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SILICON CARBIDE WHISKER REINFORCED ALUMINUM COMPOSITES

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July 1968

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Sponsored by ONR and ARPA

Development of High Performance Composites

SILICON CARBIDE WHISKER REINFORCED ALUMINUM COMPOSITES

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This review was prepared under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 873, ONR Contract Authority NR 356-484/4-13-66, "Development of High Performance Composites."

FOREWORD

The research reported herein was conducted by the staff of the Monsanto/Washington University Association under the sponsorship of the Advanced Research Projects Agency, Department of Defense, through a contract with the Office of Naval Research, N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 873, ONR contract authority NR 356-484/4-13-66, entitled "Development of High Performance Composites."

The prime contractor is Monsanto Research Corporation. The Program Manager is Dr. Rolf Buchdahl (phone 314-694-4721).

The contract is funded for $5,000,000 and expires 30 April 1970.

SILICON CARBIDE WHISKER REINFORCED ALUMINUM COMPOSITES

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A B S T R A C T

Test specimens of aluminum metal reinforced with α-silicon carbide whiskers have been made by hot pressing a preform containing oriented whiskers and aluminum powder. Flexural strengths as high as 100,000 psi and flexural elastic moduli up to 20 x 10^6 psi have been obtained with 50-60 v/o α-silicon carbide having a nominal l/d of 40. The effect of various temperatures, pressures and times for hot pressing have been investigated.

The preforms are made by wet spinning the α-silicon carbide-aluminum powder mixture in a temporary organic matrix to form a filled strand. Excellent fiber orientation results from the flow through the spinneret and subsequent draw of the fiber. By laying the fibers touching one another on the takeup reel, sheets can be formed which are easily cut to the desired size for specimen preparation. Before hot pressing, the organic material is removed by ignition at 500-600°C.

Removal of debris and classification of α-silicon carbide whiskers has been accomplished by the use of a modified, commercially available paper pulp classifier. The method is simple and gives reproducible fractions of whiskers 80μ and longer.

Single crystal whisker fibers probably represent the ultimate in properties obtainable in a fiber reinforcement for use in composites. Materials such as Si_3N_4 and Al_2O_3 whiskers are reported (1) to have elastic moduli in excess of fifty million psi and tensile strengths of one- to three million psi. Although some of these whiskers are available as unoriented wools of long fibers, no practical method of cleaning and aligning these long fibers has yet been reported.

The efficiency of a reinforcement depends, among other things, upon the orientation and volume loading of the fiber, aspect ratio (l/d) and the shear strength of the fiber-matrix bond (2). The low shear strength of organic resin matrices reduces the effectiveness of short fiber reinforcement and utilization of the tremendous strength of short whiskers requires the use of a high shear strength metal matrix. Other obvious advantages of metal matrix composites are their inherent stiffness and their ability to withstand high temperatures.

These advantages have led to a considerable interest in fiber reinforced metal composites (3). While most attention has been given to continuous filament reinforce-

ment and directionally solidified eutectic systems, some work with ceramic whiskers has been reported (4,5). In general, the use of preformed single crystal whiskers presents problems in whisker clean-up, orientation and fiber to metal bonding. Preliminary results of some efforts in these laboratories to solve these problems are reported below.

Whisker Classification

The whiskers used for this work were purchased from Carborundum Company. They are reported (6) to be: "single crystal fibers of α-SiC of 0.5-3.0μ diameter and 100-750μ length with bending strength up to 3×10^6 psi and modulus of elasticity of 70×10^6 psi." This material is readily available and appears to be reasonably uniforn from batch to batch.

As received, the material analyzes 96% SiC and is contaminated with a small quantity of black, flake-like material (presumed to be graphite) and 50% or more of very short fibers and/or particulate matter. It is quite free of large particles and fibers as well as sub-micron fibers. Figure 1 shows a dark field micrograph of the starting material.

The flake contaminant is removed as follows: 100 g raw α-SiC is ignited in air at 600°C for 30 minutes to remove all organic matter. When cool, the whiskers are placed in a 4 l beaker and 3.5 l 90/10 water/ethanol is poured into the beaker. The resulting slurry is stirred gently with an off-center 1-1/2 in. diameter propellor placed 2 in. above the bottom of the vessel. The stirring rate is adjusted to give slow turnover of the mixture without violent eddies or vortex formation. As stirring is continued, the flakes float to the surface and are skimmed off with a piece of glass or a wet 3 by 5 in. file card. The skimming is repeated until no more flakes can be removed; normally 4-24 hours.

The stirring during de-flaking causes many of the whiskers to form tightly compacted, haystack-like grains or agglomerates of fibers. (This behavior has been observed with stirred, concentrated slurries of other rigid fibers, e.g. 1/8 in. chopped glass.) For classification the fibers must be individually dispersed. This is accomplished by feeding the agglomerates into a hydraulic disperser before they enter the classifier. The disperser is shown in Fig. 2, and consists essentially of a water

jet impinging upon a piece of 100-mesh screen. The second (80-mesh) screen serves to prevent splashing and catch overflow if the disperser becomes clogged or is fed too fast. All material caught on the 80-mesh screen is recycled to the disperser. While the rugged treatment given the whiskers in the disperser undoubtedly results in some fiber damage, no less destructive method has been found.

The effluent from the disperser is fed into the headbox compartment of an M-46 Clark Pulp Classifier (7). This machine was originally designed to classify papermaking pulps and consists of a cylindrical tub divided into four compartments. Between each compartment is a circular screen, 13 inches in diameter, which rotates at 48-50 rpm in a seal. Thus all the water flowing through the machine must pass through each screen. Water flow to the machine is regulated by a constant headbox and the fibers are kept suspended by a system of baffles and weirs. Each compartment is fitted with a drain plug so the fibers can be recovered at the end of the run. For α-silicon carbide classification the machine is fitted with 140-mesh (105μ), 200-mesh (74μ), 270-mesh (53μ), and 400-mesh (44μ) screens. The headbox is adjusted to a water flow of 2.5 l/min which, with the 2.5 l/min from the 1/16" diameter hole

in the disperser nozzle, gives a total flow of 5 l/min. This gives a screen to water velocity ratio of 34:1 at the inner diameter of the screen and a ratio of 82:1 at the outer diameter. Consequently, there is little tendency for fibers to pass through the screen openings lengthwise.

The actual classification is carried out as follows: The pulp classifier is filled with water and the screens started rotating. A 25 g portion of de-flaked whiskers suspended in 3000-3500 ml water is pumped into the disperser at a rate of about 60-100 ml/minute. The rate is adjusted to the maximum that can be added without causing the disperser to overflow. (Whisker slurries are somewhat difficult to pump. The CRC "Vibrastaltic" pump has been found to work quite well and was used for this work. However, any pump that can handle the whiskers without breakage can be used.) After all the whiskers have been added (1/2-1 hour) and any material left on the 80-mesh screen put back through the disperser, the classifier is allowed to run for an additional 30-45 minutes. The screens are washed and the material in each compartment is stirred 3-5 times during this period with a jet of tap water from a hose.

At the end of this period, the water leaving the fourth screen should contain very little SiC. The classifier is then turned off and the contents of each compartment, plus what water is necessary for rinsing, is run into a 17 qt. plastic bucket. The whiskers are filtered on a 400 mesh screen and then transferred with a small amount of distilled water to a 7 cm piece of filter paper on a Buchner funnel. They are dried in an air oven at 150°C. The yields are: 0.25g (1%) on the 140 mesh screen, 3.6 g (14.4%) on the 200 mesh screen, 2.0 g (8%) on the 270 mesh screen and 3.3 g (13.2%) on the 400 mesh screen. No attempt is made to collect the 63.4% that passes through the 400 mesh screen.

Typical photomicrographs of the material from each screen are shown in Figs. 3-6.

It is not possible to obtain accurate diameter and length distribution for these fibers from photomicrographs at the same magnification. In fact, accurate diameter measurements are difficult with a light microscope. Therefore, the diameter distribution was obtained by counting a micrograph at 1870X made with a Cambridge Scanning Electron Microscope. A plot of area per cent vs. diameter is shown in Fig. 7. Length distribution for the fraction of whiskers on

each screen was obtained by counting photographs similar to Figs.3-6 and the results, in terms of length percent vs. length, are shown in Fig. 8. Further numerical data on the various fractions are given in Table 1.

The data are given in terms of area and length because it is the volume of a whisker of given aspect ratio that determines its contribution to the strength of a composite. Assuming that the diameter distribution given in Fig. 7 is typical of any group of whiskers, regardless of length, the "average" aspect ratio given in Table 1 can be calculated.

