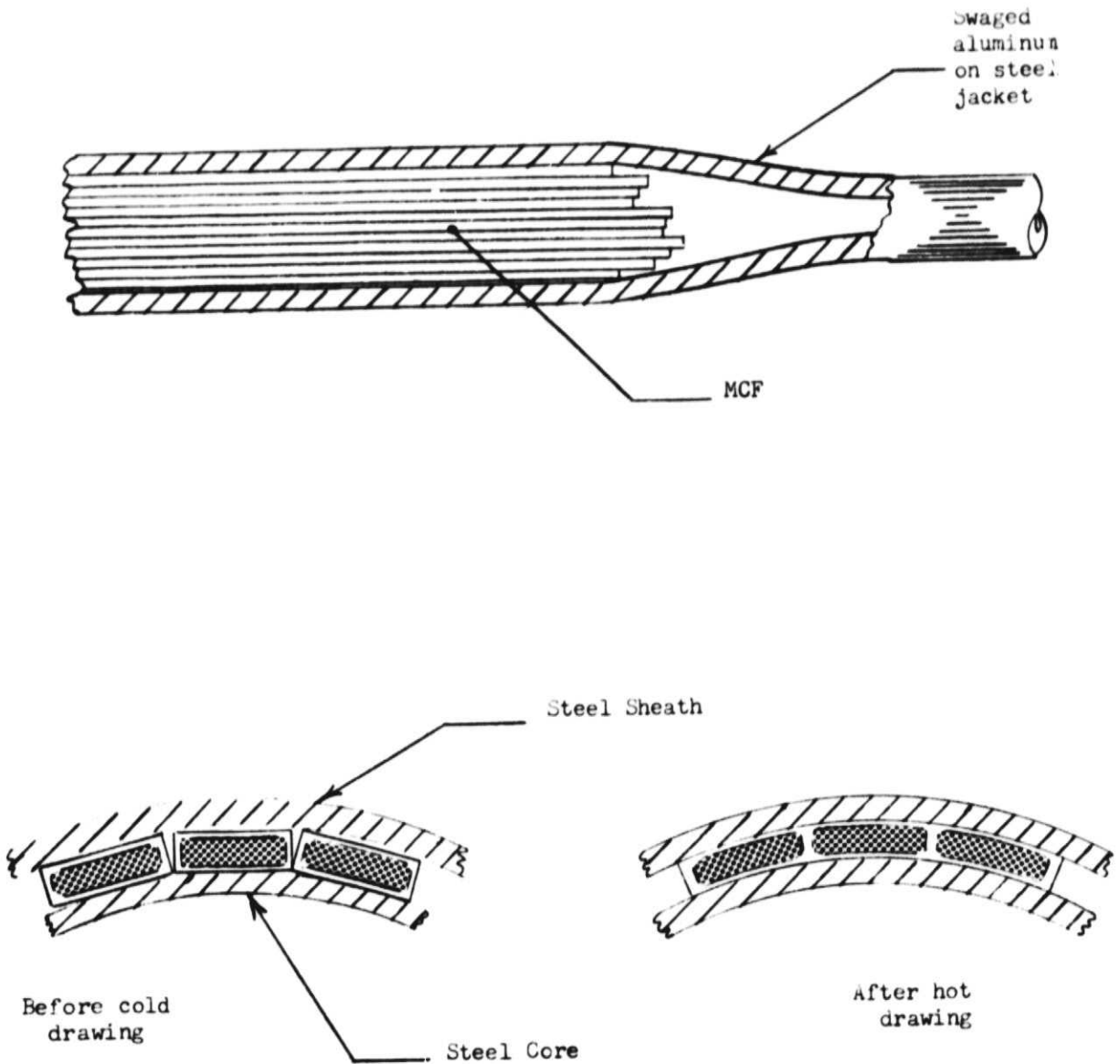


Figure 1. Modular Preform Preparation Via Drawing, A Schematic. See text for details.

It was recognized that this approach will require preliminary experimentation to evaluate its applicability to Al/B preform. Accordingly, the initial experiments were conducted with a small size module. That is, while it is possible to purchase rectangular carbide dies with dimensions as large as 2" (.0508m) wide by $\frac{1}{4}$ " (6.35×10^{-3} m) thick orifices, it was necessary to begin with a smaller rectangular module. The jacket (mild) steel material dimensions of 0.375" (9.52×10^{-3} m) O.D., and 0.035" (8.89×10^{-4} m) wall were chosen. These tubes were swaged at one end as usual to permit insertion into the dies and securing in the jaws for drawing.

Exactly 800 ends of the matrix-clad-filaments were counted and the bundle was wrapped longitudinally in a 1 mil (2.54×10^{-5} m) 6061 Al foil. These MCF ends were chemically cleaned to remove surface particulate matter, oils, grease, etc. The cold drawing sequence for this type of preform involved passage through 0.370", 0.360", 0.350" (9.398×10^{-3} m, 9.144×10^{-3} m, 8.89×10^{-3} m, respectively) to a rectangle via $0.422" \times 0.187"$ (10.719×10^{-3} m, 4.750×10^{-3} m) and/or $0.437" \times 0.156"$ (4.100×10^{-3} m $\times 2.982 \times 10^{-3}$ m) respectively. Filament alignment and filament fracture were examined at each step by treatment of a short (2", .0508m) section in HNO_3 to remove the steel jacket completely. Maximum number of broken filaments never exceeded three or under 0.004% of the total ends in the preform.



It was hoped that steel jacket removal (mechanically or chemically) would not introduce objectionable impurities in the preform or disrupt the filament alignment. Unfortunately, it was found that the steel jacket removal in HNO_3 did contaminate the preform and grinding the jacket also caused physical damage to the filament. Use of thicker aluminum cover foil on the bundle did not help in maintaining the preform integrity. Hot drawing was found to consolidate the preforms so completely that subsequent preform preparation and cold drawing would be rendered difficult.

