NASA CONTRACTOR REPORT

NASA CR-142

NASA CR-142

STUDIES OF HOLLOW MULTIPARTITIONED CERAMIC STRUCTURES

by William J. Eakins and Richard A. Humphrey

Prepared under Contract No. NASw-672 *by*
DeBELL AND RICHARDSON, INC.
Hazardville, Conn.
for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • DECEMBER 1964

STUDIES OF HOLLOW MULTIPARTITIONED CERAMIC STRUCTURES

By William J. Eakins and Richard A. Humphrey

Prepared under Contract No. NASw-672 by
DeBELL AND RICHARDSON, INC.
Hazardville, Connecticut

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Office of Technical Services, Department of Commerce,
Washington, D.C. 20230 -- Price $3.00

FOREWARD

This report describes the work done under Contract NASw-672, "Hollow Multipartitioned Ceramic Structures" for Headquarters, National Aeronautics & Space Administration, Washington, D. C. The work covered by this report represents the first half of the total planned program, therefore, any conclusions given here are of an interim nature. The contract was administered by Mr. M. Rosché, Chief of Space Vehicles Structures Programs. Mr. Norman J. Mayer, Chief, Advanced Structures and Materials Applications, is Project Manager.

Under the supervision of Mr. William J. Eakins, Chief, Glass Fibers & Composites Section, Mr. Richard A. Humphrey was Project Engineer. Able assistance was provided on all phases of the program by Mr. Dan Hess, Glass Fibers Technician. Mr. Dixon Wetherbe drew the hexagonal tubes and Mr. Lewis Heath worked on the precision winding.

ABSTRACT

Results are presented of a fifteen month program of forming glass filaments whose shape is other than a solid round. The feasibility of drawing precise geometric shapes of fibers is demonstrated. With the objective of high stiffness-to-weight ratio, most of the fibers were drawn into hollow cross sections of various shapes for subsequent filament winding. Of particular interest are hollow hexagonal, triangular and rectangular filaments. Surprisingly complex hollow fibers can also be drawn by the highly refined preform attenuation process used in this program.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS

I. INTRODUCTION

Filament strengths of over 500, 000 psi are commonly realized in certain glass formulations as circular filaments. However, it is impossible to pack these filaments to over 90. 7% by volume. On the other hand, hexagonal or rectangular forms have the advantage of 100% theoretical packing. Filaments with contoured cross sections having angular characteristics like a hexagon as well as wide and strong filaments like a wide rectangle may be chosen to dove-tail or mate with adjacent filaments and yield a packing of nearly 100% by volume.

It is possible to filament wind structures of high stiffness-to-weight and good strength using hollow fibers. The transverse modulus could be optimized by using very hollow filaments with outside shapes that can be made to mate with one another. Only a small amount of resin would then be required to fill the thin joints between adjacent fibers. If circular parallel filaments are packed in a somewhat random configuration approaching but not reaching 90. 7% by volume, the matrix (when viewed normal to the axis of the filaments) appears to be composed of a series of films and tricorn-like shapes. Stress between filaments, by-passing the tricorns, will tend to go through the films at high concentration. For a discussion of the interaction between resins and glass filament see Appendix I. In composite structures composed of filaments with mating surfaces, the low matrix-to-filament-volume ratio and the thin adhesive layers should provide much more uniform stress transfer throughout the structure.

The purpose of this program, therefore, is to study filaments composed of glass films and determine their value. As will develop, the work covered by this report is largely exploratory. To date, a way has been found to make filaments in regular and sharply defined shapes having almost every conceivable variation in construction.

It is a further purpose of this program to demonstrate to designers that they may conceive of a composite material in which the filament has a pre-engineered cross section and that these filaments may be drawn and wound.

During the past few years, DeBell & Richardson has undertaken the drawing of glass filaments whose cross section was not a simple round solid. Preliminary experiments on rectangular filaments were first made over a laboratory burner and later almost square fibers were formed in a vertical electrically-heated furnace with a localized hot zone. The latter work was undertaken as a supplement to a Navy Bureau of Weapons program (1) and it proved the feasibility of forming solid rectangular fibers. Subsequently, under a small Office of Naval Research contract (2), solid hexagonal E-glass filaments were made here. In order to maintain crisp reproduction of the shape of a preform other than round, it was necessary to refine the process substantially. Some historical notes which describe the early beginnings of the technique used on this contract can be found in Appendix II.

General Objectives

Conduct studies of hollow ceramic filaments for the purpose of providing more efficient structural use of these materials. Utilize the higher strength values which can be obtained with a multiple number of properly oriented thin glass films by providing hollow filaments of geometric shapes and multiples.

Specific Objectives

A. Investigate the following filament designs:

1. Elementary hollow cross sections: square, triangular, hexagonal, etc. filaments
2. Complex equidimensional cross sections
3. Complex tape-like cross sections
4. Special shapes and materials
5. Hexagonal tube studies

B. Throughout the above investigations conduct the following studies:

1. Evaluate structural advantages of filament forms by comparison with conventional circular solid and hollow fibers.
2. Investigate packing densities including the advantages of precision winding as well as the feasibility of bonding with resins.

3. Determine the limitations of principle designs and development processes.

4. Attempt to find methods that will provide solutions to the limitations found in designs and processes.

II. EXPERIMENTAL PROCEDURES

The Preform Attenuation Shaped Fiber Forming Process

The process utilizes a mechanical device for feeding a shaped preform slowly down into a furnace with a carefully tailored vertical temperature gradient and good temperature control, as well as a winder whose speed can be coordinated with that of the feeder. Figure 1 is a sketch showing the essentials of the process. The furnace is shown in section so details of the attenuating preform and its relation to the heating elements and the cooler can be seen.

The feeder must lower the preform vertically into the furnace at a slow, monotonic rate which can be readily adjusted. The preform must in turn enter the furnace through an opening which permits a minimum of draft to escape, minimizing the chimney effect and turbulence in the furnace.

Although there are special cases for some preform shapes, the best furnace design for most shaped fiber forming provides a gradual increase in temperature as the preform moves downward. The maximum temperature is reached at about the point where final attenuation takes place. At this point, the temperature must be lowered abruptly to provide essentially an air quench and thereby maintain the shape of the filament. This vertical temperature gradient is graphed in Figure 2.

A photograph of the forming apparatus and winding equipment is shown in Figure 3. The furnace has been partially dismantled to show the position of the attenuated section of the preform in relation to the heating elements and cooler.

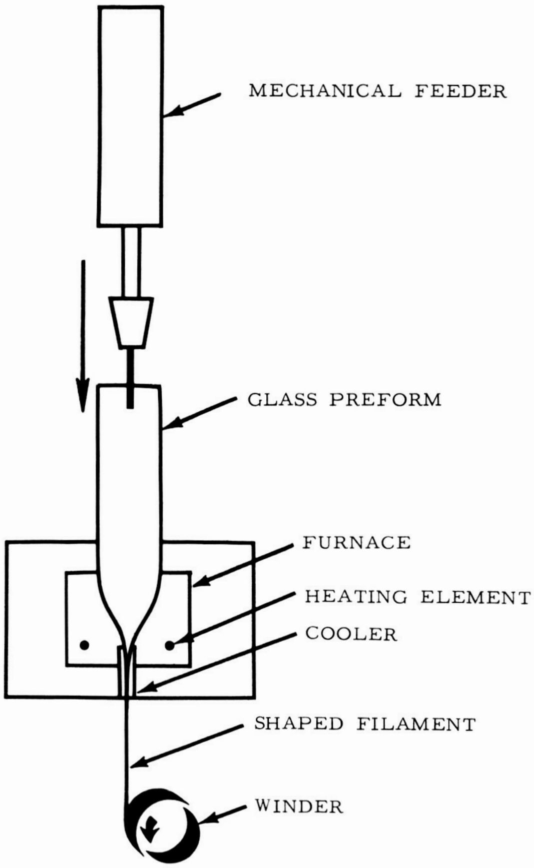
Preform Fabrication

In order to draw a shaped fiber by the preform attenuation or rod drawing process, it is first necessary to fabricate an elongated preform. Its cross section should approach that of the fiber it is desired to produce. For example, to make hollow square fibers, a long slender open ended square box is assembled from four narrow strips of glass cemented together with cellulose acetate adhesive.

The acetate burns off the glass as the preform enters the top furnace opening. The stiffness of the glass maintains the separate pieces in proximity because of the unburned cement higher up on the preform. A typical preform is shown in Figure 4. The glass plate must be cut and glued in a precise manner. If the surfaces do not abut on one another throughout their length, the sides of the filaments will tend to separate at the joints after the adhesive has burned away during the drawing down operation.

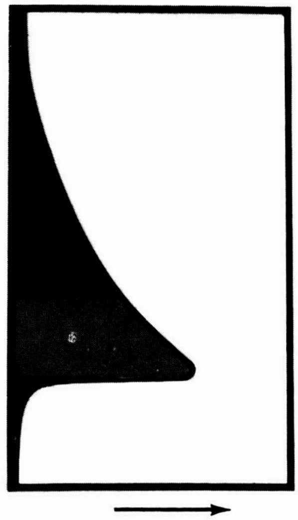
Preparation of Samples for Examination

In order to examine under the microscope the results of the filament forming studies, it has been necessary to develop a technique for mounting, grinding, polishing and lighting the bundles of shaped filament that had been wound on a mandrel. Good results have been achieved by tying fibers into a bundle about 1/8" in diameter. This bundle is then immersed in a low viscosity epoxy resin and cured. A thin wafer is cut out through the embedded bundle so the fibers run normal to its surfaces. Then the wafer is ground and polished using good metallographic specimen preparation practice. An expedient that has assisted in obtaining sufficient contrast between glass and resin, especially for photomicrographs, has been the scorching of the surface of the sample after final grinding and then polishing only a minimum amount thereafter. The light then is transmitted vertically up through the glass walls to the top surface of the wafer mounted on a microscope slide. Only the very thinnest walls are difficult to light and photograph in this way.



SKETCH OF DeBELL & RICHARDSON PROCESS

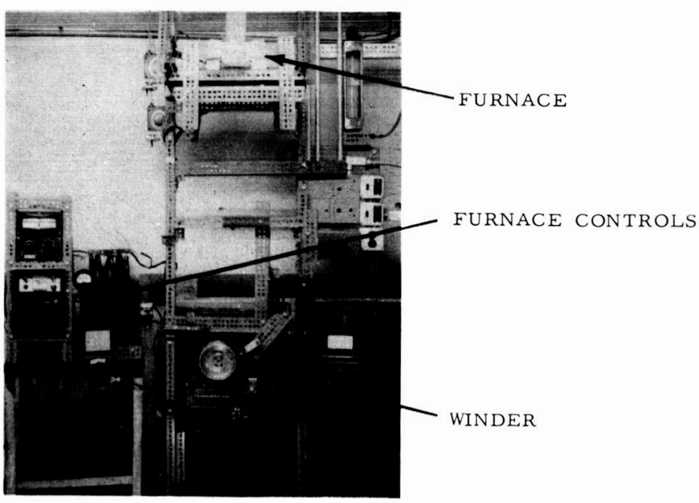
FIGURE 1



TEMPERATURE PROFILE

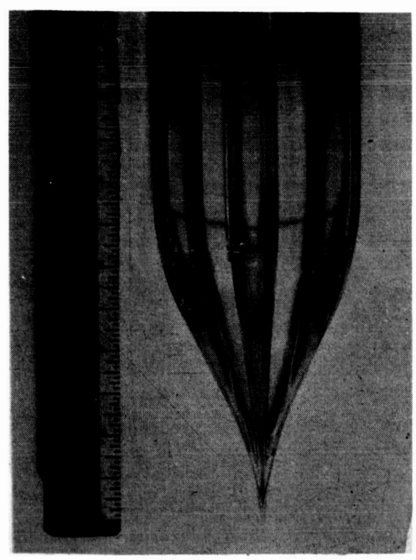
VERTICAL TEMPERATURE GRADIENT
THRU FURNACE

FIGURE 2



FORMING AND WINDING APPARATUS

FIGURE 3



TYPICAL PREFORM AFTER ATTENUATING

FIGURE 4

III. FILAMENT FORMING STUDIES

Literally hundreds of different cross sectional shaped filaments were formed and examined. Each shape has its own peculiarities that must be dealt with through proper control of the many variables in the forming process described earlier. A summary of the shapes illustrated in this report is found in Table I. It describes the shape of the preform, the attenuation conditions achieved through proper control of the variables and the characteristics of the filament formed including its size. Table I should provide a convenient guide to the 64 photomicrographs of various filament cross sections.

Elementary Shaped Hollow Filaments

Small preforms of simple cross section create fewer problems in reproducing a filament whose shape is a miniature of the original preform. A hollow triangular fiber is shown in Figure 5, a photomicrograph at 100X of a section through an embedment. The slightly convex walls are interpreted to mean that the temperature got high enough during forming that the lowered viscosity of the glass permitted the surface tension to start deformation toward a round fiber. When one thinner wall is used in the preform, the distortion becomes unsymmetrical and the thinner wall begins to thicken as in Figure 6. To avoid the bulging sides noted in the filaments of Figure 5, a vacuum of about 1.5 inches of water was applied to the interior of the preform and a series of filaments were drawn like those shown at 100X cross section in Figure 7. While almost complete collapse is the result, the filaments are interesting in that they have a Y shape that mates readily. Although hollow, they are extremely rigid. If a slight vacuum is applied to the triangular preform with one thinner side, the sides of the filament may be formed straight (Figure 8) rather than rounded as in Figure 6.

A variation of this design (Figure 9) consists of cementing a rod to each corner of the preform to give added glass thickness at the joints or corners. In Figure 10 are shown filaments made from a triangular preform like those in Figure 9 except that the plates were cut overwidth and the corner or glue joint was made at one-fifth the width, allowing a projection of the plate outward at each corner. The rod was then glued to seat within the obtuse angle formed between this protrusion of one plate and the adjacent piece. When the pressure was reduced inside the preform, a true Y shaped filament was formed with circular ends or lobes and with a small hole in the center.