TABLE 1

Weight and Distribution of Classified α-SiC Whiskers

Screen size (mesh)	140	200	270	400
Wt. % of total sample[a]	0.9	14.4	8.0	13.1
Fibers counted (no.)	1471	1369	342	993
Peak wt. % length[b]	225 μ	190 μ	185 μ	160 μ
Average wt. % length[c]	217 μ	159 μ	142 μ	122 μ
50 wt. % >	246 μ	190 μ	170 μ	146 μ
75 wt. % >	210 μ	160 μ	140 μ	120 μ
90 wt. % >	148 μ	107 μ	93 μ	77 μ
95 wt. % >	110 μ	70 μ	65 μ	55 μ
Nominal l/d, ave.	146	107	96	82

(a) Remaining 63.4% of sample passed through the 400 mesh screen.

(b) Taken from the curve, Fig. 8.

(c) Total length of all fibers divided by number of fibers.

Silicon Carbide/Aluminum Preforms

Following classification, the whiskers must be oriented, dispersed in the matrix metal and formed into the desired shape. Although strengthening of metals has been achieved with random whisker mats (13), good unidirectional properties and high volume loading require oriented reinforcing fibers.

The orientation of discontinuous fibers, particularly asbestos, by means of extrusion or wet spinning in a temporary organic matrix was first reported by Wilke et al. (8). Alginates, methyl cellulose, and viscose are among the matrices suggested in this patent. More recently, Wohrer and Economy (9) at Carborundum Co. have described the orientation of α-silicon carbide by wet-spinning in a poly acrylonitrile copolymer matrix. These strands are now commercially available (10). α-Silicon carbide reinforced composites are made by burning off the temporary matrix polymer at 550-600° and then impregnating the oriented whisker strand with resin or metal.

While impregnation of the oriented whiskers with an organic resin does not present severe problems, molten metals will not wet ceramic whiskers. To overcome this problem, the metal in powder form is uniformly dispersed in the oriented whisker strand and then formed around the fibers by hot pressing. The oriented whisker-metal powder preform is made by wet spinning the metal powder along with the whiskers in a temporary matrix and then burning off the organic matter.

To make good preforms, the temporary organic matrix must have a number of attributes. These include:

1. Good film or fiber forming characteristics and solubility in a solvent that does not react with the metal powder.
2. The ability to be wet spun from dilute solution, preferably ten per cent or under. Depending upon the aspect ratio, only one-half to two volume per cent whiskers can be handled in the spinning solution. Thus a ten per cent polymer solution will give a strand containing only five to seventeen volume per cent whiskers whereas a two per cent solution will give twenty to fifty volume per cent.
3. Sufficient solution viscosity to disperse the whiskers and metal powder and carry them through the spinneret. In general, the lowest usable polymer concentration is limited by viscosity rather than ability to form fibers.
4. Enough strength to allow draw, windup and handling of the filled strand. Polymers which form sticky strands that do not shrink much after leaving the bath are especially suited to the direct formation of tapes, which are more easily handled than single strands.

5. The ability to burn off without disturbing the orientation of the whiskers. Polymers which decompose and burn without melting are ideal. However, resins which char close to their melting points can be used if they are carefully charred before actual burn-off.

A number of temporary matrix polymers have been investigated. Methyl cellulose and polyvinyl alcohol give somewhat brittle strands which burn off well but are sensitive to water and are difficult to rid of salts from the coagulating bath. Alginic acid, cuprammonium rayon and viscose rayon spin well from dilute solutions and burn off well since they do not melt. However, they shrink considerably upon drying and formation of tapes is difficult. Cellulose triacetate gives the best strands but must be burned off with great care and requires the use of chlorinated solvents. Cellulose acetate can be used either to make strands, which are weak and difficult to handle, or tapes. For the latter it is the best polymer available. Spun as an eight to twelve weight per cent solution from methyl acetate into a Stoddard solvent bath, cellulose acetate gives a strong fiber that can be wrapped tightly around the takeup drum without breakage. The filled fiber is sticky as it emerges from the bath and, if the fiber is wound so that succeeding coils touch, an easily handled tape is obtained. The material is plasticized by water but does not swell or dissolve.

The spinning setup is quite conventional and similar to that described in standard texts (11). A schematic diagram of the apparatus is shown in Fig. 9. The design of the spinning orifice or spinneret is quite critical. Square-edged orifices, as commonly used in fiber manufacture, are readily blocked by the whisker fibers. The orifice must be tapered, preferably in the form of a venturi. The orifices for the present work were made by drawing down thin wall glass tubing.

The concentration of α-silicon carbide whiskers and aluminum powder that can be put into the spinning solution is governed by the aspect ratio of the silicon carbide. At a given whisker concentration, very little change in the handling characteristics of the mixture is observed when a volume of metal powder equal to the silicon carbide, already present is added. Thus, it is possible to make a preform with virtually any desired whisker to metal volume ratio.

Mixing the whiskers and metal powder into the viscous cellulose acetate solution is difficult. High speed mixers, vibrators and ultrasonic devices produce quite severe whisker damage. Ultrasonic mixers also degrade the polymer. The most satisfactory method found so far is slow stirring in a very

viscous spinning solution. Cooling the polymer solution during mixing helps increase viscosity. A large propellor (80% of vessel diameter) rotating at 100-200 rpm in a liquid level 1-1/2 times the vessel diameter will disperse 0.5 v/o 200μ α-SiC in a 100 ml batch in 4-8 hours. Much faster mixing, and more whisker damage, is obtained in a 50 ml blender jar equipped with special blades shown in Fig. 10. The blender is run as slowly as possible. Figures 14 and 15 show the extent of damage to the whiskers in a typical blender-mixed preform.

The orientation of the whisker fibers in a preform strand can be quantitatively measured by x-ray diffraction (12). An x-ray diffraction picture of a typical strand which contains 42.4/19.3/38.3 weight percent cellulose acetate/α-SiC/Al (equivalent to 61.8/11.5/26.7 volume percent or a SiC/Al ratio of 70:30) is shown in Figure 11. The two outer rings are from the aluminum metal and the four arcs that lie in the inner circle represent diffraction from the crystal planes of the α-SiC. Perfectly oriented single crystal whiskers will produce spots whereas completely random arrangement of the fibers gives rise to a ring similar to those observed for the aluminum.

The length of the arc produced is a quantitative measure of the degree of orientation. In the sample pictured in Figure 10, 90% of the whiskers lie within 20° of the fiber axis.

The experimental procedure for making a SiC-Al tape preform is as follows:

The cellulose acetate solution is made by dissolving 60 g fiber grade cellulose acetate (Eastman A-394-60) in 354 ml methyl acetate. This is most conveniently done by putting both materials into a 16 oz. jar and allowing the jar to rotate on a polymer solution wheel for 24-48 hours. Normally, this solution does not need to be filtered if it is allowed to settle for a day before use. The solution contains 15 w/v percent cellulose acetate.

To make up the SiC-Al spinning mixture, 33 ml of the 15% cellulose acetate solution is placed in a 50 ml blender jar (Aloe Scientific) equipped with a special large set of blades (Figure 10). Then 1.60 g classified α-SiC is placed in the jar on top of the acetate solution along with 3.16 g Al powder (Alcoa Atomized Aluminum No. 123). The powder and whiskers are wet with 7.0 ml methyl acetate added dropwise. The mixture is then stirred until the solids are completely wet out and the entrapped air removed.

The stirring speed is kept as low as possible; just sufficient to turn the solution over when no air is entrapped. Since the whiskers carry a good deal of air into the solution, short periods of stirring with pauses to allow the escape of air bubbles are best. Once the solids are wet out, an additional 10 ml methyl acetate is added and the mixture is stirred until the spinning solution is smooth and homogeneous. It is then sucked into a 50 ml plastic syringe (BD Plastipak) through a 3-4" long section of 1/8" i.d. x 1/4" o.d. polyethylene tubing expanded on one end to fit the male hypodermic fitting on the syringe. The syringe is capped and the larger air bubbles removed by holding it, delivery end up, against a 60 Hz vibrator (A few small air bubbles do not interfere with the spinning operation since they are not trapped in the fiber but escape at the orifice.)