Greatest success was achieved when an aluminum tube identical in dimensions to those of the steel was utilized. That is, $3/8"$ ($9.52 \times 10^{-3}\text{in}$) O.D., $0.035"$ ($8.89 \times 10^{-4}\text{m}$) wall, 6061 T6 aluminum tubes, were filled with 800, 700, 650 and 640 MCF ends respectively, and cold drawn through the same round and rectangle dies as before. The best MCF count was found to be 640 ends. Although considerable excess aluminum exists on the periphery, the preparation of these modules is rapid. It is possible to prepare 16 modules within 1 hour given that the cleaned and dried MCF bundles with 640 ends are available. Fortunately, the counting can be performed automatically as the filament is clad or alternately, a simple rewinder may be set up to do the counting as necessary from spools after the filament has been clad.



The large excess of aluminum on each strip or module may be removed by three methods. The simplest one is to utilize a thin wall aluminum tubing that is largely offset by the likelihood of the tube fracture during drawing and higher cost of the tubing. Etching in NaOH or HCl solution is rapid and as much as 0.030" ($7.62 \times 10^{-4}m$) can be etched fairly uniformly in 15 minutes. Since commercial grade NaOH is very cheap (\$16.00/100lbs., \$.35/kg), etching appears to be the most economical. The third method is to remove the aluminum on the edges by milling operation. Since the flat strips are quite uniform and straight, it may be possible to mill many strips simultaneously, leaving as little as 0.001" thick aluminum excess.

The main objective at the present is to examine the fabricability of tubes and hats via this modular preform approach. It, therefore, appears expedient to proceed with these modules even though relatively high aluminum concentration may exist in the composite. Concurrent efforts will continue to examine etching and milling to remove excess peripheral aluminum as described earlier. Carbide die manufacturers are being consulted to ascertain the feasibility of procuring rectangular dies with widths approaching the circumference of a 2" (.0508m) O.D. steel core tube so that a single flat may suffice to make one preform. If this is feasible, removal of excess aluminum would be required only



at two edges of the wide preform. Alternately, it may also be possible to make a reasonably wide flat by cold rolling. It is certain that excess aluminum removal may be achieved by one or more of the approaches described above.

2.2.1 TUBULAR PREFORMS

Aluminum tube jacketed modules of Al/B prepared as above will be utilized in tubular preforms as follows:

A. Fifteen modules will be placed on the steel core tube as shown in Figure 1. A thin aluminum cover foil on the core tube may be necessary.

B. A ring compressor will then be tightened around these to position each module securely on the core.

C. The sheath steel tube will be slid over the modules and the preform will be cold drawn as usual. A thin aluminum cover foil on the modules may be necessary.

Although high aluminum concentration will exist between equally spaced Al/B, it is felt that two or three preforms should be examined without attempts at aluminum removal. These experiments will reveal the bending character of each module as the drawing progresses. The voids existing at the onset of drawing should gradually disappear



as the preform is squeezed between the strong core and sheath steel tubes. That the module will assume the tube curvature during deformation was demonstrated in a simple set up where a rod was placed on the module and compressed. The load required to do this was approximately 6000 pounds at room temperature.

Once the modular preform is proven adequate, hot drawing will be attempted. Only short 6" (.1524m) or 12" (.3048m) lengths will be examined initially. Upscaling the length of the tube to 3 feet (.9144m) will then be undertaken. Wide flat fabrication will also be examined for eventual conversion into hat sections.



3.0 ALUMINUM-GRAPHITE (Al/C) COMPOSITES

Effort was directed in three broad areas, namely, refinement of the existing electron beam apparatus, composite rod and tube fabrication in the same apparatus, and design of an electron beam apparatus capable of incorporating larger and longer preforms.

The general arrangement of the apparatus is shown in Figure 2 and which was described in the previous reports. In this apparatus, the operator had to manipulate the preform movement from left to right or vice versa by precisely rotating both feed through cranks. While the arrangement was satisfactory in these initial experiments, the probability of maintaining the preform at the center was marginal. To circumvent this problem, a motor was attached to the feed thru at left. As the preform size increased from a single yarn to several yarns, the 2KV capacity high voltage feed thru was replaced by an 8 KV feed thru. Larger preform also resulted in a larger amount of aluminum evaporation which necessitated shielding various contacts and portions of the steel base plate from short circuiting. Two ceramic stands with a hole for quartz cones (Figure 3) were incorporated. The preform now could be traversed from left to right as usual but, in addition, the quartz cones facilitated positioning of the preform at the electron beam emitter center even when aluminum melting caused the preform