Similar studies were made with hollow square filaments (Figure 11). Using the same thickness of glass on all four walls, the essential shape of the preform was maintained. The glass for the triangular preforms was cut with square edges. At a sixty degree angle, these edges do not mate well but leave excess edge of each end showing. The result was that the hollow square filaments can be made with flat sides at cross sectional dimensions of 2 mils or less. By making the preform with adjacent sides of different thicknesses 1.0 : 1.5, a rectangle resulted with the heavier sides slightly bowed inward (Figure 12). When the pressure was lowered within a hollow square preform having sides of equal thickness, an X resulted as shown in Figure 13. When the pressure was slightly reduced within the hollow square preform with adjacent sides of different thicknesses (1.0 : 1.5), the particular form shown in Figure 14 was the result.

To further study the effect of thickness of the walls of a hollow preform on filament geometry, a single piece of window glass (0.070" thick) was inserted and glued within a very thin-walled (0.036") soda-lime glass tube (a fluorescent tube with the coating removed by washing) to give a cross section like Ⓘ. The resulting filament cross section is shown in Figure 15. Possibly slightly different softening temperatures caused the distortion. When plates of similar thicknesses were inserted to form an X within the tube, the resulting shape of the filament resembled a swastika (Figure 16). The glass tube remained intact although it is so thin that the transmitted light is insufficient for satisfactory photography.

Hexagonal hollow filaments were also studied. In another section of this report, the manufacture of straight one-half inch and one inch diameter hexagonal tubes for making preforms is discussed in detail. A single hollow hexagonal preform, one-half inch in diameter was formed from a glass tube of similar size. A hexagonal filament was formed from the hexagonal tube. It had thin walls and a hexagonal shape both inside and out (Figure 17). Reducing the pressure on the inside of the preform (1.5" water) and forming the filament produced a six tooth gear effect that readily interlocks (Figure 18).

It was difficult to form hollow hexagonal shaped filaments from strips of glass glued together to form a hexagonal preform. The greatest success was found with the preforms glued as illustrated in Figure 19a, while the filament cross sections are shown in Figure 19b. The inside surface went round due to the larger amount of glass at the junction points.

In forming a hollow hexagonal filament from larger hexagonal preforms, the ratio of wall thickness to preform "diameter" must be low. The wall thickness should probably be .02" or less in order to maintain the 60° angles at each corner. Some 1" hexagonal tubes with walls 0.030" in thickness were purchase(They would not draw into filaments with sharp corners. It was necessary to set up equipment to draw one inch hexagonal tubes from round tubes of the same glass formulation. These tubes, having a wall thickness of 0.015", drew to sharp angled filaments.

It was found in previous work that round hollow filaments with exceedingly thin walls can be made if the upper end of the preform tube is closed during the forming operation, thus a slightly elevated pressure caused by the heated air can be created within the tube. This pressure causes the glass filament diameter to remain relatively large as the filament wall is thinned through attenuation. Figure 20 illustrates how thin-walled these filaments may be. The walls of these filaments look thicker than they actually are due to difficulty in getting the thin, bright line onto film. The true wall thicknesses are seen better where the transmitted light is low and the contrast is therefore poor. The bulk density of these filaments is under 0.1. They may be filament wound to form a hoop with a resulting specific gravity in the order of 0.7.

Small tubes with a closed end may be used to create certain geometric effects within more complex filament structures. Three circular tubes having about the same wall thickness were telescoped one inside the other. Each inside tube was held in place by means of small diameter tubes, three in each course. Figure 21 shows a number of filaments drawn from this preform. The small tubes of the inner layer were offset 60° from those of the outer layer. Each of these smaller spacer tubes were closed on the upper end while the upper end of the larger preform tubes was left unsealed. The lower end of the preform entered the furnace and was attenuated into the filaments illustrated. The smaller or spacer tubes became very thin-walled but on attenuating downward kept the inner telescoped tubes from joining the outer tube. The result is the filament shown. If the number of tubes in the outer layer were doubled in number, direct contact between the middle and outer telescoped glass was avoided in the drawn filament (Figure 22).

A recent study (3) has shown that increased transverse stiffness may be obtained in filament wound cylindrical structures if elliptical fibers instead of round fibers are used and if these fibers are wound with the long axis of the elliptical cross section parallel to the axis of the cylinder. Some time was devoted to developing techniques for making both solid and hollow elliptical filaments.

Hollow rectangular preforms yielded slightly elliptical filaments but with ends of the long axis squared (Figure 23). When two cross members were added, reducing the segment to a 3:1 width-to-thickness ratio, the degree of bulging was controlled (Figure 24). When an additional cross member was added, reducing the segment ratio to 2: 1, a rectangular form with straight sides resulted (Figure 25). The section photographed here was of some windings made into a ring on a mandrel, showing how well they packed.

Hollow elliptical fibers were first studied using four cut plates of glass segmented to make a preform having a diamond shaped cross section. The surface tension of the softened glass caused the diamond to distort into an elliptical or football shape, as can be seen in the photomicrograph of the cross section (Figure 26). To eliminate the pointed ends, the technique discovered while investigating hollow rectangles (Figure 23) was applied. By slightly overheating a hollow rectangular preform, especially on the ends or short dimension, a short radius could be formed tangent with the long radius on the length of the rectangle. Figures 27 and 28 show cross sections of these filaments with different length-to-width ratios.

Hollow elliptical fibers may be made in still another way. A one inch diameter tube with relatively thick walls (0.03") and a flat glass plate inserted across its internal diameter may be drawn to form a filament like those shown in Figure 37.

Solid elliptical fibers were made using a variety of preform shapes. The work done at General Electric (3) had shown analytically that the major to minor axis ratios should be four to one or greater. In one technique to produce elliptical fibers, glass rods of various sizes were arranged in a line with the large diameter rods in the middle and gradually smaller diameter rods to the edges to form the shape of an ellipse. Operating at slightly elevated temperatures caused them to fuse together while being drawn into a fiber. It was found difficult to make high ratio ellipses (Figure 29) because a temperature sufficient to cause complete fusion also caused excessive shortening of the longer axis by surface tension. Although a hollow rectangular preform would yield a somewhat elliptical shape (Fig. 23), a solid rectangle yields an almost flat filament with rounded ends (Figure 30). However, by grinding off the corners of a narrow strip of glass, it was found that the crude elliptical section of the preform could be refined in drawing the filament form (Figure 31). Should it be required to make a quantity of high ratio preforms for drawing into elliptical shaped fiber, the desired preform shape could probably be obtained by hot rolling.

Complex Equidimensional Cross Sections

Honeycomb filaments may be made by several techniques. Making a preform with six round tubes in a hexagonal array (but without a center tube) drew down to an interesting filament. An enlargement of this filament at 250 X is shown in Figure 33. The surface tension at the inside corners has straightened the inside surfaces to make a hexagonal hole. This unit structure may be filament wound (Figure 32). The outside semicircles are mechanically interlocked to form a hollow, light structure with low resin content.

Hollow filaments with honeycomb walls are readily made using an array of round tubes cemented together. Elliptical shapes with different major to minor axis ratios are shown in Figures 34, 35 and 36. Square filaments with segmented walls were also made (Figure 38).

A completely honeycomb filament was also investigated. A square filament was made from a 4 x 4 stack of sealed tubes and an outside plate about 50% thicker than the tube walls. The resulting filament is shown in Figure 39. Note that the "quadri-corn" voids between the tubes has disappeared. An enlargement of one of these filament cross sections (Figure 40) at 250X shows that the slightly increased pressure within the tubes with a sealed end caused a flattening of the sides of the holes. Presumably, at still higher pressures, the holes could be developed to yield almost a square shape. Figure 41 shows hexagonal shaped filaments having a honeycomb interior. Tape shapes can also be made in a similar manner. Figure 42 shows an enlargement at 250X of a honeycomb tape filament cross section. Note that the holes at the outside short edges of the tape are circular but become quickly oblate with the most elliptical holes in the center. Note also that the center section is thinner than at the ends. These effects suggest that the motion of the edges in the drawing down of the preform created a slight stress across the center section of the attenuating tape, distorting and thinning the tape between the ends as indicated. Circular honeycomb filaments have also been formed using a one inch diameter thin shell outer tube filled with 1/8" diameter tubes with one closed end. The wall thicknesses of the outer tube and the smaller tubes are about equivalent. Such a shape suggests filter applications.

Honeycomb filaments and tapes can be drawn directly from a bundle of small sealed tubes that have been glued together. The cross sections shown in Figure 43 are an example. Note that the outside surface is extremely thin. The inside partitions were formed from two tube walls while the outside wall was formed from only one.

Complex Tape-like Cross Sections

Solid Filament Tape:

Concurrently with this program, another quite different study (4) is being conducted which uses one specific shape of filament. In this parallel program, hoops and cylinders are being made of a flat rectangular-shaped filament about 0.0005" thick and 0.016" wide. These microtapes can be wound to form composites with a glass content approaching 100%.

There are many varients possible in the basic tape shape. For example, a preform may be made by glueing the edge of narrow strips to the base plate. The resulting filament is like a zipper (Figure 44). When three small rods were glued in a triangular stack to opposite sides of the plate, a hump-backed tape resulted (Figure 45). Tapes may also be made from rod. When the rods were offset ±120°, a tape with a mechanical locking feature resulted (Fig. 46). E-glass rods without a plate to support them, slightly offset and drawn to a tape form, retained a slight interlocking effect (Figure 47). A tape form was even made that retained the letters of the preform - NASA (Figure 48). Alternating large and small diameter rods, each in line in the preform, gave a cross section of a tape filament form which has a series of shallow bumps on its surface (Figure 49). However, when two small diameter rods are inserted in line between two large diameter rods, a useful interlocking shape appeared (Figure 50). It was formed to give equal spacings across its five unit width. Figure 51 shows a five unit tape with the same basic preform unit groupings as in Figure 45. When the three rod pyramid groupings of these preforms were spaced farther apart on the plate, the unusual three unit tape shown in Figure 52 resulted. When single relatively large diameter rods (2X thickness of plate) were mounted in spaced fashion back to back on either side of the plate and the resulting preform was attenuated, the somewhat ungainly tape resulted (Figure 53). Had the spacing between the adjacent rods been slightly greater, the corrugations would have meshed.

When three sealed tubes were stacked in a pyramid, as were the rods, to form the preform used to make the tape filaments of Figure 51, the filament cross section drawn looked like those in Figure 54. When only single sealed tubes back to back were used on the same plate base, the tape filaments shown in Figure 55 resulted. This form compares with the filaments in Figure 53 drawn from plate and rod.

Hollow Filament Tape:

The simplest form of hollow filament microtape is shown in Figure 56, a line of sealed tubes that have the appearance of being laterally stretched. In forming the microtape, the glass at the edges must travel a longer lateral distance than the glass at the center section. When the heating zone is too short and the difference in travel distance changes rapidly, the edges tend to remain wider apart applying a stress to stretch in the middle tubes. The filaments in Figure 57 were made from a preform in which the sealed tubes were offset as were the rods in Figure 46. Sealed tubes, aligned to form a preform with a rectangular cross section, instead of producing a filament like those in Figure 23, made the collapsed filament structure in Figure 58. However, adding a sealed tube spacer made a tape with a relatively well-oriented structure (Figure 59). This filament compares favorably with those drawn from plate (Figure 24 and 25).

Hollow filament tapes were made using a line of tubes with pyramids of three tubes periodically placed to corrugate the surface of the tape. Two different spacings are illustrated in Figures 60 and 61. In the latter microphotograph, note that some of the stacks of three did not seal to the center line of microtubes during drawing.

Two hollow tape shapes were studied that would have high rigidity. These are pictured in Figures 62 and 63. The first was made like a bridge truss and would have high stiffness-to-weight from edge to edge. The second was from a preform made by mounting plates on either side of a line of tubes. It would display high stiffness-to-weight from flat to flat.

Special Shapes

While working on these new forms, it became evident that certain types might be chopped and used to make improved molding compounds. A very simple form is shown in Figure 64. The preform used was made by cutting a slit in the circumference of a tube. When chopped, a considerable number of these C-shaped filaments might interlock to form strings in all three places. The polymerized resin within the radius of curvature is restrained in one direction of movement and, therefore, should more tightly grip the glass of another filament that has been inserted within this enclosure. Figure 65 shows an X-shaped filament of four filaments connected with films of glass. They could fit with the filaments shown in Figure 66 in a tongue and groove manner. Once the tongue has been shoved into the groove, it should be difficult to remove.

Packing Density and Precision Winding

In order to take advantage of most of the shaped filaments which it has been demonstrated can be formed, it is essential to be able to wind them in proper relationship to one another. Typically this means close packing with flats against flats or mating surfaces against each other.

During the course of the work which has been done on the aforementioned microtape program for NASA Lewis Research Center (4), a great deal of effort has been applied to the difficult task of winding filaments in perfect juxtaposition. The larger dimension of a typical microtape cross section is 0.015" while the thickness is only .0005". This means that there is little or no "curb" to slide one microtape against its neighbor. Some additional work was done under this contract to solve the problem using microtape as a model shape. Since it is impossible to postimpregnate a properly wound microtape specimen with resin, it required wet winding. A substantial improvement was achieved by combining wet winding and accurate guide advancement with wiping or squeegeeing right at the point of tangency of the microtape with the mandrel.

Figure 67 is typical of the poor placement readily obtained by imperfect winding. Figure 68, on the other hand, illustrates the improvement possible when all aspects of winding were functioning properly and wiping was helping to provide perfect placement. This means the tape width was in near perfect agreement with the traverse advancement, that the width remained nearly constant, that the resin was at proper viscosity and that the wiper was doing its job in a satisfactory manner.