For spinning, the syringe is equipped with a 0.030" glass orifice (drawn from 0.087" i.d. x 0.157" o.d. tubing) attached with a short polyethylene tubing ell. The syringe is placed in a syringe driver (Sage Model 255-1) with the orifice tip just under the surface of a Stoddard solvent coagulating bath. The bath is 36" long x 3" wide x 2" deep and is filled to a depth of 1-1/2". It is desirable to

operate the entire spinning apparatus in a hood. The syringe pump is set to deliver 3 ml/minute. When the fiber emerges from the orifice, it is grasped with a pair of tweezers, pulled through the bath, led under a hook below the surface of the Stoddard solvent at the far end of the bath and then up over a traveling guide and onto the drum where it is secured by wedging it under a clip attached to the rin of the drum. The 5-3/16" o.d. perforated aluminum drum (Mirro) is run at a surface speed of 42-45 ft/min which gives a draw ratio of 2:1. The guide, which is placed close (1/8") to the drum for good control, moves at 0.4-0.5"/min along the axis of the drum. This causes the fiber to be laid down so that each turn touches the next but does not overlap. When half the spinning mixture has been used, the guide travel is reversed to give a second fiber layer. Sufficient solvent is present in the fiber to cause it to stick together to form a coherent tape. When the run is over, the drum of tape is dried for 30 min at 80°C in an air oven. When cool, the drum and tape are immersed in water for 1-2 minutes, the tape is slit along the drum axis with a razor blade, removed, and placed under a weighted piece of Plexiglas for a day or two to flatten. The tape, which is 16-1/2" long by 3-4" wide, is now ready to be cut to size, burned off and fabricated into composites.

Composite Fabrication

Powdered metal parts are normally made by cold pressing the powder at very high loads followed by sintering (14). Pressing ruptures the oxide layer surrounding each powder particle and creates metal to metal contact. The sintering promotes bonding to give a strong part. When reinforcing whiskers are dispersed in aluminum powder, cold pressing severely damages the fibers. In addition, the metal is not sufficiently ductile at room temperature to flow around the fibers and form a continuous metal phase. Just below its melting point, however, aluminum is soft and ductile enough to permit encapsulation of the whiskers and rupture of the oxide film at modest pressures. Above the melting point, the metal will not wet the whiskers.

The procedure for making composite specimens by hot pressing of the SiC whisker/Al powder preforms described above is as follows:

A preform mat containing the oriented SiC whiskers and Al powder dispersed in cellulose acetate is cut into 1/2" x 1-1/2" rectangular pieces. Enough of these to give one gram of SiC/Al after removal of the organic material are carefully stacked on a ceramic plate and put into a 350°C muffle furnace. One half hour is required to char the cellulose acetate after which the stack is transferred to

a 550°C furnace for one hour to get complete burn-off. The cooled stack, which has sufficient green strength to permit careful handling, is then put into a 1/2" x 1-1/2" stainless steel pressing die. The die walls and the mating surfaces of the close-fitting plungers are coated with colloidal graphite to prevent sticking. The die is placed inside a wire-wound resistance furnace between the platens of a small laboratory press, brought to temperature, and the load applied. Temperatures are measured with thermocouples placed 1/16" above the bottom surface of the top plunger and at the center of one die wall. The sample is kept at temperature and pressure for one hour and allowed to cool under load. The specimen is pressed out of the die and ground smooth (through 600 grit) prior to mechanical testing. For tensile tests a constant radius reduced section is ground into the specimen. Flexural tests are made with a three-point jig using a one inch span and flexural moduli calculated from the plot of load vs. cross-head movement. All tests are done with an Instron Model TM-L at a crosshead speed of 0.02 in/min. Volume loading of SiC is determined by weight analysis for Si and densities determined by buoyancy in water.

Strengths and moduli are essentially constant over the pressure range 2000 to 5000 psi. Pressures less than 1000 psi give porous samples that are too weak to test. This is attributed to insufficient metal flow. Pressures greater than 5000 psi were not tried.

Temperature is a much more critical variable than pressure. Hot pressing below about 610°C gives specimens too weak to test. Strengths and moduli are highest at temperatures between 620 to 630°C and decrease rapidly above 640°C. This appears to be caused by poor whisker-metal bonding at the higher temperatures. Figure 12 is a scanning electron micrograph of the tensile fracture surface of a sample which was hot pressed at 630°C. The whiskers are broken at the metal surface with no sign of whisker pull-out. This indicates good metal-whisker bonding. The sample whose fracture surface is shown in Figure 13 was hot pressed at 648°C. The whiskers show no adhering metal and are pulled out of the matrix.

Silicon carbide whiskers are fragile and therefore sensitive to the various fabrication steps. Figures 14, 15, and 16 are optical photomicrographs showing the same batch of whiskers after classification by wet screening, after wet-spinning and burn-off of the polymer, and after hot pressing and dissolution of the aluminum matrix, respectively. Whisker damage has occurred during each fabrication step.

Liquid phase hot pressing has been used by other workers (5) in the fabrication of metal matrix composites. The Al-Si alloy has a eutectic temperature of 577°C and, at an alloy composition of 2.5 wt % Si, the phase diagram (15) shows liquid in equilibrium with solid over a 70°C temperature range. In the hope that the hydrostatic character of this liquid-solid (slushy) region would lessen whisker damage, several specimens were made by heating burned-off SiC/Al (2.5 Si) alloy powder preforms to ∿ 600°C and then applying the load to the die. Next, the sample was cooled to a few degrees below the eutectic temperature at the pressing pressure and held at these conditions for about an hour. Unfortunately, these specimens show no significant decrease in whisker damage. Strengths and moduli are comparable to those obtained with pure aluminum at 620-630°C. Failure to drop the temperature below the eutectic temperature of the alloy results in low strength samples exhibiting little metal to whisker bonding. Poor results are also obtained if temperatures much above 600°C (e.g. 615°C) are used during the first part of the cycle.

The reason for the rapid decrease in metal-whisker bonding at temperatures near the melting point in the case of pure aluminum and well into the liquidus range with the alloy

is not known. Silicon carbide whiskers are believed to have a layer of silica on their surface (16). The metal-whisker bond may result from diffusion of silicon and oxygen into the aluminum to form a mutually compatible layer at the interface. At the higher hot pressing temperatures, this layer may become molten or dissolve in the matrix metal causing de-wetting of the whiskers and destruction of the bond. Since good bonding between the metal and the reinforcement is a key factor in the strength of a composite, future work needs to be done in this area.

Time may also be a factor in the fabrication of metal-whisker composites by hot pressing. One experiment in which the temperature and pressure were maintained for three hours instead of the customary one hour gave a specimen which exhibits about twenty per cent higher strength than the normal samples. Tentatively, this suggests the importance of diffusion in the formation of a well-bonded specimen.

Normally, some metal extrudes out of the die during hot pressing, even when pure aluminum is used below its melting point. Burned-off preforms containing 5, 20, 30 and 40 volume per cent silicon carbide are found by chemical analysis to

yield composites with about 30, 45, 55 and 70 volume per cent silicon carbide, respectively. Specimens containing 70 volume per cent whiskers are friable and usually too weak to test. Evidently there is a limit to whisker loading above which it is difficult to spread the metal uniformly around all the whiskers.

Strength and modulus are plotted against v/o silicon carbide in Figs.17 and 18. The data are linear, although there is some scatter. (The data for unreinforced aluminum were obtained from samples made from preforms containing aluminum powder but no whiskers. These were burned off and handled in the normal manner except that hot pressing was done at temperatures lower than those used for the reinforced samples to prevent excessive extrusion of the metal out of the die.) Tensile strengths are as much as three times and flexural moduli as much as two times that of the unreinforced aluminum. Moduli fall considerably below rule-of-mixture values as would be expected for short fibers lacking perfect orientation. Extrapolation of the modulus curve to 0 v/o gives a value considerably below the modulus of unreinforced aluminum; no stiffening occurs until loadings exceed thirty volume per cent. This may be due to voids and Fig. 19 shows the measured densities

plotted against the chemically determined volume per cent silicon carbide. The theoretical line shown was calculated using 3.18 g/cc and 2.71 g/cc as the densities of silicon carbide and aluminum, respectively. The experimental points fall somewhat below theoretical. This difference probably represents voids and may explain the lack of stiffening obtained at low loadings.

Table 2 compares the strength and modulus of silicon carbide whisker-reinforced aluminum with literature values for continuous filament boron-aluminum (3) and commercial aluminum alloys (17). The "best sample" represents the three hour specimen described above and the average sample values were taken from Figs. 17 and 18.

Samples were also tested transverse to the whisker axis and the data show significant strengthening and stiffening as compared to unreinforced aluminum. This results from the lack of perfect whisker orientation and is aided by good metal-whisker bonding. It also indicates that better longitudinal properties should be obtainable with better whisker orientation. Misorientation beyond that in the original spun strand probably occurs during the flow of metal in the initial stages of hot pressing.

While the strengths of the silicon carbide-aluminum materials reported here do not compare to those obtained for boron-aluminum in the fiber direction, the results are encouraging, especially in view of the excellent transverse properties of the hot pressed samples. Compared to the alloys, the strengths of the silicon carbide-aluminum composites are similar to 2024 and lower than 7075 at room temperature. However, at elevated temperatures the alloys lose strength rapidly whereas the reinforced materials do not (18). Where stiffness is the primary concern, both the boron-aluminum and silicon carbide-aluminum systems exceed aluminum and its alloys at all temperatures.