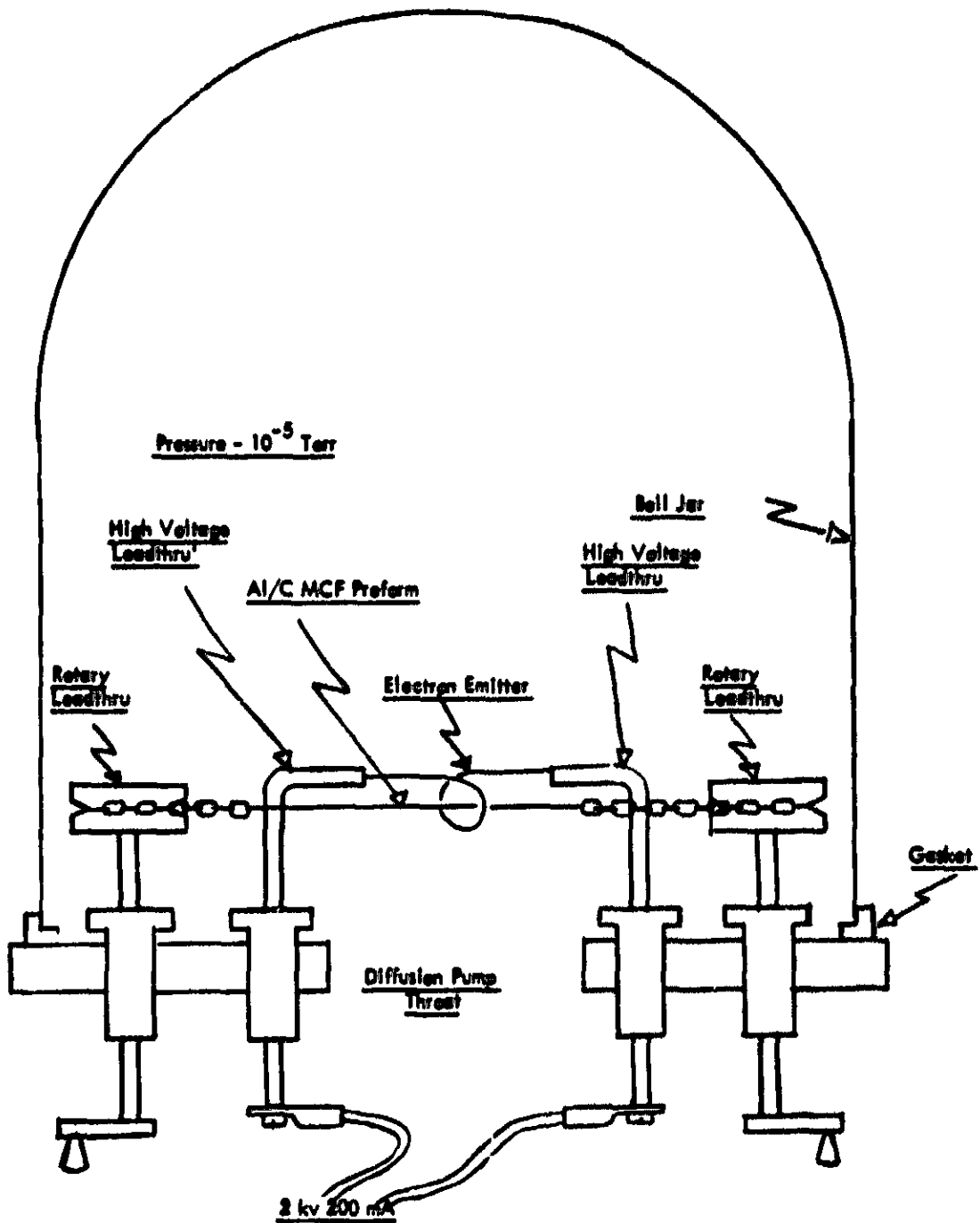


Figure 2. Electron Beam (EB) Apparatus for Fabrication of Al/C Composites, A Schematic.



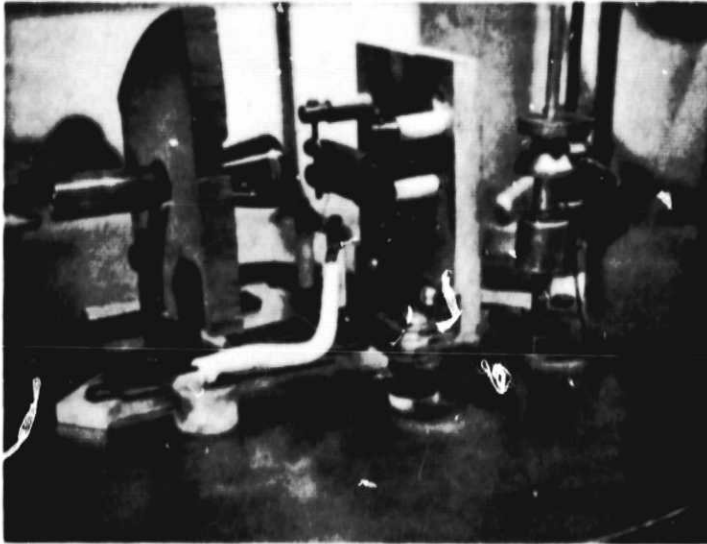


Figure 3. An actual photo of the apparatus in Figure 2 with ceramic stand and a hollow quartz cone for positioning the preform at the electron beam emitter center. The tungsten filament loop is barely visible from this angle.

to sag. As discussed later in this section, a graphite or quartz rod at the preform center nearly completely eliminated sagging.

Larger preforms and attendant rapid aluminum evaporation frequently resulted in a rise in the electron current. When this occurred, the power supply shut off automatically and thus prematurely terminating the experiment. Restarting the experiment was possible but not desirable. After eight resistors (in parallel) were incorporated in the power supply, this problem disappeared completely. It is realized that the voltage drop due to these resistors is reflected in the meter reading but this value is expected to be relatively constant for a given set of conditions and would not affect the experiment adversely.

The design of the larger E.B. unit which will utilize vertically moving preforms has been completed. As the purchased and machined components arrive, they are being incorporated in the E.B. system. When completed, the new system will appear as shown schematically in Figure 4. This system will be operational in early May 1974.