The improved winding technique should help to utilize the multitude of shaped filaments DeBell & Richardson, Inc. has developed under this contract in high stiffness-to-weight structures and other special purpose composites.

Although this contract speaks of ceramic fibers, there was some interest expressed in applying the preform attenuation process to certain of the more or less amorphous organic materials. Appendix III is some notes on the results of a cursory examination of drawing organic filaments.

Hexagonal Tubes

In view of the perfect packing possible with hexagonal fibers, it was planned at the outset that this program should include a substantial effort on hollow hexagonal tubes and, more especially, hollow filament drawn from them as well as honeycomb filament made from a number of hexagonal tubes joined together.

First, a search was made to find a commercial supplier of hexagonal glass tubing. Precision Electronic Glass Co., by altering their process somewhat, were able to extend the length of tube they could supply to about 18 inches. This is still quite short for use with the preform attenuation process, especially for making a long run under stable conditions such as are required for winding a complete hoop or short cylinder. Another distinct disadvantage, besides high cost, that these hexagonal tubes had was the relatively thick wall; that is, the degree of hollowness was too low (low ratio of I. D. to O. D.) and the corners were rounded on the exterior. Specifically, the manufactured tubing was 28 mm outside across the flats and 25 mm inside. This wall thickness of 1.5 mm is too great for fabricating preforms where walls may be doubled in making up honeycombs. The thick-walled tube was also difficult to draw down even singly into a hollow hexagonal filament while maintaining crisp corner detail.

In view of the unavailability of suitable hex tube stock, a process was developed wherein it was possible to draw 1.5 meter long hex tubes, 12.7 mm outside across the flats with a 0.9 mm wall and larger tubes, 2 meters long 25.8 mm across the flats and only 0.7 mm wall.

It was soon demonstrated that a round tube could be drawn over a hexagonal graphite plug to form hex tubing. However, making uniform wall thickness and straight, long, untwisted tubes took a great deal of refinement.

Basically, a modification of the shaped fiber forming process was used. The round tube was fed slowly down into a furnace with a thin horizontal hot zone. The tube was fed down over the hexagonal graphite plug which was supported so it extended vertically through the hot zone. The lower end of the tube was grasped beneath the furnace and connected by a cable to a constant torque device some distance beneath the furnace.

The first arrangement had the graphite plug supported by a water cooled brass plug while the water tubing in and out supplied the mechanical support. Thus, the maximum length of hex tubing that could be drawn was limited by the graphite plug support. In other words, the support is located where the stretched tubing accumulates rather than above the furnace where the unstretched feed tube starts out. Therefore, the plug support doubly limited the amount of tubing to be made.

The constant torque device was used to pull the tubing when a low enough viscosity (high enough temperature) was reached to achieve draw down. The use of the constant torque puller was essential to determining operating conditions for optimum hex tube forming. On the other hand, it permitted the drawing speed to vary even though the feed speed was constant. This speed change could be attributed to slight temperature variations changing viscosity and variations in friction on the plug caused by any slight misalignment. The result was wall thickness variation. A guide had to be installed to minimize any twisting.

Using the operating conditions established by these studies, a new apparatus was built with feeding and drawing run off the same motor. Through an adjustable ratio device the degree of draw down for suitable hexagon forming and the final wall thickness were controlled. The hexagonal graphite plug was supported from above the furnace which permitted much longer tubes to be produced. Some were made with over two times the length of the feed tube. Only after very careful alignment was it possible to make long straight tubes. Finally, complete lengths could be made with no more than 2 or 3 mm curvature.

Once successful operation was achieved, a stock of about 50 each of large and small hexagonal tubes was made up. These are being used as required to make up preforms and draw fibers in the shaped filament studies.

TABLE I

FILAMENT FORMING STUDIES SUMMARY

A Key to the Photomicrographs

Figure No.	Preform Cross Section (a)	Attenuation Conditions (b)	Filament Characteristics and Cross Section Dimensions (c)
5	composite equilateral triangle, spiral overlap	normal	side walls convexed triangles - .0025" walls - .0003"
6	equilateral triangle one thin wall	normal	arrowhead - .003" walls - .0007"
7	same as Fig. 5	normal furnace vacuum inside preform	concave walls Y-shaped - .008"
8	same as Fig. 6	normal furnace slight vacuum	arrowhead - .003" flat thick walls - .0005"
9	equilateral triangle plus rod at each corner	normal	good reduction of preform .0025" O.A. walls - .0002"
10	equilateral triangle spiral overlap, rod in each obtuse angle	normal furnace vacuum inside preform	lobed Y - .0035" wall - .00035"
11	hollow square	normal	good square - .002" walls - .0003"
12	hollow square	normal	thick walls, .0003", convex, thin walls, .0002", concave

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
13	same as Fig. 11	normal furnace vacuum	X with quadricorn hole .003" O. A.
14	same as Fig. 12	normal furnace vacuum	flat thick walls - .0005" concave thin - .0002" .003" O. A.
15	theta with thick wall bridge	normal	wrinkled diameter - .004" O.A. bridge - .0005" sheath - .0002"
16	thick walled X in thin walled tube	normal	swastika in sheath - .0025" O. A. X - .0006" sheath - .0002"
17	1/2" hollow hex tube	normal	thin walled (.0002") hollow hex - .0023" O. A.
18	1" thin walled hex tube	normal furnace vacuum	six toothed gear - .0015" O. A.
19	composite spiral hex	normal	thick walled hex - .003" O. A. circular inside - .002" I. D.
20	round tube	normal, top of tube sealed	very thin walled (<.0001") random size .0025" typical O. D.
21	3 concentric tubes spaced with tubes	normal, top of spacer tubes sealed	voids of spacer tubes exaggerated - .0055" O. D.
22	3 concentric tubes more spacers	normal, top of spacer tubes sealed	improved symmetry .0075" O. D.

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
23	hollow rectangle	normal	distortion approaching ellipse - .003" x .012" walls - .0003"
24	hollow rectangle 2 bridges	normal	minimum distortion - .0023" x .011" walls - .0003"
25	hollow rectangle 3 bridges	normal	crisp rectangular hollow fiber - .002" x .015" walls - .0004"
26	diamond	slight overheat	football shape .0033" x .006" walls - .0005"
27	hollow rectangle	slight overheat	hollow ellipse .002" x .0045" walls - .0004"
28	high ratio hollow rectangle	slight overheat	hollow ellipse .0017" x .0083" walls - .00035"
29	various diam. rods arranged in ellipse	marked overheat	fair elliptical cross section - .0025" x .0055"
30	solid rectangle	overheat	oval with flat sides .0013" x .003"
31	solid ellipse ground to shape	normal	solid ellipse .0007" x .0025"
32	6 round tubes in hexagonal arrangement	normal	perfect hex center scalloped .007" O.A.

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
33	same as Fig. 32	normal	enlargement of 32 .007" O.A.
34	hollow ellipse of 3 mm tubes	tubes sealed normal	hollow honeycomb ellipse .0035" x .013" .00055" walls of .0001" films
35	hollow ellipse of 3 mm tubes	tubes sealed normal	hollow honeycomb ellipse .0023" x .0045"
36	hollow ellipse of 3 mm tubes	tubes sealed normal	hollow honeycomb ellipse .0021" x .0046"
37	thick walled tube, thick walled diameter	normal	ellipse with solid major axis - .0025" x .0085" .0005" wall
38	hollow square of 3 mm tubes	sealed tubes normal	hollow honeycomb square .0045" x .0045" .0015" wall .0001" films
39	solid square of 3 mm tubes - outside plates	sealed tubes normal	solid square honeycomb exaggerated holes .006" x .006"
40	same as Fig. 39	same	enlargement of Fig. 39 .006" x .006"
41	solid hexagon of 3 mm tubes - outside plates	sealed tubes normal	solid hex honeycomb exaggerated holes .006" O.A.
42	high ratio rectangle of 3 mm tubes - outside plates	sealed tubes normal	honeycomb tape, holes near center stretched .020" x .003" O.A. .0006" holes

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
43	oval of 3 mm tubes no outside plates	sealed tubes normal	honeycomb, enlarged holes - thin walls .002" x .007"
44	sheet of glass with narrow strips on edge	normal	zipper-like cross section good reproduction of preform - .0015" x .011"
45	sheet of glass with 3 stacks of 3 rods on each face	very slight overheat	good mating surfaces .0015" x .004"
46	zig zag arrangement of rods	normal	corrugated tape .002" x .027"
47	zig zag arrangement of E-glass rods	normal	corrugated tape .0013" x .016"
48	letters NASA between plates	normal	good reproduction of intricate preform .010" x .002" O. A. each letter - .0015" x .002"
49	alternate large and small rods in line	normal	variation of corrugated tape - .0012" x .007"
50	alternate 1 large and 2 small rods in line	normal	mating corrugated tape .0033" x .026"
51	wide spaced 3 unit tape	very slight underheat	crisp corrugated tape .002" x .006" .0005" bridge
52	large rods spaced on sheet	slight overheat	corrugated tape variation .003" x .014"

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
53	five rods in row	slight overheat	corrugated tape variation .0017" x .0065"
54	sheet of glass with stacks of 3 tubes on each face	sealed tubes normal	corrugated tape variation .0045" x .016" .0001" films
55	sheet of glass with single tubes on each face	sealed tubes normal	corrugated tape variation .003" x .015" .0001" films
56	line of tubes	sealed tubes normal	hollow microtape .0006" x .015" .00015" wall
57	zig zag arrangement of tubes	sealed tubes normal	corrugated hollow micro-tape - .0015" x .010" .0001" wall
58	hollow rectangle of 3 mm tubes	sealed tubes normal	collapsed structure .002" x .015"
59	hollow rectangle of 3 mm tubes with bridges	sealed tubes normal	fair reproduction .0025" x .016"
60	hollow tubes in place of plate in Fig. 51	sealed tubes normal	extremely low density corrugated tape .002" x .0085"
61	variation of Fig. 60	sealed tubes normal	some distortion .004" x .021"
62	thin walled tubes between thin plates	sealed tubes normal	low density high stiffness .0035" x .018"

TABLE I (continued)

Figure No.	Preform Cross Section	Attenuation Conditions	Filament Characteristics and Cross Section Dimensions
63	row of tubes with plates top and bottom	sealed tubes normal	fair reproduction .003" x .017"
64	slit tube	normal	C-shaped .0035" O.A. .0003" wall
65	X of sheet with rods on outside edges	normal	X-shaped lobed filaments .007" O.A. .0003" wall
66	sheet with rods on edges	normal	I-shaped filaments .004" O.A. .0002" wall
67	sheet of glass	slightly underheated	flat thin microtape poor packing .016" x .0005"
68	sheet of glass	slightly underheated	flat thin microtape good packing .016" x .0005"

TABLE I - NOTES

(a) Preform Cross Section: description of preform assembly

(b) Attenuation Conditions: conditions within furnace and preform

1. normal - conditions as described under Experimental Procedures. Temperature and gradient to form accurate cross section reproduction.

2. vacuum inside preform - vacuum applied to top of hollow preform.

3. overheat - sufficient heat to cause partial fusion with some loss of detail.

4. underheat - lower temperature to accentuate crispness.

5. tubes sealed - causes slight internal pressure due to heating of entrapped air.

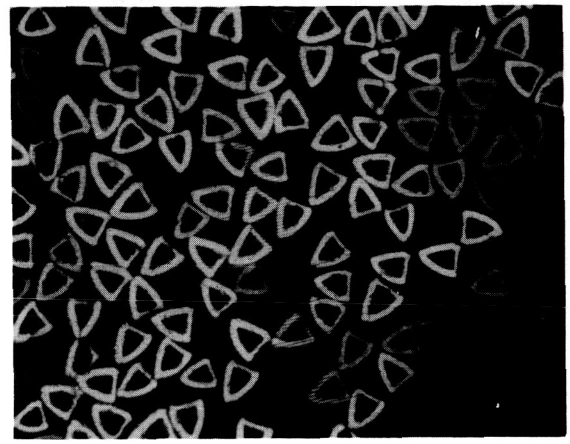
(c) Filament Characteristics and Cross Section Dimensions: Capsule description of filament cross section and dimensions.