TABLE 2

Comparison of Reinforced Aluminum and Aluminum Alloys

Sample	Ultimate Tensile Strength (psi x 10^{-3})	Flexural Modulus (psi x 10^{-6})
Hot pressed Al powder (0 v/o SiC)	20	10
Hot pressed Al/SiC (50 v/o SiC)* (best sample - 3 hrs.)	58	16
Hot pressed Al/SiC (50 v/o SiC)* (average for this work)	45	16
Hot pressed Al/SiC (50 v/o SiC)* (transverse means)	31	13
Al/B (50 v/o B)	160	30
2024 - T6 Al alloy	68	10.5
7075 - T6 Al alloy	83	10.5
1100 - 0	13	10
1100 - H 18	24	10

*Normalized values.

ACKNOWLEDGMENT

This work was sponsored by the Advanced Research Projects Agency Department of Defense, through contract with the Office of Naval Research N00014-67-C-0218 (formerly N00014-66-C-0045), ARPA Order No. 873, ONR contract authority NR. 356-484/4-13-66, "Development of High Performance Composites".

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Captions For Figures

Fig. 1. Unclassified α-SiC, 84 X.

Fig. 2. Schematic diagram of Disperser.

Fig. 3. Classified α-SiC from 140 mesh screen, 84 X.

Fig. 4. Classified α-SiC from 200 mesh screen, 84 X.

Fig. 5. Classified α-SiC from 270 mesh screen, 84 X.

Fig. 6. Classified α-SiC from 400 mesh screen, 84 X.

Fig. 7. Area per cent vs. diameter for α-SiC whiskers.

Fig. 8. Length per cent vs. length for classified α-SiC whiskers.

Fig. 9. Schematic diagram of wet spinning apparatus.

Fig. 10. Photograph of special blade assembly for 50 ml blender.

Fig. 11. X-ray diagram of SiC/Al/Cellulose acetate strand.

Fig. 12. Scanning electron micrograph of tensile fracture surface showing good metal-whisker bonding of a composite hot pressed at 630°C, 9190X.

Fig. 13. Scanning electron micrograph of tensile fracture surface showing whisker pull-out of a composite hot pressed at 648°C, 2168X.

Fig. 14. Optical micrograph of silicon carbide whiskers following wet-screening, 170X.

Fig. 15. Optical micrograph of silicon carbide whiskers after wet-spinning and burn-off of polymer, 170X.

Fig. 16. Optical micrograph of silicon carbide whiskers after hot pressing and dissolution of the aluminum matrix, 170X.

Fig. 17. Flexural and tensile strength vs. volume per cent silicon carbide.

Fig. 18. Flexural modulus vs. volume per cent silicon carbide.

Fig. 19. Composite density vs. volume per cent silicon carbide.

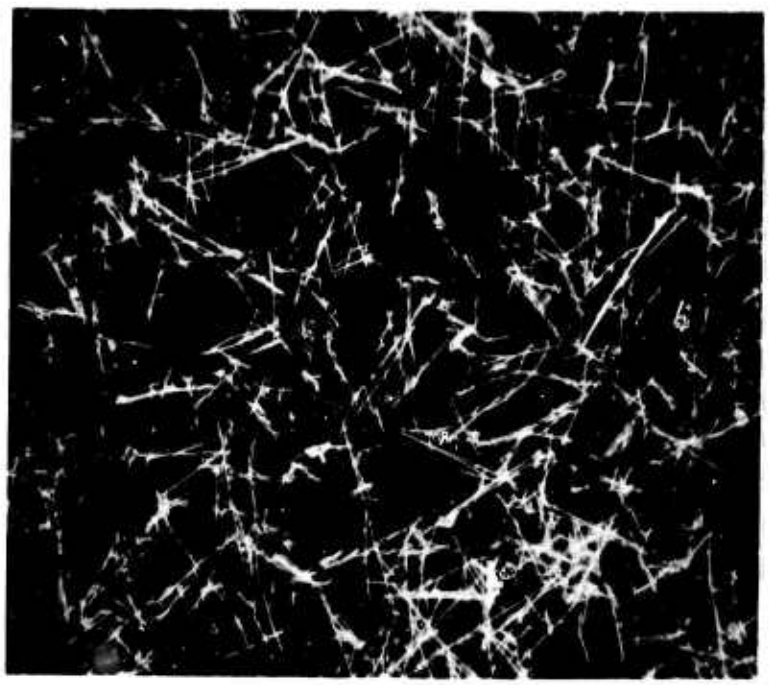

FIGURE 1

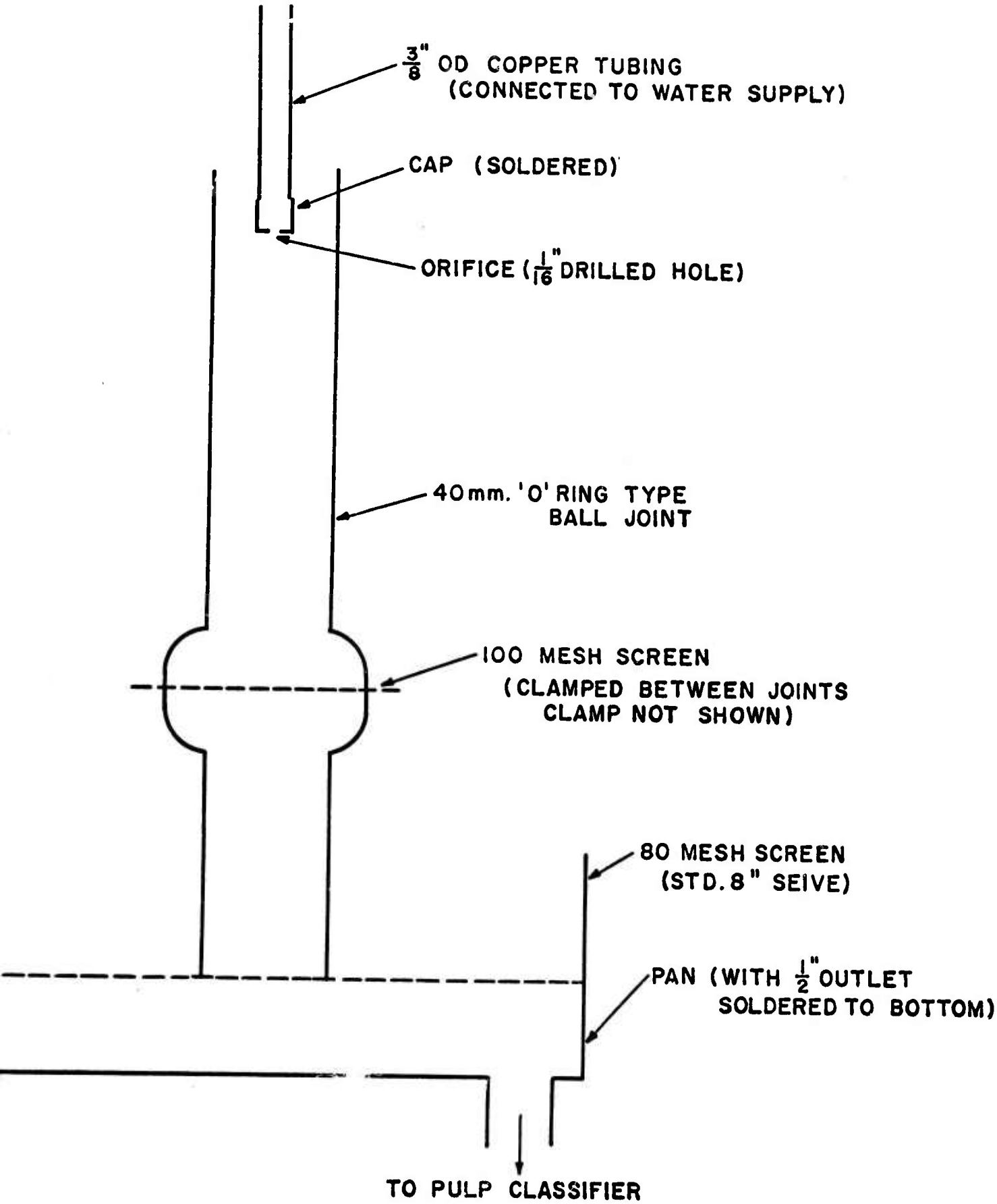
3/8" OD COPPER TUBING
(CONNECTED TO WATER SUPPLY)
CAP (SOLDERED)
ORIFICE (1/16" DRILLED HOLE)
40mm. 'O' RING TYPE
BALL JOINT
100 MESH SCREEN
(CLAMPED BETWEEN JOINTS
CLAMP NOT SHOWN)
80 MESH SCREEN
(STD. 8" SEIVE)
PAN (WITH 1/2" OUTLET
SOLDERED TO BOTTOM)
TO PULP CLASSIFIER