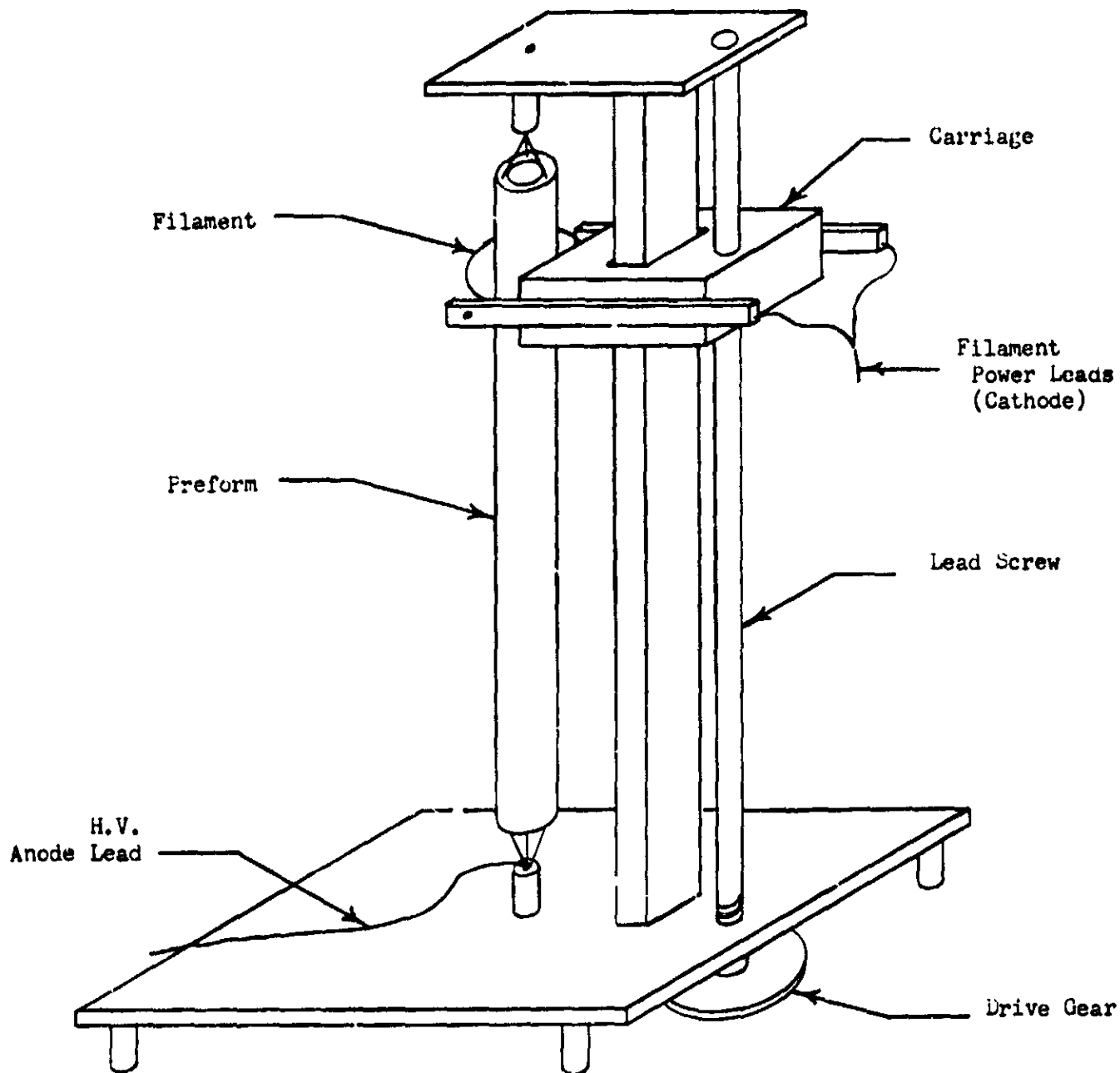


Figure 4. Schematic of the Electron Beam Apparatus with vertically moving preforms. Larger diameter and longer preforms will be fabricated in this system expected to be operational by May, 1974. The bell jar is omitted in this illustration for clarity.



3.1 WIRE, ROD AND TUBE FABRICATION EXPERIMENTS

Several experiments were conducted to prepare a single Thornel yarn infiltrated with aluminum. The primary objective of these experiments was to gain confidence in preparing small cross-section samples reproducibly. It was also hoped that the electron beam parameters established for a small size may also yield a good estimate for larger composites.

Although two samples, 2"-4" (.0408m-.1016m) long, were obtained, it was discovered that manual traversing of the reformer via the feed-thru could not always be performed with care. The main problem was gripping the matrix-clad-filament (MCF) securely at two ends without crushing the underlying Thornel (50 or 75) yet maintaining the continuity electrically to assure electron bombardment. Wrapping an excess aluminum foil at the two ends and then securing in an alligator clip alleviated this problem.

Five yarns with 718 (Al-12% Si) alloy were sufficiently straight to permit tensile testing. It was recognized that these tests would not yield accurate data due to excess aluminum on the yarn periphery and difficulty in measuring the cross-sectional area. Nevertheless, since the composite would fail at its weakest area, the degree of infiltration (or lack of it) along the entire length would be quickly



revealed. Furthermore, since each yarn is known to break at 18 to 20 lbs. (8.10 kg to 9.00 kg), (as specified by the manufacturer), the testing would at least reveal the reinforcement efficiency regardless of fiber content.

Of the five testable yarns, only two failed properly without fiber pull-out at fracture surfaces. The load to failure was 15 and 23 lbs. (6.75 kg and 10.35 kg) respectively, indicating that good reinforcement was achieved. In the remainder, loads as low as 5 lbs. (2.25 kg) and attendant pull out was observed.

Even if all the yarns had been fully infiltrated and shown good reinforcement efficiency, the ultimate use of these single yarns in making larger composites would be probably economically unjustified; that is, approximately 60,000 ft. (18,288m) would have to be processed to yield 2 lbs. (.90 kg) of composite wire. It was, therefore, decided to pursue multiple clad yarns bundled together for infiltration. The attachment of a bundle to the chain would be easier as compared to a single yarn and the total strength of the preform would be greater simply due to greater cross-sectional area.