SCALE = .010"

CROSS SECTION OF HOLLOW TRIANGULAR FIBER

FIGURE 5

SCALE = .010"



TRIANGULAR FIBERS - ONE THIN WALL

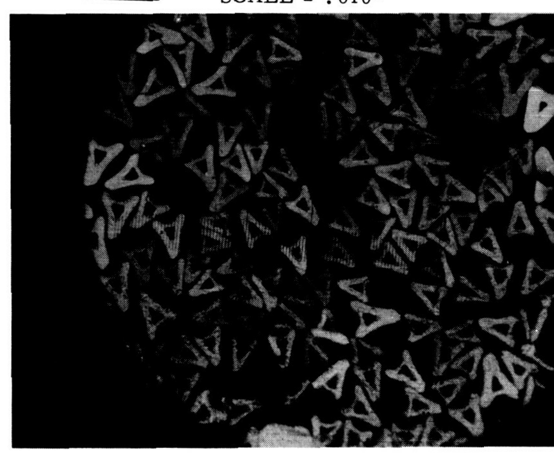
FIGURE 6

SCALE = .010"

TRIANGULAR FIBERS WITH VACUUM

FIGURE 7

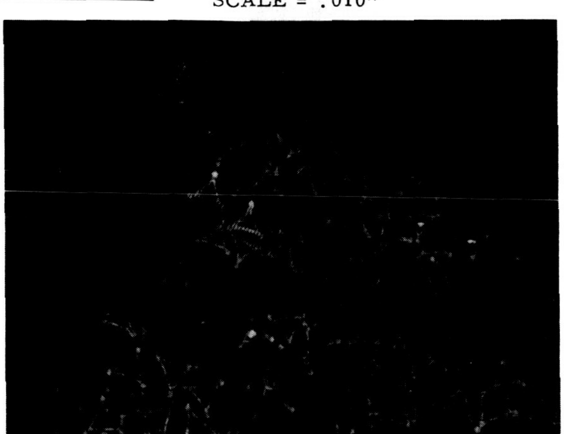
SCALE = .010"



TRIANGULAR FIBERS - ONE THIN WALL - VACUUM

FIGURE 8

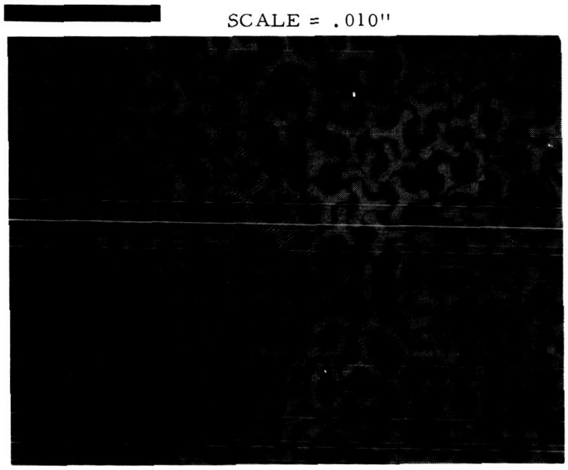
SCALE = .010"



TRIANGULAR FIBERS WITH HEAVY CORNERS

FIGURE 9

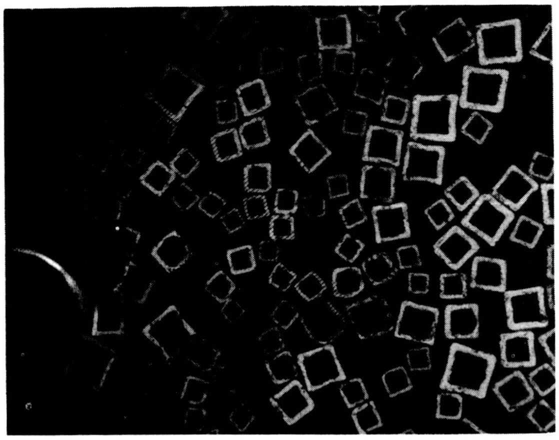
SCALE = .010"



VARIATION OF FIGURE 9 - VACUUM

FIGURE 10

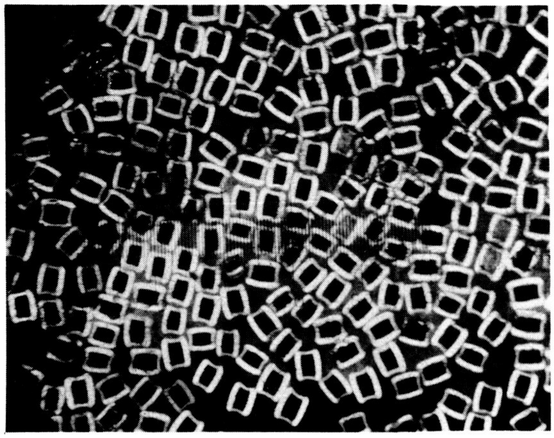
SCALE = .010"



SQUARE FILAMENTS

FIGURE 11

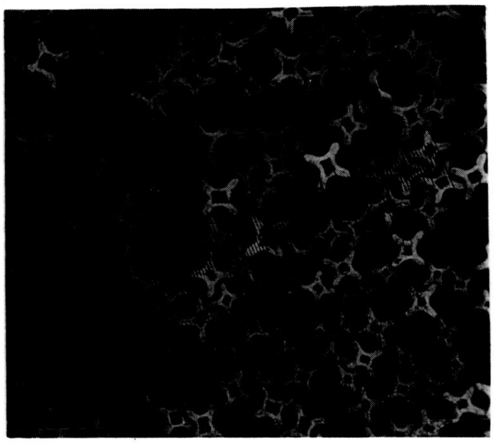
SCALE = .010"



SQUARE FILAMENTS - ALTERNATE THIN WALLS

FIGURE 12

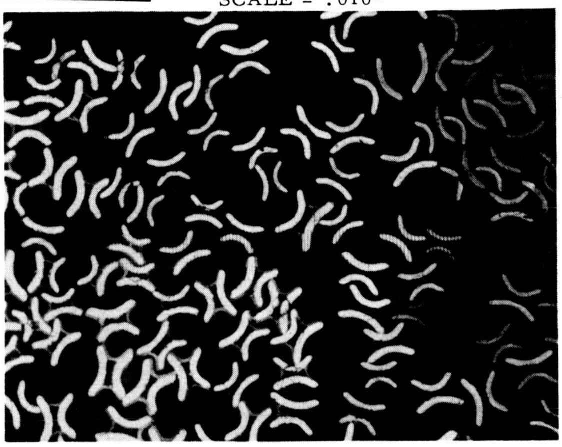
SCALE = .010"



SAME AS FIGURE 11 - VACUUM

FIGURE 13

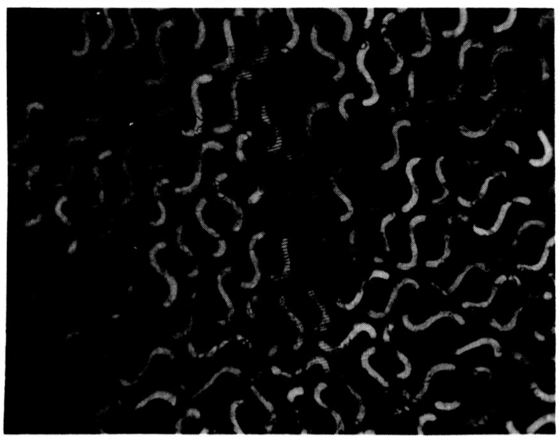
SCALE = .010"



SAME AS FIGURE 12 - VACUUM

FIGURE 14

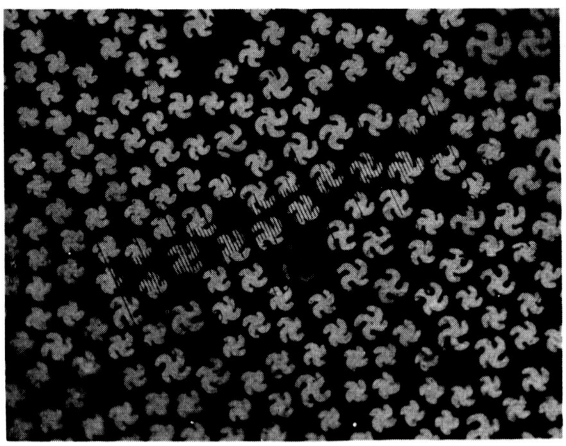
SCALE = .010



THIN WALLED TUBE - THICK SHEET DIAMETER

FIGURE 15

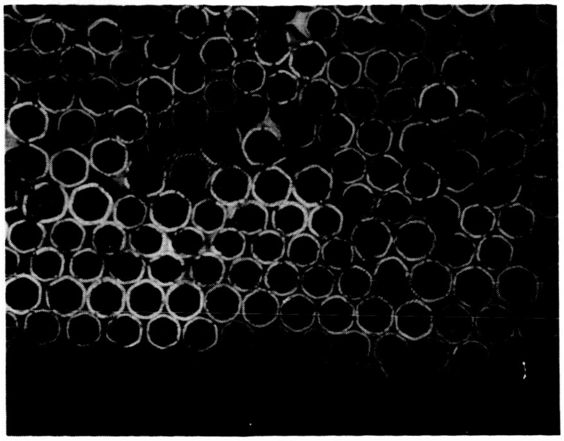
SCALE = .010"



FIBER FROM CROSS IN TUBE

FIGURE 16

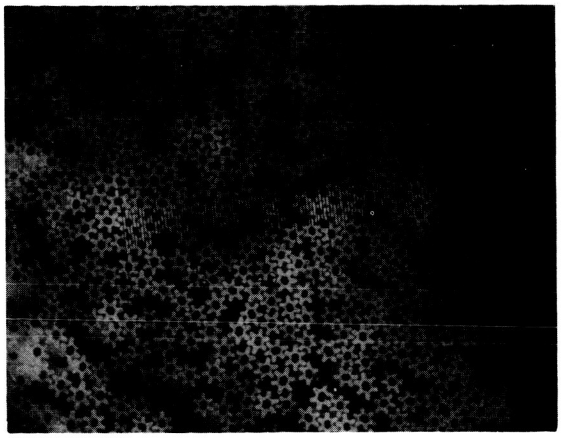
SCALE = .010"



WELL PACKED HOLLOW HEX FILAMENTS

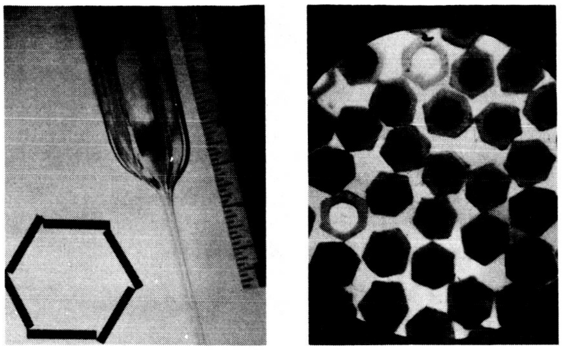
FIGURE 17

SCALE = .010"



VACUUM COLLAPSED HEX FIBERS

FIGURE 18

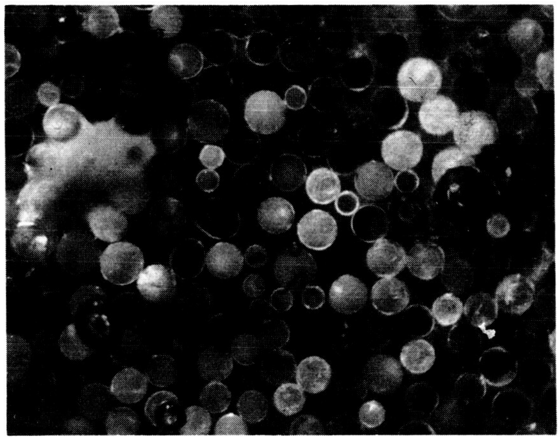


SCALE = .010"

SIX PIECE HEX PREFORM AND FIBERS

FIGURE 19

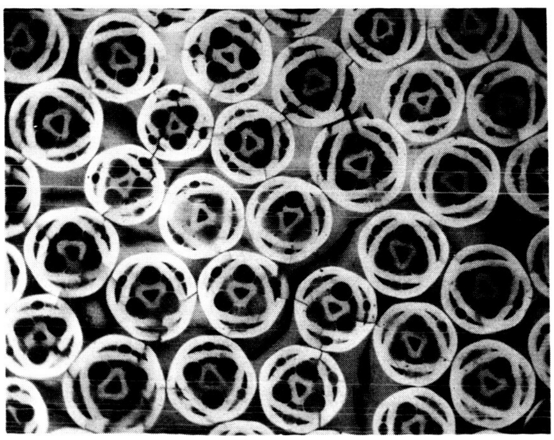
SCALE = .010"



HOLLOW FIBERS - WALL = 1 MICRON

FIGURE 20

SCALE = .010"



COMPLEX EQUIDIMENSIONAL FILAMENT

FIGURE 21

SCALE = .010"

VARIATION OF FIGURE 21

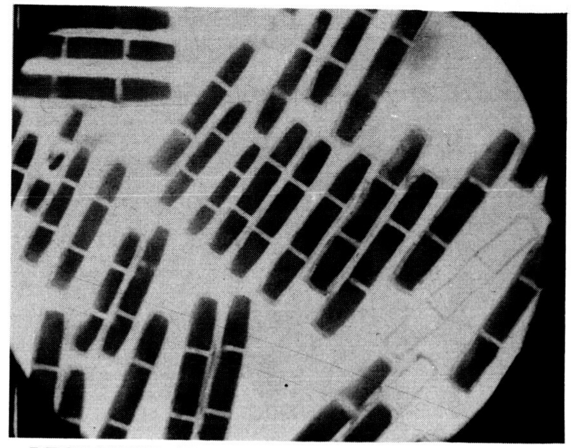
FIGURE 22

SCALE = .010"

NEAR-ELLIPTICAL HOLLOW FILAMENTS

FIGURE 23

SCALE = .010"



RECTANGULAR FILAMENTS - TWO BRIDGES

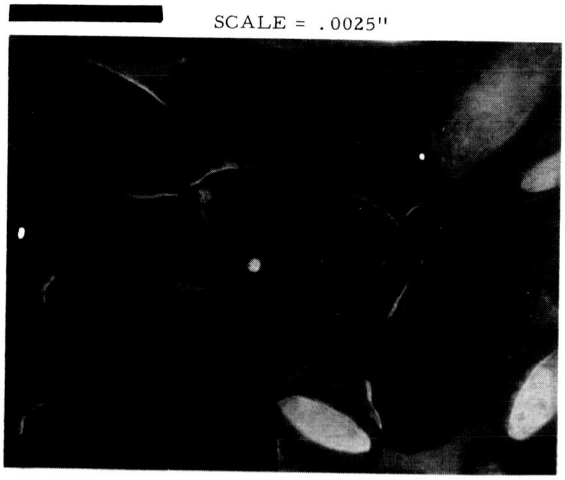
FIGURE 24

SCALE = .010"

RECTANGULAR FILAMENTS - THREE BRIDGES

FIGURE 25

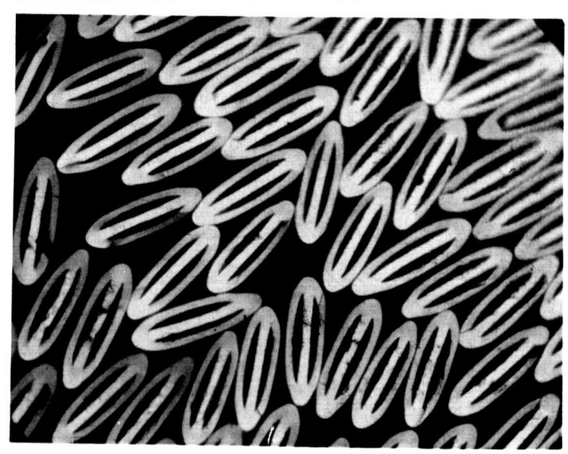
SCALE = .0025"