FIGURE 2

FIGURE 3

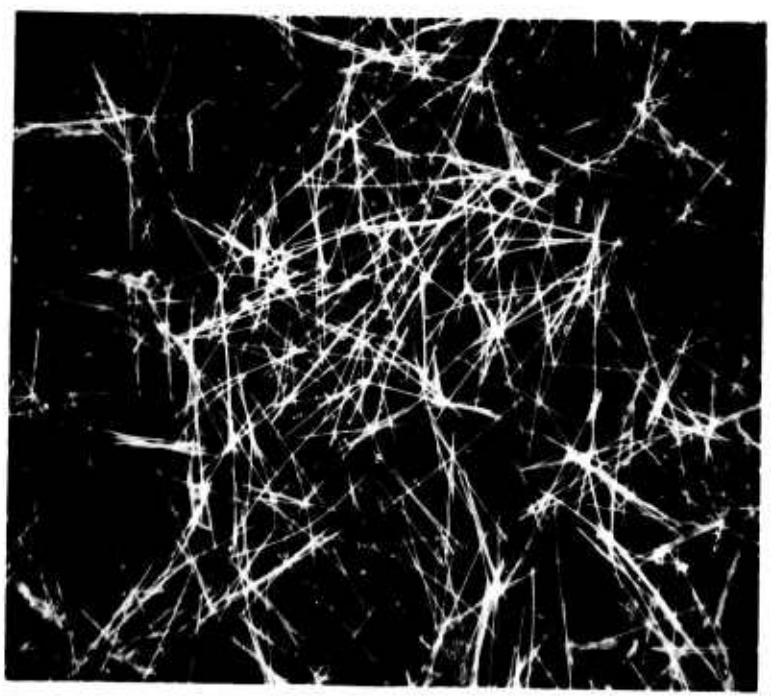

FIGURE 4

FIGURE 5

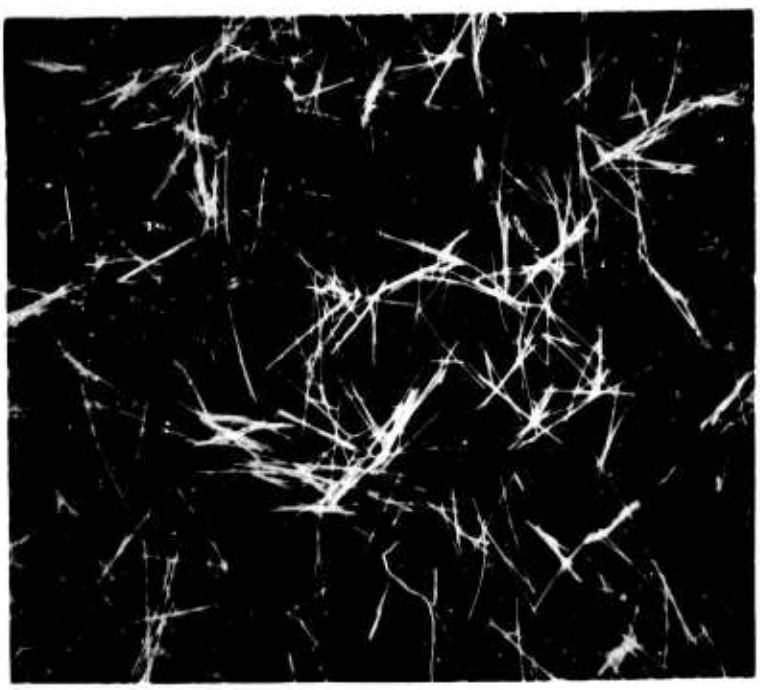

FIGURE 6

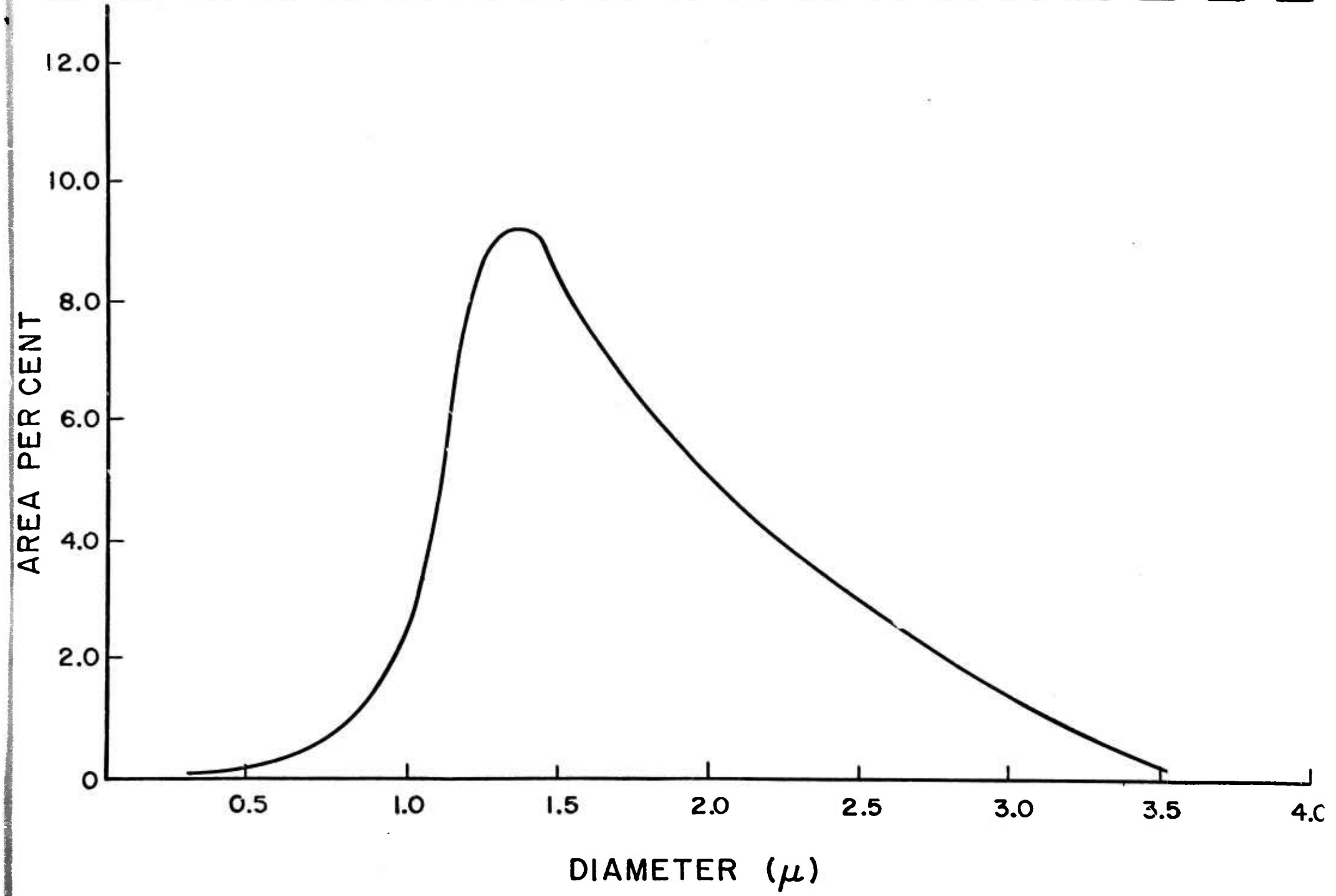
12.0
10.0
8.0
6.0
4.0
2.0
0
AREA PER CENT
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
DIAMETER (μ)