Experiments involving five, six and seven matrix clad yarns were quite successful in attaining bundle infiltration. However, each bundle retained its original identity and refused to merge with adjacent yarn(s). This is shown in Figure 5 wherein each yarn is well infiltrated



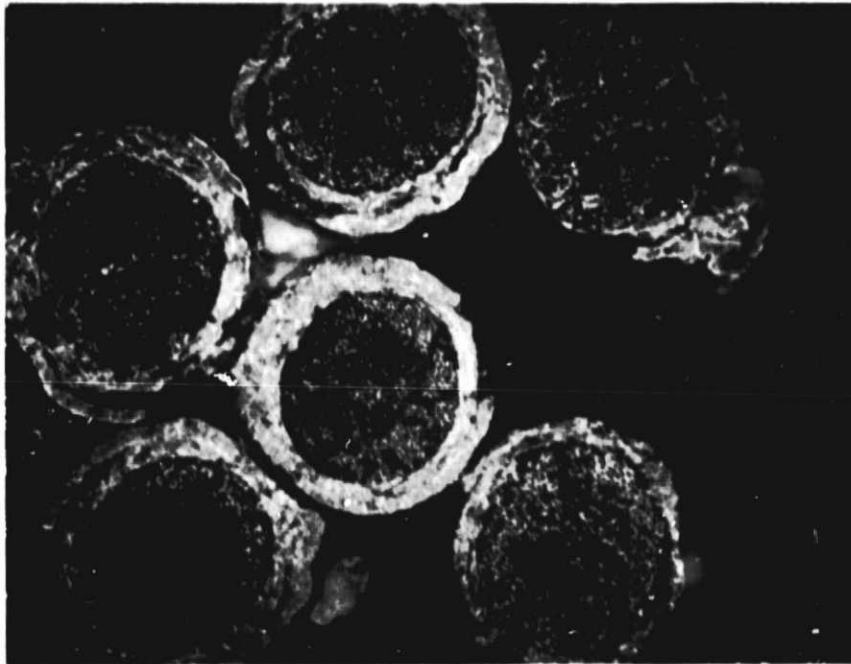


Figure 5. A typical example of multiple matrix clad Thornel yarns to separate even though each yarn is well consolidated. As polished, Dark Field, 80x.

but there it is clear that the yarns remain separate. Providing a large excess of aluminum in the form of thicker cladding did not completely alleviate the problem, though it was attempted several times (Figure 6). Within each yarn, however, infiltration was quite satisfactory as shown in Figure 7 with Al-12 Si matrix. There appears to be a distinct tendency for the silicon phase to solidify near and around Thorne1 particularly in the section. Closer examination reveals numerous entectiferous platelets also surround the graphite.

It was felt that although the electron beam was capable of infiltration, the lack of homogeneity should be overcome. Several experiments were, therefore, conducted by wrapping 7 nickel coated Thorne1 yarns together in aluminum foil. Typical result is shown in Figure 8. Infiltration is adequate but the composite cross-section is very irregular. As the 7075 aluminum melted and penetrated, the filament bundle the preform was moved gradually. The gravity and capillary forces combined must be responsible for this shape. Additional experiments proved this to be true.

The highly localized heating afforded by electron beam could be advantageously utilized if a pair of rolls or die could be positioned in close proximity to the electron emitter. Two half inch (.01270m) 2" long (.0508m) long titanium diboride-boron nitride rods



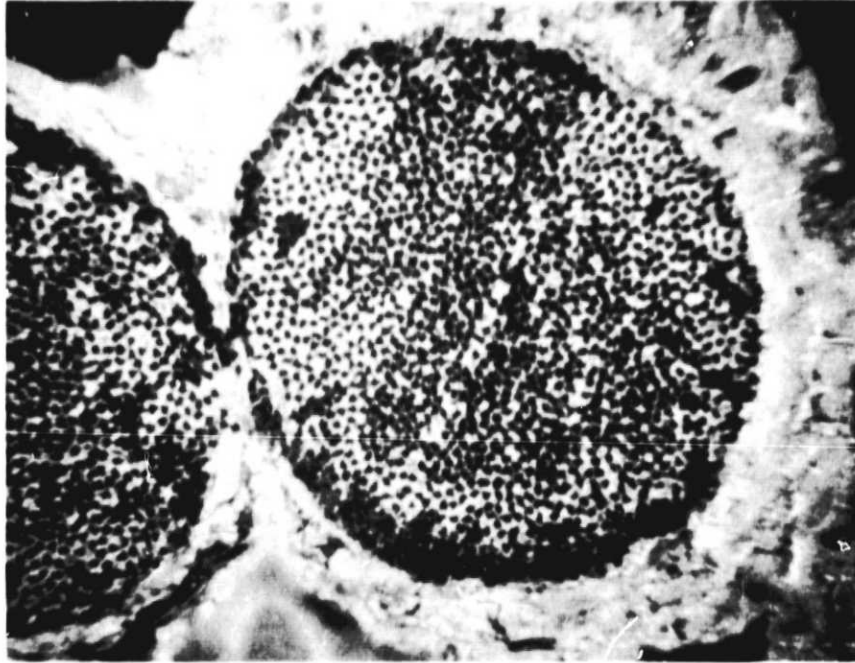


Figure 6. Large matrix excess in the form of cladding is advantageous in joining the infiltrated yarns. However, each yarn maintains its identity as shown in this micrograph. Etched (HF $\frac{1}{2}\%$), Bright Field, 200x.