HOLLOW ELLIPSE WITH HONEYCOMB WALL

FIGURE 36

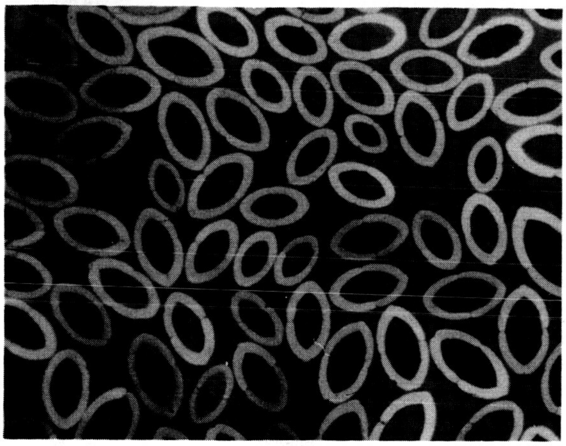
SCALE = .010"



HOLLOW ELLIPSE - SOLID MAJOR AXIS

FIGURE 37

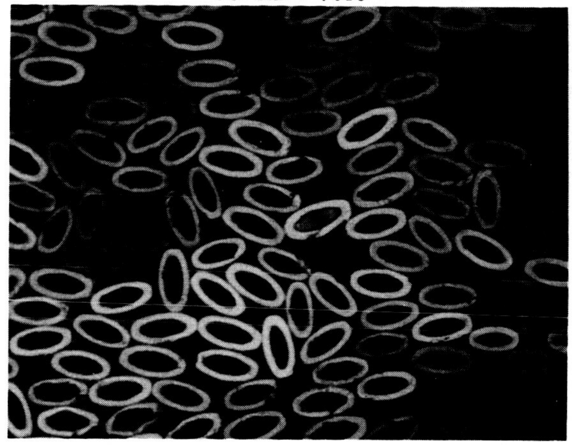
SCALE = .010"



ELLIPSES FROM DIAMONDS

FIGURE 26

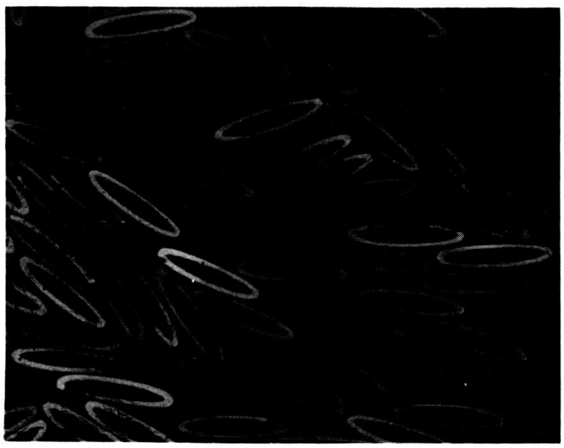
SCALE = .010"



HOLLOW ELLIPTICAL FIBERS

FIGURE 27

SCALE = .010"



HOLLOW ELLIPTICAL FIBERS 5:1

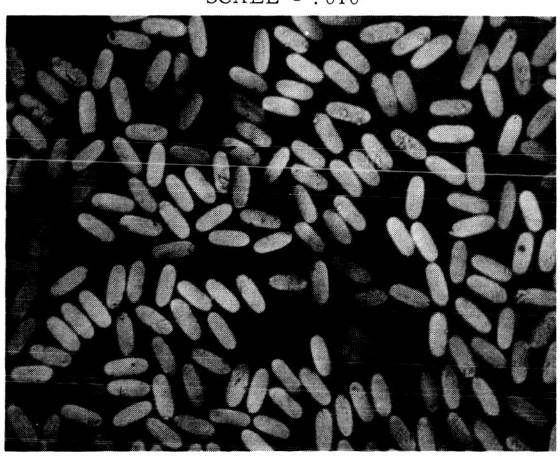
FIGURE 28

SCALE = .010"

SOLID ELLIPTICAL FILAMENTS

FIGURE 29

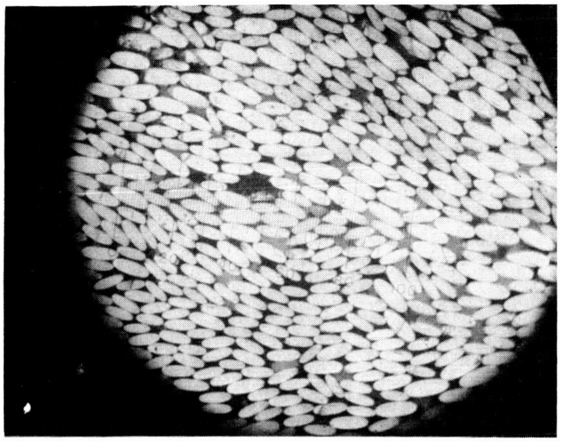
SCALE = .010"



FLAT OVALS FROM RECTANGLES

FIGURE 30

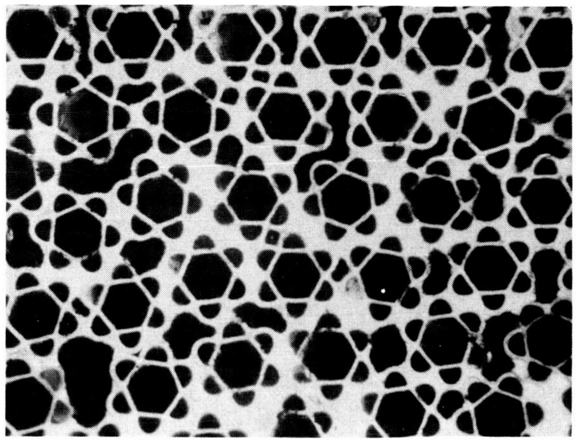
SCALE = .010"



SOLID ELLIPTICAL FIBERS

FIGURE 31

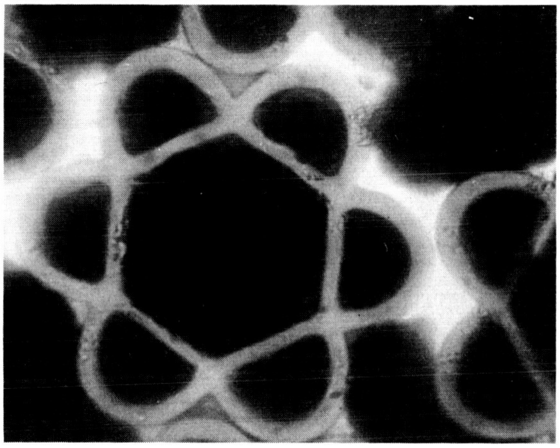
SCALE = .010"



SIX TUBE FIBERS - NOTE CENTER HEX

FIGURE 32

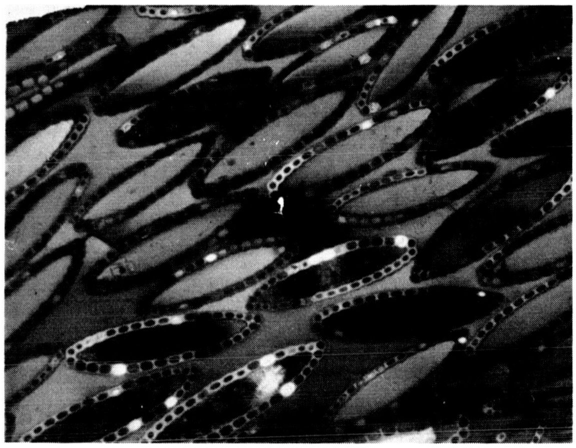
SCALE = .0025"



SINGLE SIX TUBE FIBER

FIGURE 33

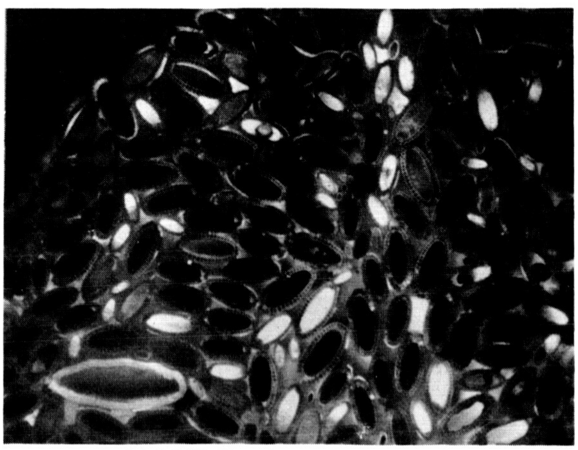
SCALE = .010"



HOLLOW ELLIPSES WITH HOLLOW WALLS

FIGURE 34

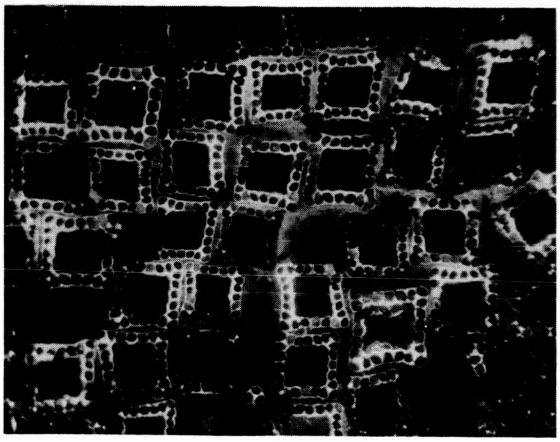
SCALE = .010"



HOLLOW ELLIPSES FROM TUBES

FIGURE 35

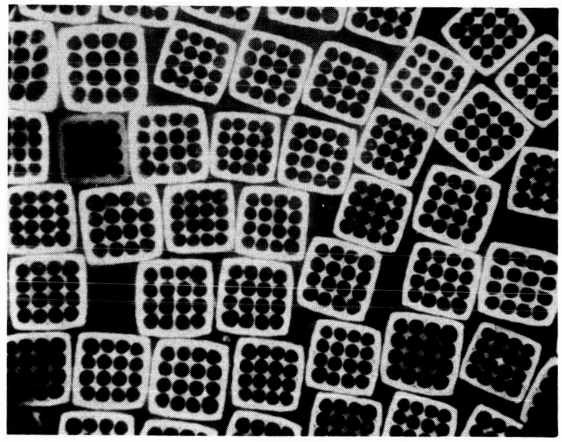
SCALE = .010"



HOLLOW SQUARE FIBERS - SEGMENTED WALLS

FIGURE 38

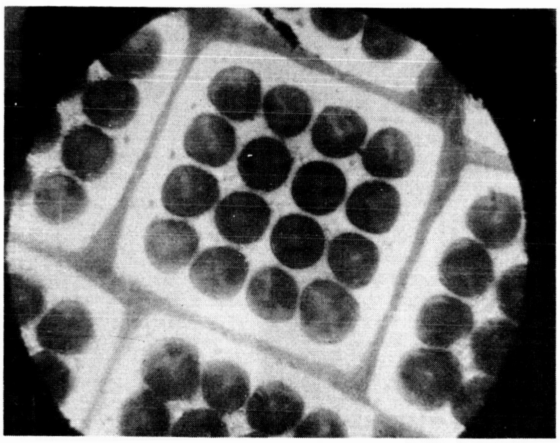
SCALE = .010"



SQUARE HONEYCOMB FILAMENT

FIGURE 39

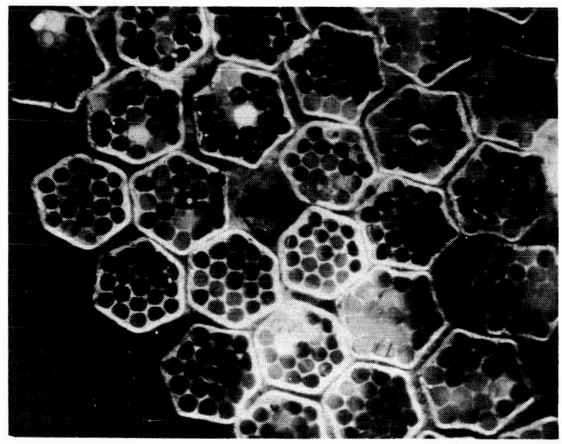
SCALE = .010"



SAME FILAMENT ENLARGED

FIGURE 40

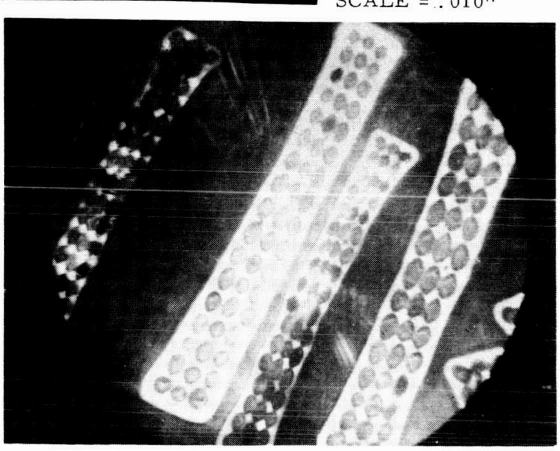
SCALE = .010"



HEXAGONAL HONEYCOMB FILAMENT

FIGURE 41

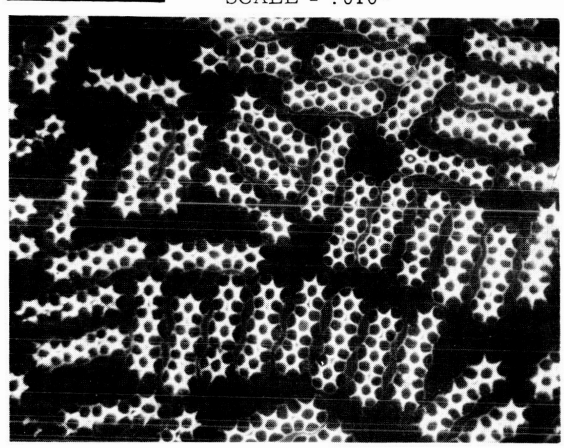
SCALE = .010"