FIGURE 7

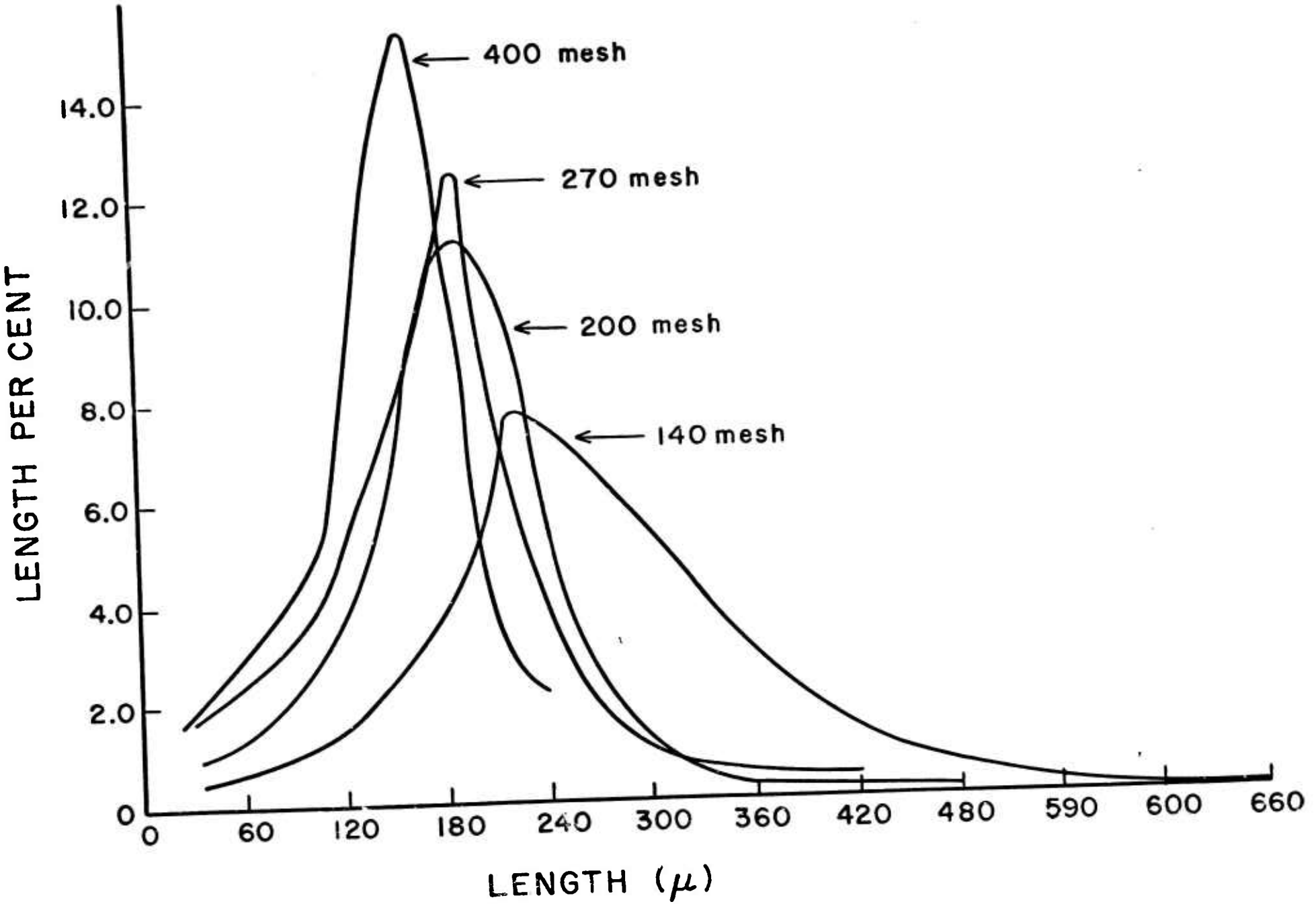
400 mesh
270 mesh
200 mesh
140 mesh
LENGTH PER CENT
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0
0
60
120
180
240
300
360
420
480
590
600
660
LENGTH (μ)

FIGURE 8

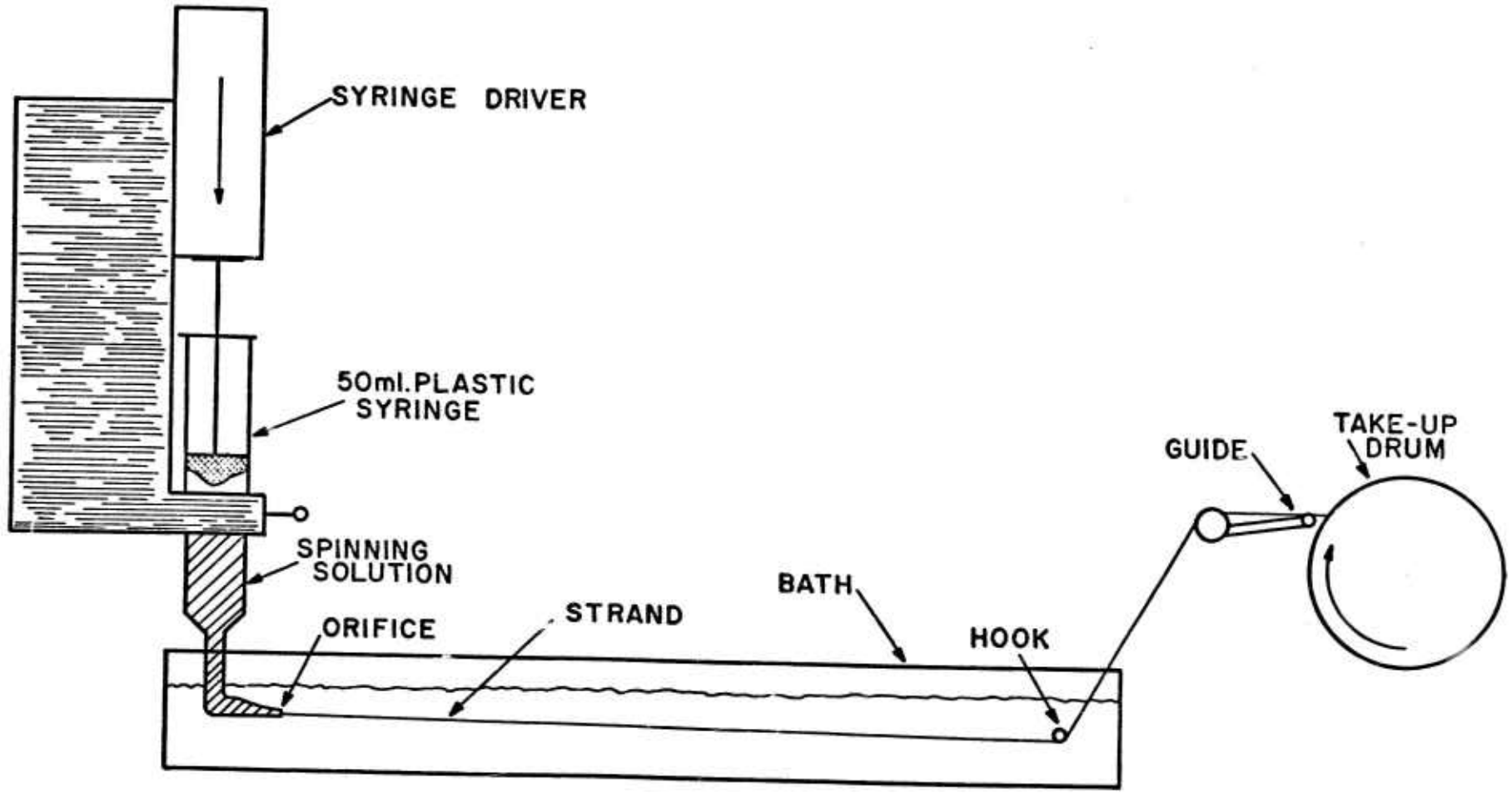
SYRINGE DRIVER
50ml. PLASTIC SYRINGE
SPINNING SOLUTION
ORIFICE
STRAND
BATH
HOOK
GUIDE
TAKE-UP DRUM