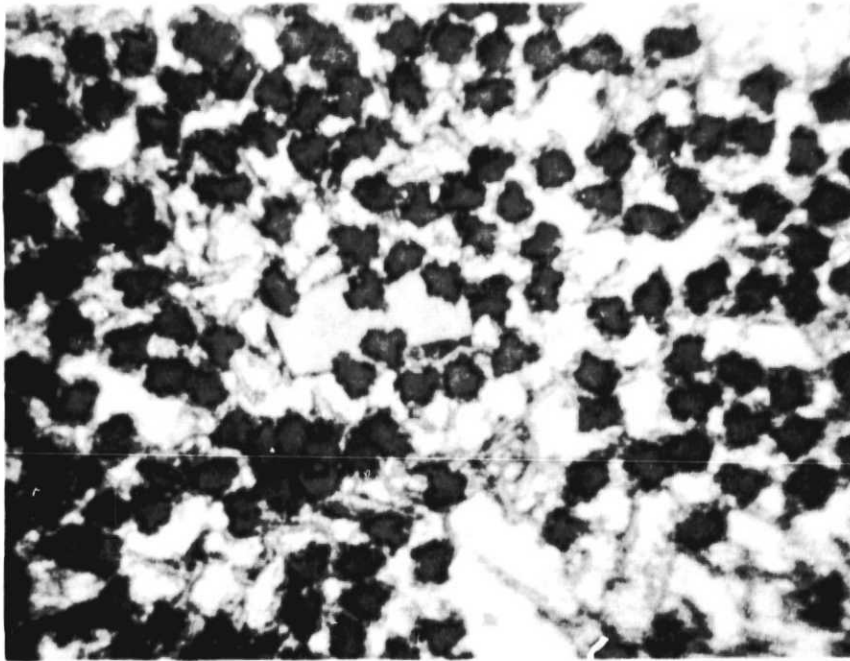


Figure 7. Portion of an aluminum-12% Si matrix infiltrated yarn at higher magnification shows good filament distribution and absence of carbides. Etched ($\frac{1}{2}\%$ HF), Bright Field, 800 x.

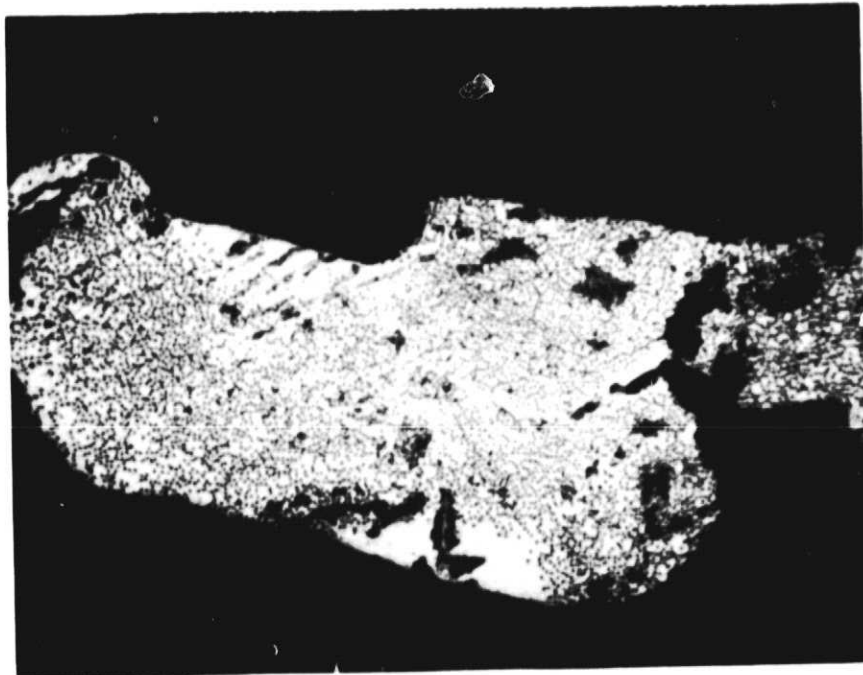


Figure 8. Irregularly shaped composite containing seven Thornel 50 yarns. As polished, Bright Field 80x.

mounted on a rigid ceramic frame were incorporated into the electron beam apparatus. The upper 'roller' was free to slide up or down so that the forces acting on the preform would be minimal. Figure 9 shows a preform in the rolls just prior to evacuation of the chamber (not shown). The rolls were approximately $\frac{1}{2}$ " (.01270m) away from the electron beam plane. Similar ceramic stand with a carbide die (not shown) could be positioned in place of the ceramic rolls. It may be noted here that these approaches were quite similar to those of drawing and rolling of Al/B rods and tubes.

Preforms for these experiments comprised of 10 aluminum clad Thorne1 ends inserted into an 1/8" (.00317m) O.D. al tube cold drawn just enough to squeeze the underlying material. In each of the six such experiments conducted it was found that successful infiltration could be achieved only with much greater sophistication in roll and die design. Despite the use of the TiB₂ intermetallic composite which is known to be resistant to molten aluminum, there were definite indications of reaction and wetting. The tendency of aluminum to pile up at the die entry was also quite persistent despite rapid preform movement. This approach was temporarily abandoned in favor of tube fabrication attempts discussed below.

Tubular composites are of prime interest in this investigation. To fabricate a tube via electron beam, it would necessarily require



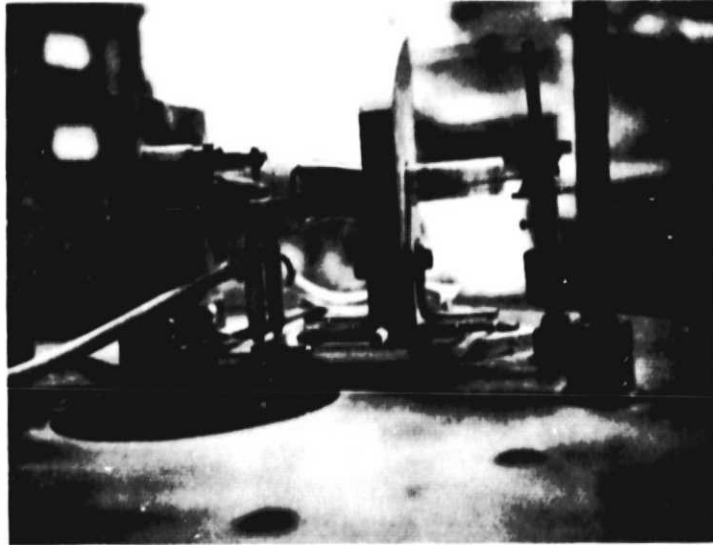


Figure 9. Electron beam apparatus showing the TiB_2 -
TiN rolls and preform in position prior
to evacuation.

an expendable mandrel. It, therefore, appeared expedient to examine tube fabricability even though all experimental parameters to produce a small wire or rod had not yet been generated or optimized. This mandrel would also support the preform as it was moved through the beam. It was also reasoned that aluminum rich areas (see Figure 8) formed due to gravity may be at least partially overcome and a more homogenous composite may be fabricated.