HONEYCOMB TAPE

FIGURE 42

SCALE = .010"



HONEYCOMB FROM ROUND TUBES

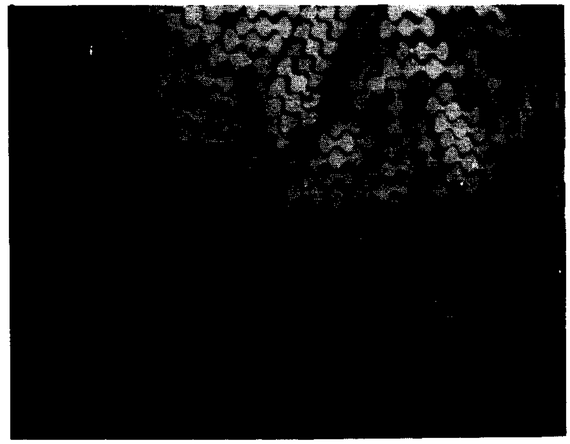
FIGURE 43

SCALE = .010"

CORRUGATED TAPE

FIGURE 44

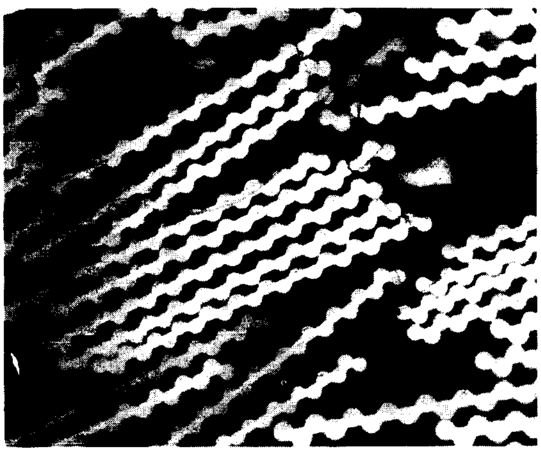
SCALE = .010"



NESTED CORRUGATED TAPE

FIGURE 45

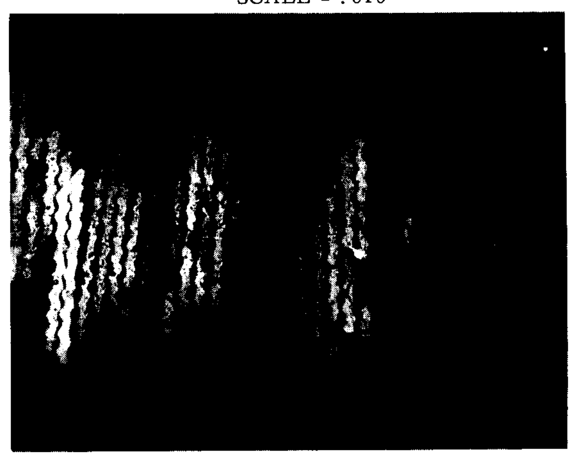
SCALE = .010"



CORRUGATED TAPE

FIGURE 46

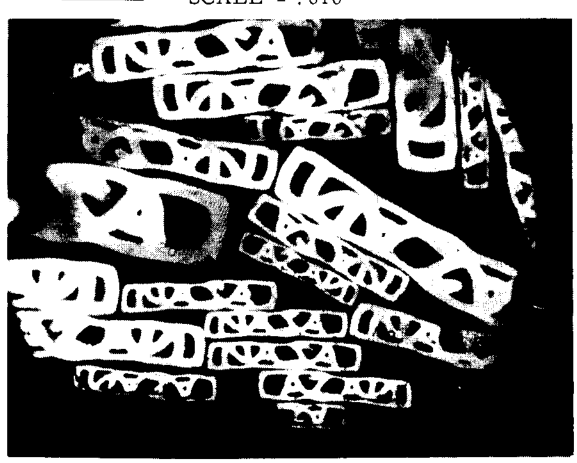
SCALE = .010"



CORRUGATED TAPE FROM ROUND RODS

FIGURE 47

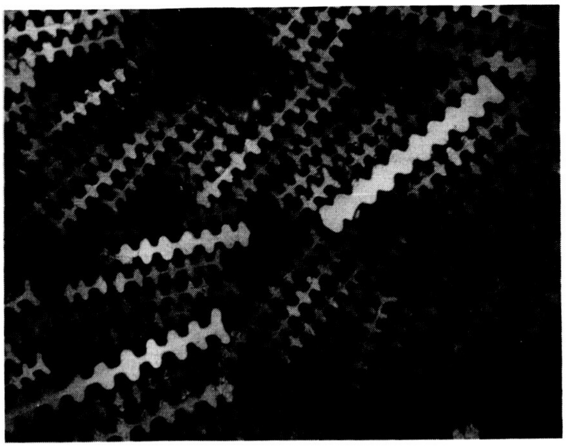
SCALE = .010"



"NASA" IN A FILAMENT

FIGURE 48

SCALE = .010"



CORRUGATED TAPE

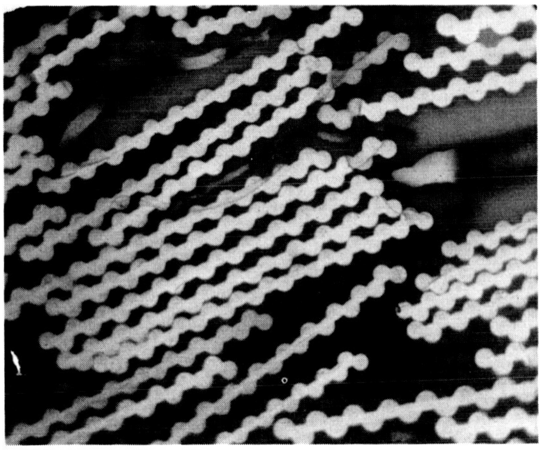
FIGURE 44

SCALE = .010"

NESTED CORRUGATED TAPE

FIGURE 45

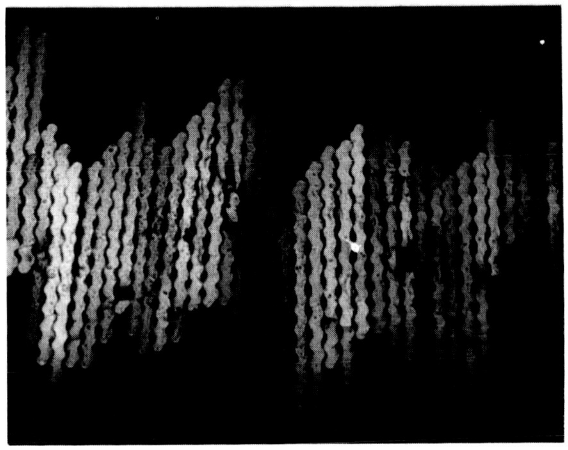
SCALE = .010"



CORRUGATED TAPE

FIGURE 46

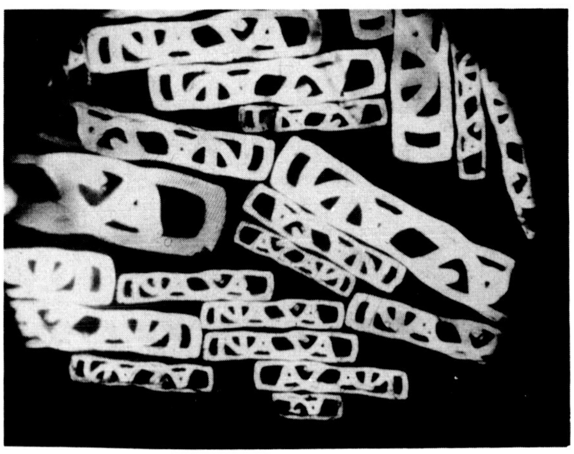
SCALE = .010"



CORRUGATED TAPE FROM ROUND RODS

FIGURE 47

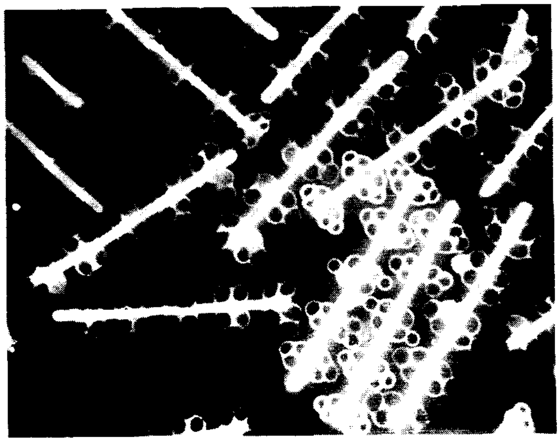
SCALE = .010"



"NASA" IN A FILAMENT

FIGURE 48

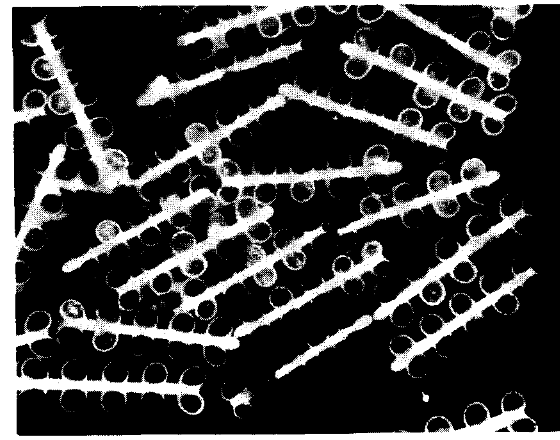
SCALE = .010"



CORRUGATED TAPE FROM PLATE AND TUBES

FIGURE 54

SCALE = .010"



SIMPLER VERSION OF FIGURE 54

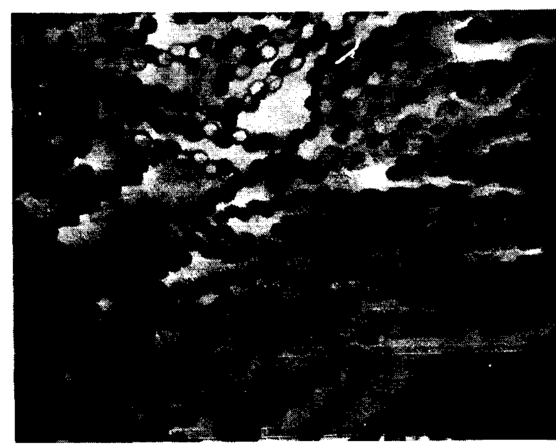
FIGURE 55

SCALE = .010"

TAPE FROM TUBES

FIGURE 56

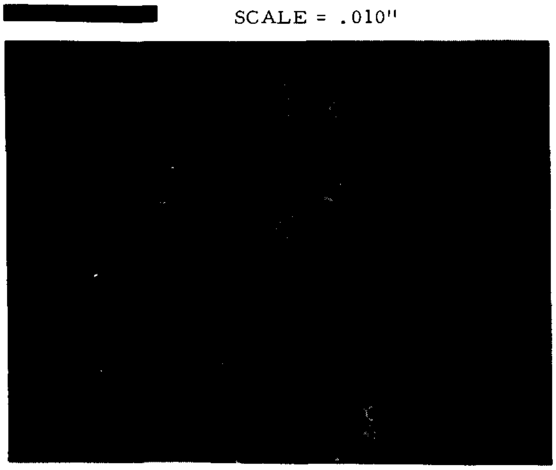
SCALE = .010"



CORRUGATED MICROTAPE FROM TUBES

FIGURE 57

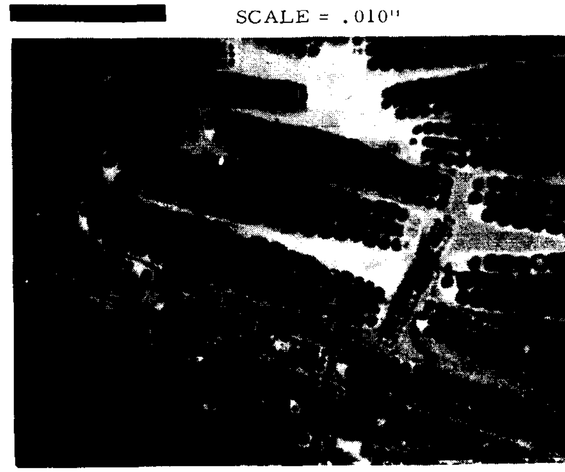
SCALE = .010"



COLLAPSED RECTANGULAR HONEYCOMB

FIGURE 58

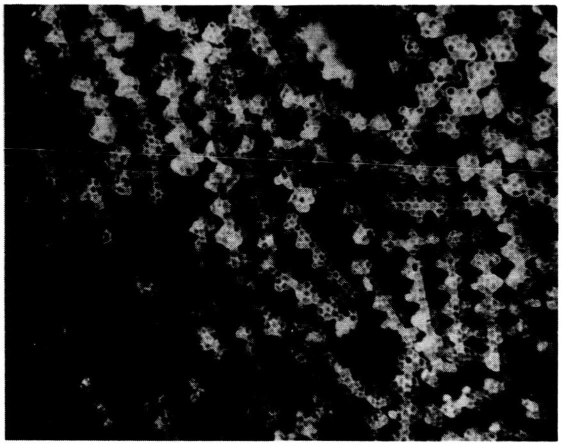
SCALE = .010"