FIGURE 9

FIGURE 10

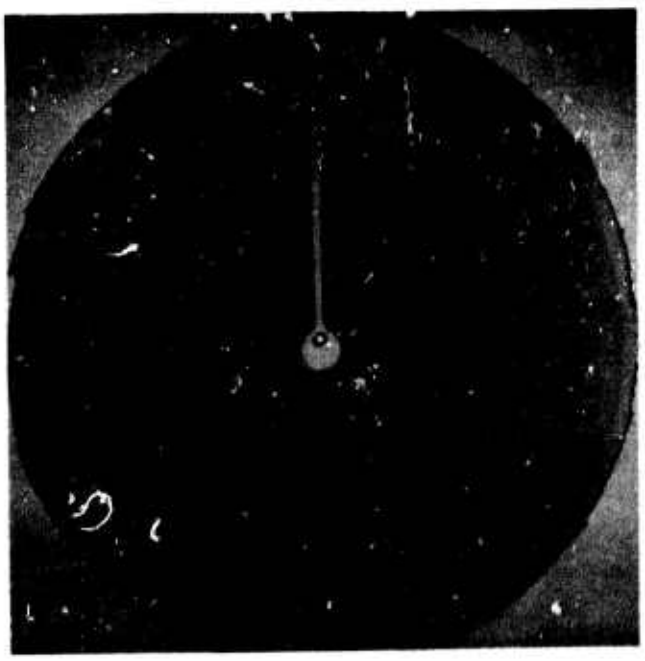

FIGURE 11

FIGURE 12

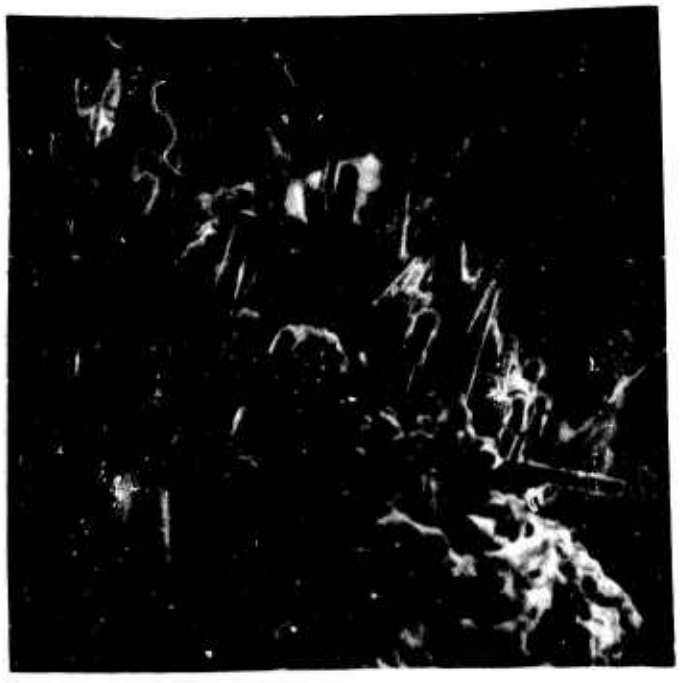

FIGURE 13

FIGURE 14

FIGURE 15

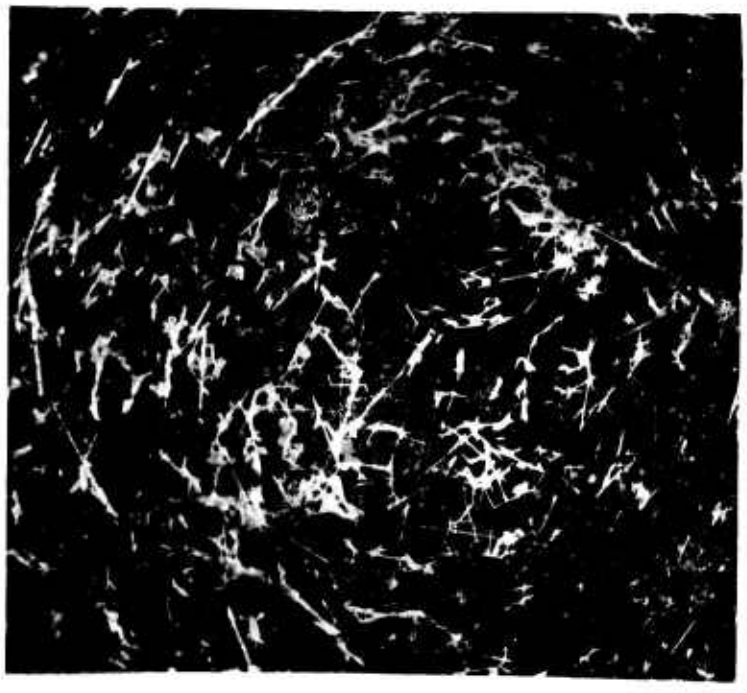

FIGURE 16

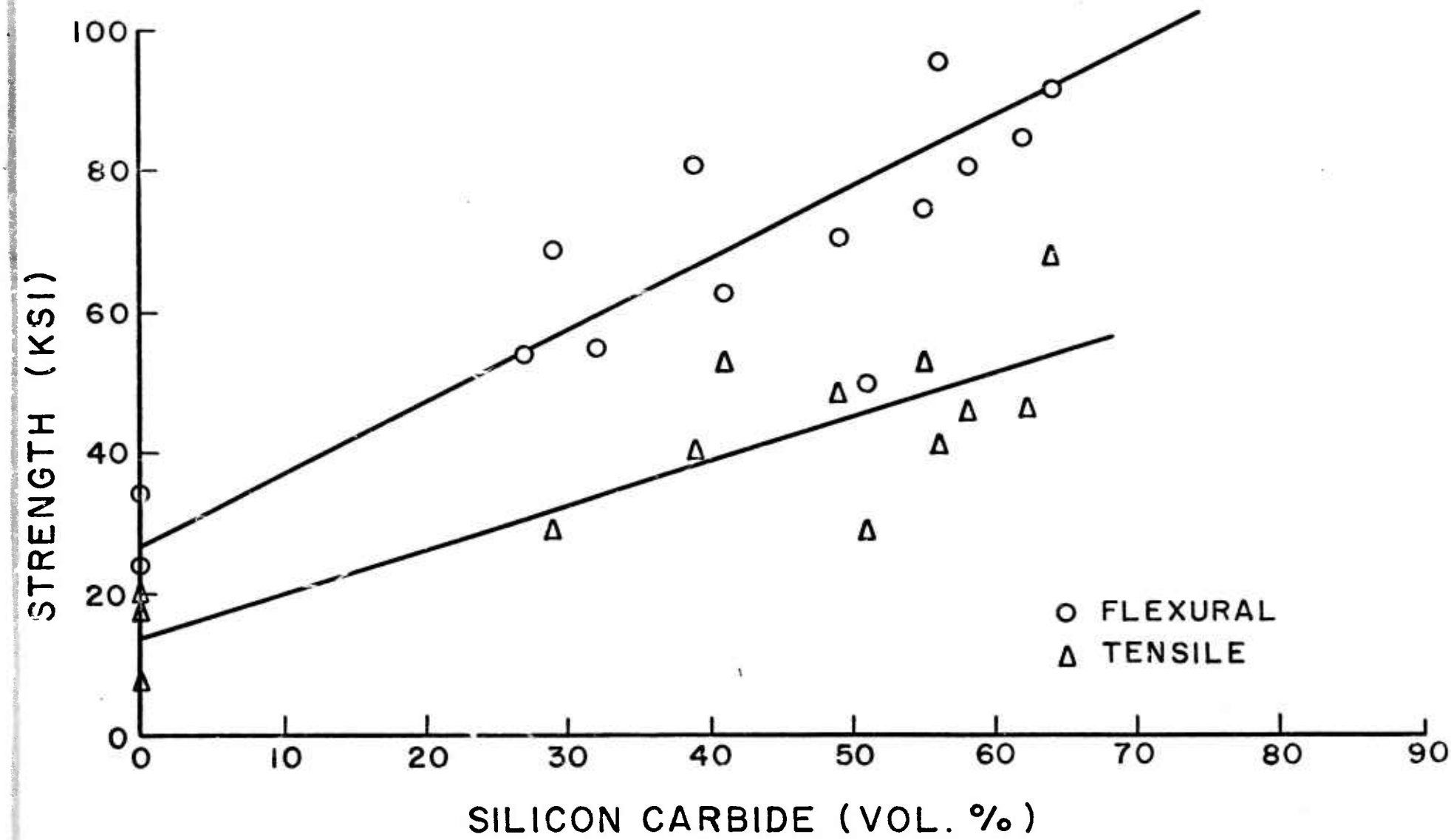
100
80
60
40
20
0
STRENGTH (KSI)
0
10
20
30
40
50
60
70
80
90
SILICON CARBIDE (VOL. %)
FLEXURAL
TENSILE