A series of experiments were conducted to examine the approach above. Glass and bulk graphite in rod form were appropriate choices as mandrel materials.

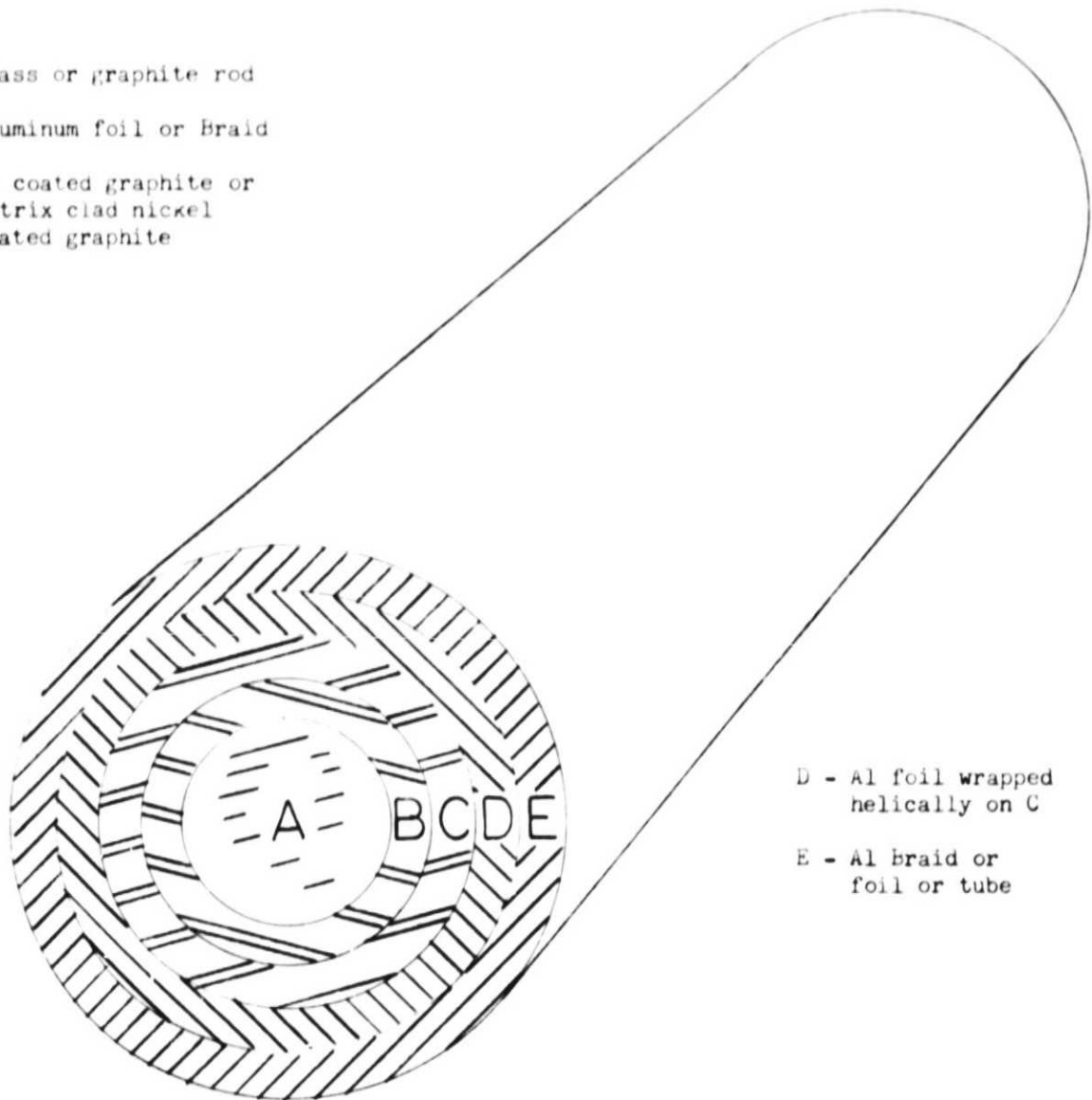
The basic preform configuration developed and utilized in these experiments is shown schematically in Figure 10. From center outward, the preform comprised of the mandrel (A), aluminum cover (B) on the mandrel, longitudinally aligned nickel coated graphite layer securely and finally, massive aluminum layer (E).

Wherever the outside layer E was a tube or a thick foil, it melted suddenly and the preform (under slight tension) pulled apart exposing the underneath layer of Thorne1. Helically wrapped aluminum foil in layer E exhibited somewhat superior melting character but infiltration was sporadic.

Best success was achieved in recent experiments by utilizing aluminum braid in layers B and E. That is, a glass rod was inserted



- A - Glass or graphite rod
- B - Aluminum foil or Braid
- C - Ni coated graphite or matrix clad nickel coated graphite



- D - Al foil wrapped helically on C
- E - Al braid or foil or tube

Figure 10. Basic arrangement of materials including filament and the matrix for tubular composites. A schematic.