RECTANGULAR HONEYCOMB WITH BRIDGES

FIGURE 59

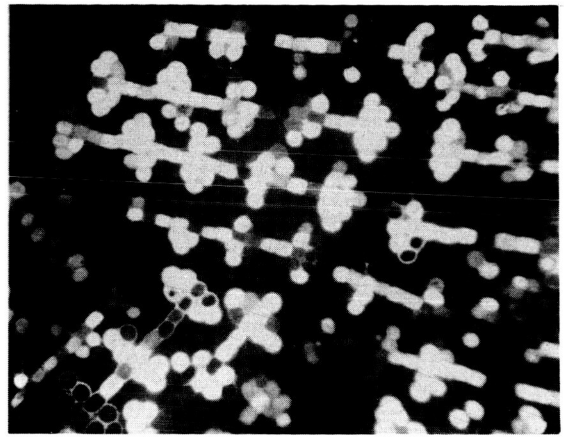
SCALE = .010"



TUBULAR ARRAY CORRUGATED TAPE

FIGURE 60

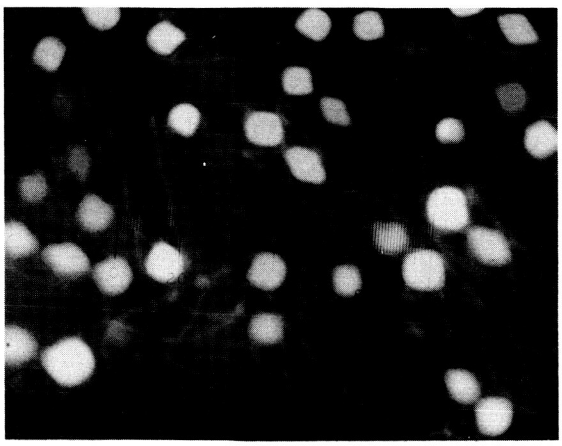
SCALE = .010"



VARIATION OF FIGURE 60

FIGURE 61

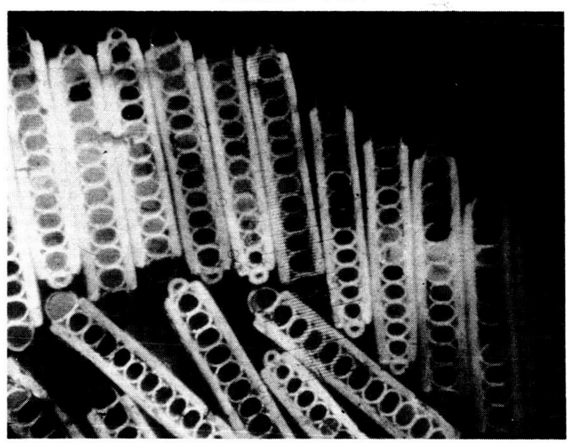
SCALE = .010"



TRUSS-LIKE HOLLOW TAPE

FIGURE 62

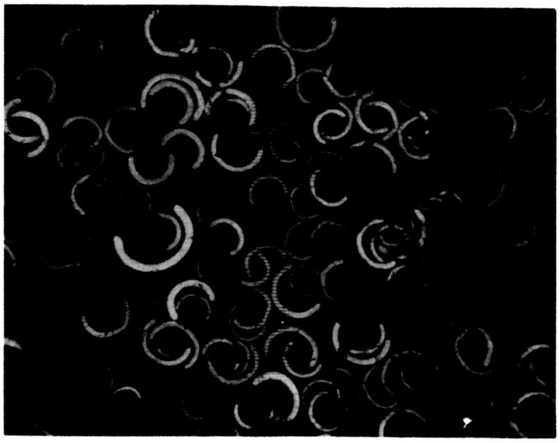
SCALE = .010"



HOLLOW TAPE - PLATES OUTSIDE TUBES

FIGURE 63

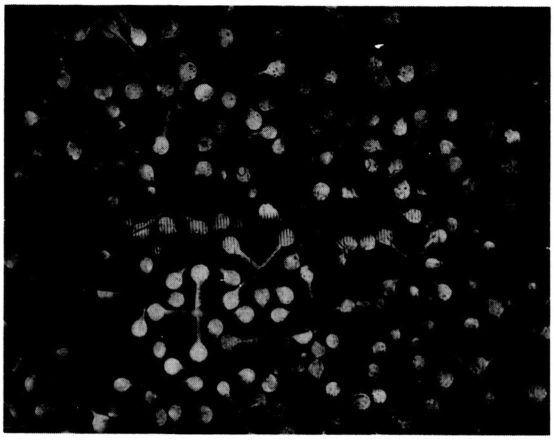
SCALE = .010"



C-SHAPED FILAMENTS

FIGURE 64

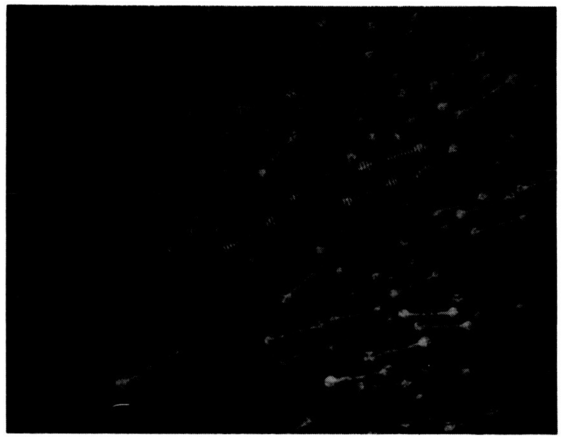
SCALE = .010"



X-SHAPED FILAMENTS

FIGURE 65

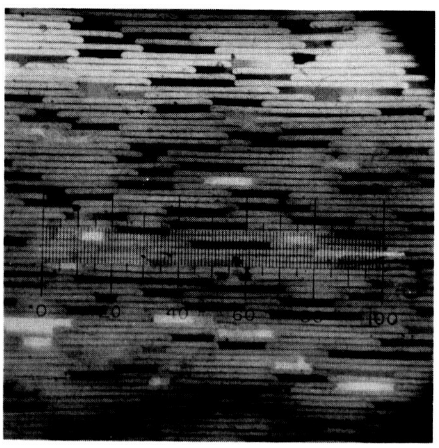
SCALE = .010"



I-SHAPED FILAMENTS

FIGURE 66

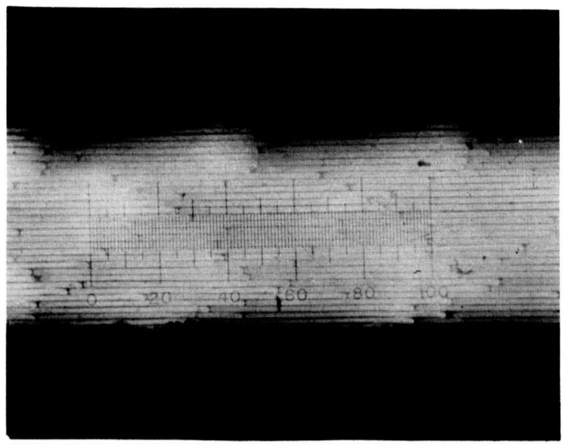
SCALE = .010"



IMPERFECTLY PLACED MICROTAPE

FIGURE 67

SCALE = .010"



WELL PLACED MICROTAPE

FIGURE 68

IV. DISCUSSION

Several general principles govern the forming operation, whatever size or shape filament is being produced. As a hollow, shaped fiber is being drawn, the cross section is reduced about one hundredfold while the attenuation speed is about 10,000 times the feed speed. In studying what happens during the drawing operation, the first reaction is to look at the horizontal flow of glass from the preform to the filament. Similarities of shape make this seem logical and the cross sections of the preform and the filament are readily available for examination and comparison. However, it is important to consider that the major flow of material during attenuation was at right angles to the cross section being examined. This longitudinal or vertical flow can be the ruling factor in the resultant shape of the filament.

Glass is a material whose viscosity is extremely high even at forming temperatures. Common processes, such as blowing a bottle, fabricate glasses at 1,000 to 10,000 poises. In the preform attenuation process, the glass probably does not reach a viscosity much lower than 100,000 poises. At that point the viscosity changes about 1% per degree fahrenheit, a property which necessitates good temperature control in the fiber forming process.

At lower temperatures, the rate of increase of viscosity with lowering temperature is much greater. The viscosity at 1,000°F would be about

10^{13} poises. While the viscosity changes prominently with temperature, the surface tension changes little. These considerations all have their bearing on the analysis of the behavior of shaped filaments during forming. They also emphasize how accurately one must control and coordinate furnace temperature, vertical temperature gradient, horizontal temperature uniformity, rate of feed and rate of fiber take-up.

When the actual heating rate of the glass in the attenuating preform is considered, it becomes evident that two other factors effect the temperature gradient curve within the glass itself. First, because the preform is attenuating, the glass is moving downward at an ever increasing rate of speed. Thus although the temperature in the furnace is increasing, the time for the glass to be heated is rapidly decreasing. The other somewhat compensating though smaller factor is the fact that the mass of material to be heated is decreasing as attenuation proceeds.

The hexagonal tube drawing described earlier also is done at unusually high viscosity. Temperature control is again an important function in the forming of hex tubes with flat sides and crisp corner detail. The viscosity of the glass causes the side walls of the tube to collapse onto the graphite plug during draw down. Too high a temperature permits the viscosity to drop too low. Then size and shape both deteriorate because intimate contact between the glass and the graphite plug is lost.

Although no strength studies of the shaped fibers have been made to date, it is well-known that a glass filament exhibits higher tensile strength than does a glass rod of the same glass formulation. As the filament becomes smaller in diameter, the strength continues to rise until a plane of strength is reached. For example, a soda-lime glass formulation may have a tensile strength as an untreated 0.125" dia. rod of 10,000 - 20,000 psi. Drawing to a filament 0.0004" dia. increases the strength to 60,000 - 90,000 psi. Initial and filament strengths can be further improved by acid polishing the surface with a mixture of hydrofluoric and mineral acids. It is reasonable to postulate then that a thin glass film with a surface area to volume relationship approaching that of a filament 0.0004" thick would have a strength of the same order of magnitude as the filament.

V. CONCLUSIONS AND RECOMMENDATIONS

Conclusions

During the course of this first phase of the program, it was shown that a large number of different cross sectional shaped glass filaments could be drawn. This required learning how to control the complex process while dealing with extensive elongation, about 10,000 times, and substantial cross section reduction, approximately 100 times; in the face of surface tension, varying high viscosity, regulation of temperature, preform internal pressure, feed rate, and take-up rate. (A summary of the results of shaped fiber forming is found in Table I, page 27. This table serves as a guide to the many photomicrographs which show the variety of shapes that may be formed.)

A degree of skill has been reached where, given a specific complex filament cross section, a preform can be designed to yield that filament although they may not be identical to one another.

A satisfactory method was developed for drawing long, straight, thin-walled hexagonal tubes.

The precision winding technique was improved to the state where flat tape-like filaments can be wound into closely packed structures.

It was found difficult to draw filaments from large preforms; for example, 4" x 4", even with inside heaters to supplement the more normal outside heaters. Complex preforms, however, if kept small (about 1-1/2" in diameter) can be drawn into filaments.

Recommendations

Although there is still a great deal of work to be done forming filaments of various shapes, it seems important at this time to examine in detail the properties of shaped filaments and wound structures made from them. Of special interest with shaped fibers should be the transverse moduli of these structures. Not only is there controlled, oriented hollowness and stiffness but many of the shaped filaments have peculiar interlocking surfaces which should be capable of displaying stiffness not found in ordinary glass fiber composites.

A more exhaustive study should be made of the dynamics of forming these shaped filaments. Preliminary tests with a high temperature grid fused to the preform are most instructive. More precise temperature profile information would assist in this study and facilitate designing furnaces to work with certain preform shapes.

Other problems worthy of attention include: the comparison of properties of filaments made from cemented preforms with those from the same shape monolithic preforms, the precision winding of other shapes than tape such as equidimensional fiber and, eventually, a comparison of properties versus composition. The preform attenuation process is radically different from organic fiber forming from spinnerettes. It seems plausible that organic fibers with unusual, useful properties could be made by variations of this process.

The potentialities of shaped filaments are so broad they can only be partially realized at this time. Obviously the hollow, well-braced filaments can contribute to building structures with a very high stiffness-to-weight ratio. The fibers with interlocking surfaces should make high shear strength composites. However, a study by engineers and scientists of different backgrounds of the variety of shapes possible should turn up new, yet unthought of, applications for each discipline.

APPENDIX I

STUDY OF COMPONENT INTERACTION AND INITIATION AND CONTINUATION OF FAILURE MECHANISM

Introduction

Many problems are present in filament wound composites. In order to obtain a circular filament of highest strength, it has been necessary to draw the filament from a selected glass formulation (E, for example) down to a relatively small diameter filament (0.0005" or less). It is impossible to handle these filaments individually in textile operations because the strength of individual filaments is so low. When two hundred or more of these filaments are simultaneously and continuously formed from the orifices of a bushing as in the hot melt process, it has been impossible to wind them so that each filament is under precisely the same tension. A bonding resin with one twentieth the modulus must transfer the stress between the unequally strained filaments. Another difficulty also arises from the unequal tensioning of the individual filaments. When these filaments are wet with resin, tensioned and wound as a strand into place on a mandrel, the individual filaments have slightly different lengths. Therefore, they do not react equally to the tensile force applied to the strand and do not form an equally spaced geometric pattern within the matrix. As a result, stress concentrations can occur at specific submicroscopic regions of the matrix where a relatively high loading is transferred between two filaments through a polymerized resin layer of exceedingly small cross section, essentially a line.

Discussion

A start was made on this work during the final three months of this program in the hope that, in a continuation of the overall program, considerable light could be shed on microscopic and submicroscopic stress concentrations in composite materials in general, and on the glass-resin interaction in particular.

