

April 23, 1968

H. H. WEBBER ET AL

3,379,000

METAL FILAMENTS SUITABLE FOR TEXTILES

Original Filed March 14, 1963

FIG. 1

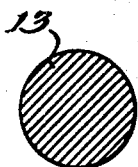


FIG. 2

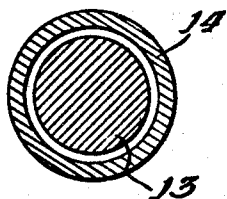


FIG. 3

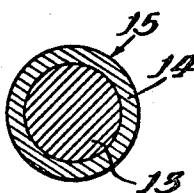


FIG. 4

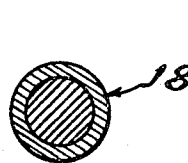


FIG. 5

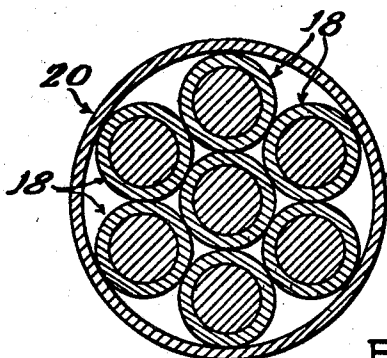


FIG. 6

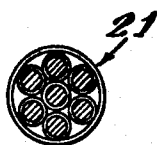


FIG. 7

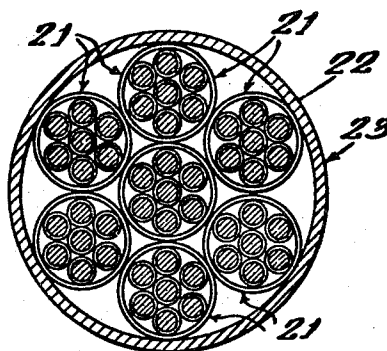


FIG. 8

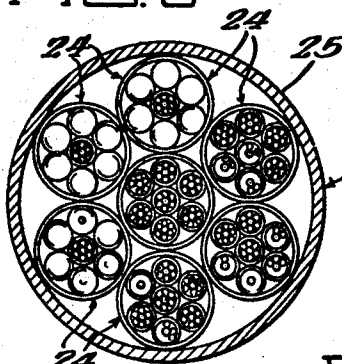


FIG. 9

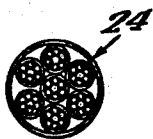


FIG. 10

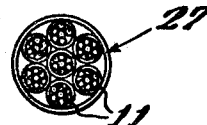


FIG. 11

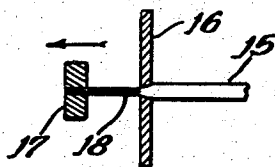
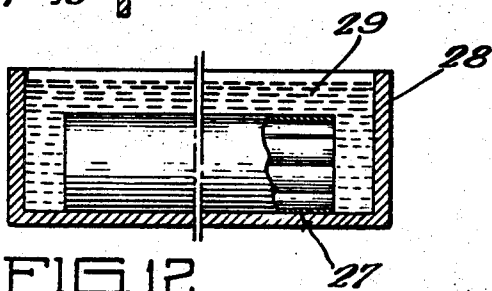


FIG. 12



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3,379,000

METAL FILAMENTS SUITABLE FOR TEXTILES

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Application Mar. 14, 1963, Ser. No. 265,262, which is a continuation of application Ser. No. 463,759, June 14, 1965, now Patent No. 3,277,564, dated Oct. 11, 1966. Divided and this application Sept. 15, 1965, Ser. No. 487,559

7 Claims. (Cl. 57—139)

ABSTRACT OF THE DISCLOSURE

A tow of metal filaments and method of forming the same by constricting a bundle of rods in a suitable matrix material whereby the rods define filaments having a maximum cross section of less than approximately 10 microns and a length of greater than approximately 50 feet and having a trace amount of a different material diffused in the outer surface thereof. The constriction is effected by a drawing of the rods in the matrix to the desired final diameter.