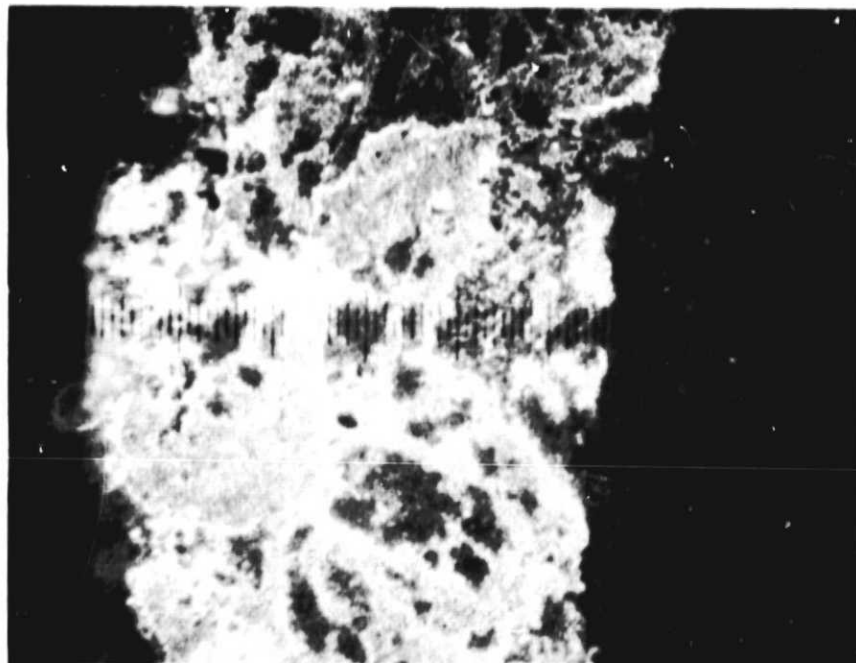


Figure 11. Micrograph of a portion of a tube showing partial infiltration. The black areas show clusters of filaments not penetrated by the matrix. In well-infiltrated areas, the filament distribution is fairly satisfactory. As polished, Dark Field, 80x.

into an 1/8" (.00317m) I.D. followed by the nickel coated Thornel layer (C) 0.001" (.0000245m) aluminum foil wrapped helically, and the braid, in that order. The braid alloy was 5056 and it was woven identically as the braided shielding generally found in coaxial cables. The braid wire diameter was 0.005" (.0001225m).

For a reason as yet unknown, the braid melting and evaporation characteristics were quite different than other materials such as tube and foil. It was possible to observe the melting distinctly and simultaneous shrinkage in the O.D. of the preform signalling the onset of infiltration. The preform was moved through the beam at this point to expose melted aluminum along its entire length.

Five tubes measuring approximately 3/16" O.D. (.00459m), 0.030"-0.040" (.000735m - .000980m) wall, 3" (.0735m) long were prepared. Although these tubes invariably showed large totally uninfiltreated areas, the metallographic examination shows considerable promise. This is shown in the micrograph of Figure 11. The dark spots are bunched up filaments not adequately infiltrated and this is largely or completely attributable to the present method of aligning the nickel coated Thornel in the preform. Despite considerable care, the filament bundle will shift. The twist in the two ply yarn also creates areas of varying filament concentration along the preform length. Spreading the nickel coated yarn is exceedingly difficult and non uniform.



When the preform is heated in the beam, the outer aluminum, be it braid or foil or tube (D and/or E in Figure 10), expands away from the underlying graphite. Layer B aluminum, however, is always at a lower temperature than layer D or E as the electron beam bombards and heats this layer first. Additionally, the aluminum evaporation and consumption on the outside is not easily controlled. It has often been observed that aluminum supply depletes very rapidly exposing the graphite (bare or partially infiltrated) to the beam directly. This is not desirable as the small amount of aluminum in the partially formed composite is rapidly consumed thus 'reversing' the infiltration process.

Majority of the difficulties enumerated above can be largely circumvented by 1. reducing the aluminum depletion through evaporation, 2. uniform filament placement during preform preparation and subsequent infiltration, and 3. good physical contact between aluminum and graphite bundle.

The first condition may be fulfilled by superimposing a steel jacket over the preform by drawing. Steel dissolution in aluminum may be expected though a parting compound may prevent excessive reaction. Filament alignment could be improved by use of a fugitive binder. The physical contact may be improved substantially by heating the outer braid in the beam along the entire length to just under its melting point while maintaining a uniaxial tension so that it squeezes



over the preform when it is cooled. More of the aluminum on the outside will then be utilized in infiltration instead of being largely consumed in evaporation. These and other approaches are being implemented at the present.



4.0 FUTURE WORK

Al/B System

Modular preform preparation will be evaluated thoroughly to ascertain the advantages gained by the absence of binder. Excess aluminum at periphery of each module is not desirable and, therefore, methods such as chemical etching and/or milling will be examined to eliminate all or most of the matrix excess. If this is not readily achieved, wider module preparation will be undertaken. It is felt that though this approach involves purchasing of additional rectangular dies, the ability to produce 2", 3" or 6" (.508, .762, or .1016m) widths will help in hat section fabrication as well.

Al/c Composites

Vertical preform traversing system now being designed and constructed will be installed and made operational. Attempts at eliminating the inconsistency in infiltration in tubes will also receive major emphasis. Upscaling to larger diameter tubes will be undertaken after smaller tubes have been obtained reproducibly.



Delivery of Al/B to MSFC

At least two Al/B tubes, 2" (0.508m) O.D. and 6" (.1016m) long will be delivered to NASA before June 30, 1974. If the modular preform approach is successful with short lengths, longer tubes will be fabricated and evaluated at Commonwealth Scientific Corporation, and also delivered to MSFC as soon as possible. It is hoped that modular preforms may also enable hat section delivery within the same time frame.