There is little or no information in the literature regarding the submicroscopic effects on either side of the glass-resin contact area resulting from a strong bond capable of transferring stress between the two physically dissimilar materials. The glass or other ceramic filament is a relatively high (Young's) modulus material in the order of 10, 000, 000 psi or greater while the polymerized resin in contact with it has a modulus of less than 1, 000, 000 psi, usually in the range of 300, 000 to 600, 000 psi. These materials must maintain intimate contact when subjected to internal stresses (shrinkage, differences in thermal expansion, fiber spacing, etc.) or when subjected to externally applied loads. The maintenance of intimate contact is preserved by applying a glass-resin coupling agent to the glass surface either in forming the composite by addition to the resin or by other means. The resin reacts chemically with the coupling agent and the coupling agent presumably reacts with the hydroxyls on the glass surface to form stress-transferring bonds.

The contact layer (about one molecule thick of a polymerized resin) that is well bonded to a glass surface must, in order to maintain contact with that surface, physically react to stress in almost precisely the same manner as glass with which it is in intimate contact. In successive molecular layers of resin, the restraining effect of the glass surface becomes less, decreasing at a rate at least as great as the square of the distance from that surface. Depending upon the differential modulus, glass to resin and the packing density and orientation of the filaments, the restraining effect of the glass may affect the physical properties of the resin from the glass surface to an estimated minimum of 0.2 μ to a maximum in the order of 1 μ from that surface. This can only be estimated because measurements have not been made of this phenomena.

For convenience, the effect of the glass on the resin has been called "The Restrained Resin Layer Theory". The effects of the restraint of the glass on this resin layer are more than simply those involved with transfer of stress. For example, thermosetting resins swell on absorbing moisture. Obviously, the resin that is restrained by the glass surface cannot absorb water to the degree that unrestrained resin absorbs water. In fact, the resin within one molecular layer is likely to absorb only a quantity of moisture sufficient to fill lattice holes having dimensions greater than the water molecules.

Another effect of the glass surface on the resin in this "restrained resin layer" is to increase the energy required to propagate a flaw in that resin. Some investigations have indicated that cured resins are composed of micelles (little lumps of a higher modulus material surrounded by a lower modulus, more friable material). In a non-restrained resin, the micelle on either side of a growing flaw may move to allow the flaw to grow. However, if only one of these two micelles is free to move, as would be the case in a resin in the restrained resin layer, it is likely to take a magnitude or more increase in energy.

As this work develops in a continuation program, not only the presence and magnitude of a restrained resin layer will be studied but the relationship between the effects of this layer and the initiation and propagation of failure producing flaws.

Experimental Work

One of the easiest ways to prove that this restraining layer does exist is to measure the effect of an externally-applied compressive load on the capacity of a cured resin casting to absorb water.

In order to apply the information obtained to composite structures, the effect of internal stress and changes in the geometry of the matrix will need to be understood. Samples have been made and some are now under test.

The castings in this initial work are of two sizes: a relatively stress-free size, 1/8" dia. by 1/4" long and a larger size 1/2" x 1/2" x 1" long. In the larger size, two types were made. One is without holes while the other has 16 holes, 0.041" in diameter, spaced on 0.01" centers. The cylindrical shapes were cast in Teflon tubing that was snugly drawn inside a copper tube. The rectangular forms were cast inside hollow square tubing with inside dimensions 1/2" x 1/2". Copper wires, 0.041" in diameter were pulled straight and parallel between two dimensional drilled end plates. All surfaces contacting resin were coated with parting agent. After the resin had been cured, the copper wires were stretched to release them, cut close to one side of the mold and withdrawn. After the piece was removed from the mold, the ends were machined to smooth parallel surfaces.

Resins of varying formulations, shear strength and water absorption were chosen (Table II). The cure cycle used was sufficient to give a full, reproducible cure.

Figure 69 shows in schematic form the method by which the samples were loaded and subjected to water immersion at room temperature.

Figure 70 shows a picture of the apparatus with 5 of the 6 stations under load. Figure 71 shows a rectangular solid casting under pressure with a stream of water flowing over it. Figure 72 shows a small cylindrical casting under the same condition. Three loading conditions were tried: no load, 15 lb. load and 30 lb. load. Only the small cylinders were tested under all three conditions.

Results

Since only six samples are tested under load at one time and it takes five of the smaller samples under test to obtain a statistical weight difference, only one set of preliminary values are available (Table III). This resin system has relatively low water absorption properties and good shear properties. (ASTM D-732 of 10,000 psi). From this meager data, it appears that when a threshold external stress is exceeded, there is a reduced amount of moisture absorbed. As external stress was applied, the resin immediately reacted (351 psi loading) by showing a marked reduction (33%) in the water it would absorb. In a piece with "locked in" internal stresses, the equilibrium moisture content may be greatly reduced also. When holes are cast into the larger specimens, the "locked in" stresses are greatly reduced. The larger casting subdivided by the holes absorbed the same quantity of water (0.67%) as the smaller castings under a "no load" condition. However, in contrast to the over 3700 psi needed to reduce the water absorption of the small cylinders, 400 psi produced a marked reduction. The geometry of the resin around the hole as an annular layer may provide the answer.

Very preliminary results of the 0300/MPDA system indicate that it may be too water-sensitive to study at this time.

Preliminary Conclusion - Failure Mechanism Study

Evidence gained to date with an epoxy resin of low moisture absorption supports the thesis that the amount of moisture a thermoset polymer will absorb depends upon the degree it can swell. Any factor that restricts this swelling will reduce the moisture the polymer can absorb. Such factors are: internal stress, external loading and polymer shape. All of these elements are present in a glass-resin composite. Since there are indications of a relationship between total stress and water absorption in submicroscopic regions, it may be possible to use radioactively tagged water to trace the variation in quantity of water absorbed in the restrained resin layer and thereby measure the stress variation in that layer.

TABLE II

Moisture Absorption Studies on Cast Resins

Resin Formulations to be Studied

1. 100 pts Epon 828, 91 pts methyl nadic anhydride, 0.6 pts N,N benzyl dimethyl amine

2. 100 pts Epon 828, 41 pts methyl nadic anhydride, 0.6 pts N,N benzyl dimethyl amine

3. 100 pts ERRA 0300, 28.3 m-phenylene diamine

4. 100 pts P-43, 10 pts styrene, 1% Bz_2O_2

5. 100 pts IC-312 polyester, 1% Bz_2O_2

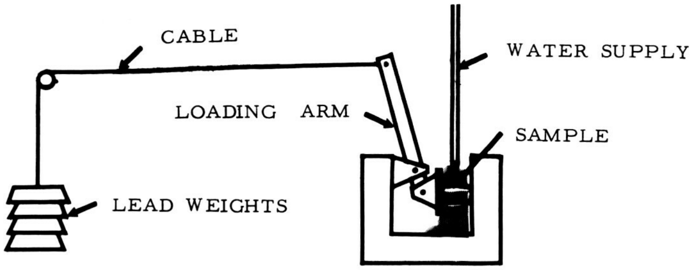
Cure Cycles Required

Formulations 1 and 2	30 min @ 150°F 30 min @ 200°F 15 min @ 250°F some samples gradually brought to 350°F for 4 hrs.
Formulation 3	30 min @ 150°F 30 min @ 200°F 30 min @ 250°F 180 min @ 300°F
Formulations 4 and 5	30 min @ 150°F 30 min @ 200°F 180 min @ 250°F

TABLE III

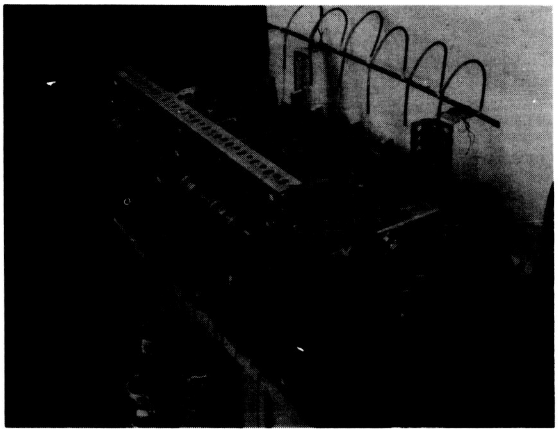
Castings: Water Absorption Data

100 pts E-828/91 pts MNA/0.6 pts BDMA Resin	
Small castings - 1/4" long x 1/8" dia.	**% Increase**
5 days soak under 7300 psi	0.17
equilibrium soak under 7300 psi	0.25
5 days soak under 3650 psi	0.56
equilibrium soak under 3650 psi	0.67
5 days soak under no load	0.56
equilibrium soak under no load	0.68
Large castings - 1" x 1/2" x 1/2"	
No holes - 351 psi load	
(% Compaction 0.19%)	
4 days soak	0.05
equilibrium	0.20
No Holes - No Load	
4 days soak	0.14
equilibrium	0.30
Holes - 402 psi load	
(% Compaction 0.33%)	
4 days soak	0.28
equilibrium	0.52
Holes - No Load	
4 days soak	0.32
equilibrium	0.67



METHOD OF LOADING AND SOAKING RESIN CASTINGS

FIGURE 69

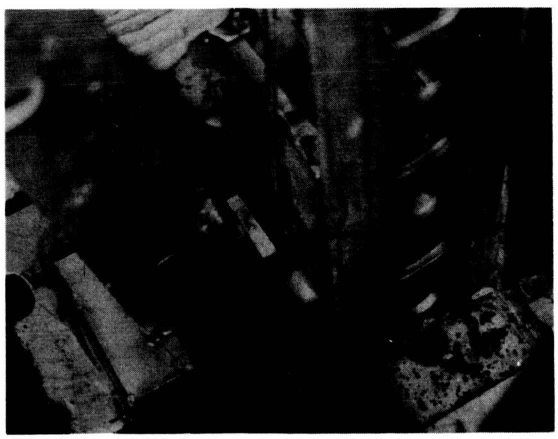


VIEW OF LOADING AND SOAKING APPARATUS

FIGURE 70

CLOSE-UP OF LARGE CASTING
UNDER LOAD AND WATER

FIGURE 71



CLOSE - UP OF SMALL CASTING
UNDER LOAD AND WATER

FIGURE 72

APPENDIX II

HISTORICAL NOTES

The art of drawing glass filaments was practiced by the ancient Egyptians in the New Kingdom times dating from about 1600 B.C.(5). These filaments which had irregular diameters in the order of twenty times our present day glass filaments were drawn from rods of glass reheated on one end and attenuated. The rods were made by turning a hot ductile lump of crude silica-alkali glass between metal rods until the lump had elongated to a cylinder with the thickness of a pencil. Glass filaments drawn from these rods in gay colors were wound in spirals around the walls or were embedded in the metal of vases. The technique of forming glass filaments from the heated end of a rod was highly refined as an operation by the Venetian bead manufacturers in the 16th to 18th centuries although the intention to use glass filaments in textiles was not evident.

The method developed has been described as follows (6): "The 'Glass Spinning Apparatus' consisted of a narrow rimmed reel (wheel) about one metre (40 in.) in diameter and kept turning at approximately 650 RPM by a crank driven rope belt - onto which the glass filament was wound as it was drawn from a rod of glass kept moving steadily into a jet of flame. After a time, the skein on the reel was cut through at one point to make many filaments each about 3 metres long."

The rod drawing process is still a commercial operation today but is confined to companies in West and East Germany. Alkali glass is used and diversified products such as thermal insulation and chopped strand reinforcing mats as well as spun yarns and continuous rovings are made. The glass fiber from the commercial rod drawing process has only two-thirds the strength of the filament made from a bushing. The technique used for filament forming shows modest improvement over the ancient process. A number of rods (125 or more) about 4 millimetres in diameter are mounted in a "spinning frame" and kept slowly moving vertically downwards into individual gas flames. At first, drops of glass fall downward drawing glass filaments after them. Via an inclined plane, these are led to a rapidly revolving cylinder on which they are wound next to but independent of one another. Some firms use an electrically heated chamber.

APPENDIX III

ORGANIC FILAMENTS

A brief feasibility study was undertaken during the final stages of the program to determine if organic (plastic) filaments could be formed by the preform attenuation technique.

The plastics experts felt that the most promising candidate would be the rather amorphous polystyrene. Filaments down to about 0.0005" diameter were drawn readily from an 1/8" diameter polystyrene rod. A wide range of temperatures and drawing speeds could be used to form filament. Too low a temperature would cause a failure at the point of draw down while several hundred degrees higher would produce brittle or weak filaments with low elongation-to-break.

Polypropylene rod produced fibers over a somewhat limited temperature range. The fibers demonstrated high elongation (100 - 200%) at which time they increased in tensile stiffness just before failure.

Polycarbonate was more difficult to handle with bad foaming at the forming "onion" until careful drying of the preform for 24 hours prior to drawing the filament was done. Qualitative results indicate low tenacity.

A number of other organic compositions are candidates for this type of forming. All the likely materials need to be investigated more thoroughly to determine optimum drawing conditions. On the basis of having drawn simple tape of polystyrene, shaped organic filaments can be formed by this technique.

In summary then, plastic filaments could be drawn from polystyrene, polypropylene and polycarbonate. Polystyrene was drawn into both round and flat filaments. Tensile strengths of the filaments were about equivalent to fibers drawn by the more usual spinnerette synthetic fiber forming technique.

BIBLIOGRAPHY

1. Bureau of Naval Weapons Contract NOrd 18610 (FBM), "Research and Development for the Improvement of the Strength and Modulus of Continuous Filament Wound Cylindrical Structures Through Application of Quality Control".

2. Office of Naval Research Contract Nonr-3885(00)(X), "Feasibility Study on Hexagonal Glass Filaments".

3. B. Walter Rosen, Norris F. Dow and Zvi Hashin, "Mechanical Properties of Composites", NASA CR-31.

4. Lewis Research Center, NASA Contract NAS3-3647, "Research Specimens Filament Wound from Glass Microtape".

5. "Glass Filaments and Textiles", Ciba Review, 1963/5, page 19.

6. Historical Documents

NASA-Langley, 1964 CR-142