FIGURE 17

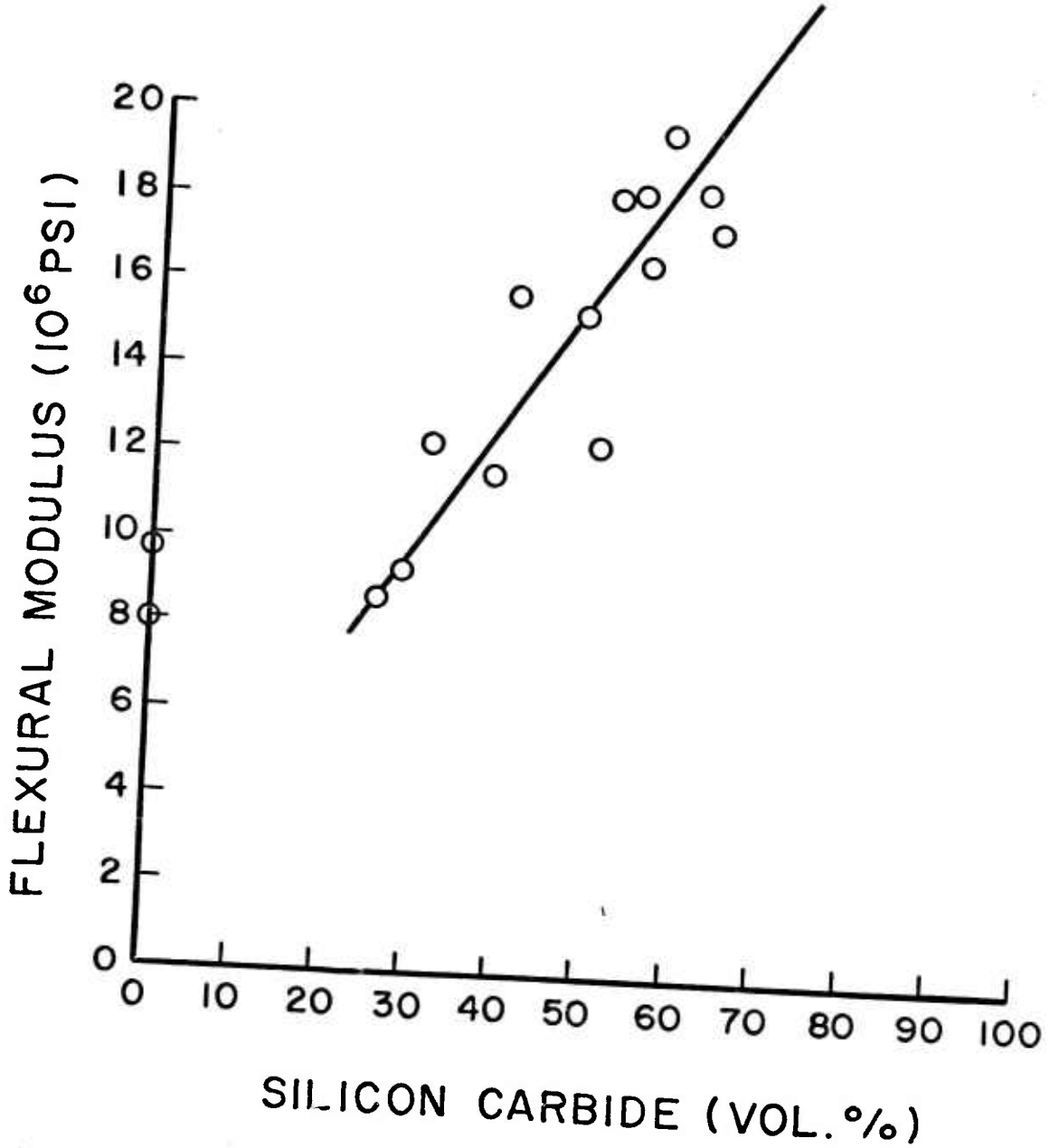
FLEXURAL MODULUS (10^6 PSI)
0
2
4
6
8
10
12
14
16
18
20
0
10
20
30
40
50
60
70
80
90
100
SILICON CARBIDE (VOL. %)

FIGURE 18

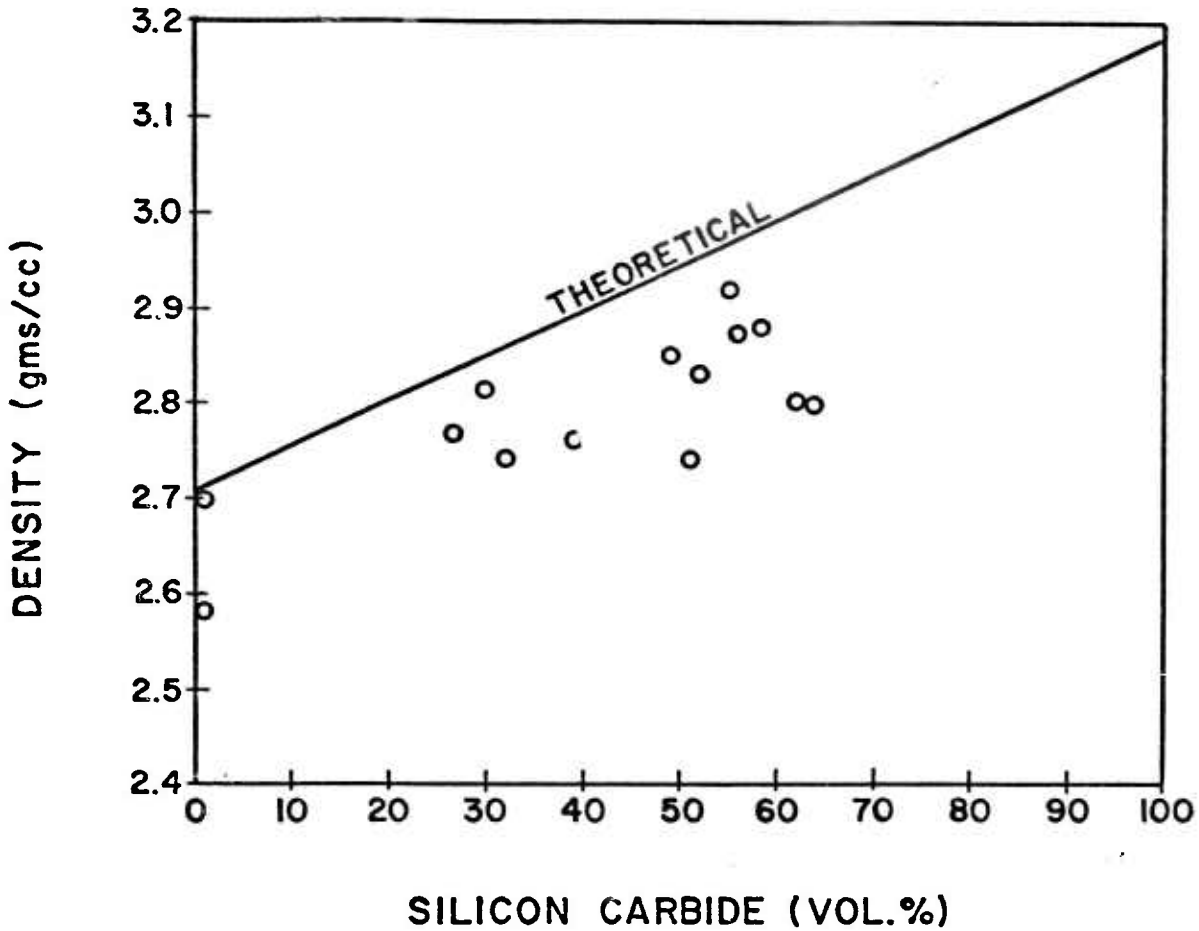
3.2
3.1
3.0
2.9
2.8
2.7
2.6
2.5
2.4
DENSITY (gms/cc)
THEORETICAL
0
10
20
30
40
50
60
70
80
90
100
SILICON CARBIDE (VOL.%)

FIGURE 19

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)*

Monsanto Research Corporation

2a. REPORT SECURITY CLASSIFICATION

Unclassified

2b. GROUP

3. REPORT TITLE

Silicon Carbide Whisker Reinforced Aluminum Composites

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

5. AUTHOR(S) *(First name, middle initial, last name)*

Oliver deS. Deex
Royce G. Schierding

6. REPORT DATE

July 1968

7a. TOTAL NO. OF PAGES

57

7b. NO. OF REFS

18

8a. CONTRACT OR GRANT NO.

N00014-67-C-0218

b. PROJECT NO.

c.

d.

9a. ORIGINATOR'S REPORT NUMBER(S)

HPC 68-65

9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)*

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11. SUPPLEMENTARY NOTES

12. SPONSORING MILITARY ACTIVITY

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13. ABSTRACT

Test specimens of aluminum metal reinforced with α-silicon carbide whiskers have been made by hot pressing a preform containing oriented whiskers and aluminum powder. Flexural strengths as high as 100,000 psi and flexural elastic moduli up to 20 x 10^6 psi have been obtained with 50-60 v/o α-silicon carbide having a nominal l/d of 40. The effect of various temperatures, pressures and times for hot pressing have been investigated.

The preforms are made by wet spinning the α-silicon carbide-aluminum powder mixture in a temporary organic matrix to form a filled strand. Excellent fiber orientation results from the flow through the spinneret and subsequent draw of the fiber. By laying the fibers touching one another on the takeup reel, sheets can be formed which are easily cut to the desired size for specimen preparation. Before hot pressing, the organic material is removed by ignition at 500-600°C.

Removal of debris and classification of α-silicon carbide whiskers has been accomplished by the use of a modified, commercially available paper pulp classifier. The method is simple and gives reproducible fractions of whiskers 80 μ and longer.

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S/N 0101-807-6801

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Security Classification

14. KEY WORDS	LINK A ROLE	LINK A WT	LINK B ROLE	LINK B WT	LINK C ROLE	LINK C WT
whiskers						
metal matrix						
composites						
classification						
wet spinning						
hot pressing						

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(PAGE 2)

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