This application comprises a divisional of application Ser. No. 463,759 filed June 14, 1965, now U.S. Patent No. 3,277,564, comprising a continuation of application Ser. No. 265,262 filed Mar. 14, 1963, now abandoned.

This invention relates to the forming of filaments and in particular to the forming of fine filaments having a diameter of approximately 10 microns or below.

There has long been a need for high strength metallic filaments and the like for use in fabrics having improved physical characteristics as compared to the conventional textile fabrics such as cotton, wool and the like. One example of such a need is that of the automotive vehicle tire industry wherein reinforcing fabrics of many different types have been employed over the years in an attempt to find a completely satisfactory carcass reinforcing fabric.

Further, recent investigations of the tensile strength of small diameter metal filaments such as whiskers of iron, copper, silver and the like having a diameter of under 15 microns have indicated that filaments of this type having a diameter of approximately 4 microns or less have increased tensile strength substantially beyond the expected tensile strength as determined by Hooke's law. Thus the desirability of forming such small diameter filaments in substantial quantities and a relatively low cost is manifest. The present invention comprehends an improved process for producing such filaments at such low cost and thus makes available for the first time commercially practical high strength filaments of metal and the like for use in improved fabrics, cables, filter media, superconductive bodies, etc.

A principal feature of the invention is the provision of a new improved filament product.

A further feature of the invention is the provision of a tow of filaments having a diameter of approximately 10 microns and below and in substantially continuous lengths such as in lengths of approximately 50 feet and over.

Still another feature of the invention is the provision of such filaments having controlled uniformity.

Yet another feature of the invention is the provision of such a tow comprising unburnished metal yarn.

Still another feature of the invention is the provision of such a tow wherein the filaments are formed in a novel twisted relationship.

Other features and advantages of the invention will be

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apparent from the following description taken in connection with the accompanying drawing:

FIG. 1 is a transverse cross-section of a metal wire from which a tow of filaments may be formed in accordance with the invention;

FIG. 2 is a transverse cross-section of the wire disposed within a coaxial sheath as in a first step of the method of forming thereof;

FIG. 3 is a transverse cross-section of the wire and sheath assembly with the sheath reduced in diameter as in a subsequent step;

FIG. 4 is a transverse cross-section of the sheathed wire structure as reduced in diameter in a further subsequent step;

FIG. 5 is a transverse cross-section of a plurality of sheathed wire structure of FIG. 4 arranged within a further sheath to define a bundle;

FIG. 6 is a transverse cross-section of the bundle of FIG. 5 as reduced in diameter in a subsequent step;

FIG. 7 is a transverse cross-section of a plurality of the reduced-diameter bundles of FIG. 6 disposed in a further sheath to define a bundle of said bundles;

FIG. 8 is a transverse cross-section of the bundle of FIG. 7 as reduced in diameter in a subsequent step;

FIG. 9 is a transverse cross-section of a plurality of the reduced diameter bundles of FIG. 8 disposed in a further sheath to define a bundle of said bundles;

FIG. 10 is a transverse cross-section of the bundle of FIG. 9 reduced in diameter in a subsequent step;

FIG. 11 is a fragmentary diagrammatic vertical section of an apparatus for drawing the filaments in practicing the method embodying the invention;

FIG. 12 is a vertical cross-section of a tank wherein the reduced diameter bundle of FIG. 10 is disposed to be acted upon by a suitable fluid within the tank to remove the sheath material from the bundle; and

FIG. 13 is a tow of filaments embodying the invention.

In the exemplary embodiment of the invention as disclosed in the drawing, a tow generally designated 10 of filaments 11 is formed by a process wherein a plurality of relatively large diameter wires 13 are constricted or otherwise reduced in diameter in a bundle arrangement so as to result in the individual filaments 11 being of extremely small diameter while yet of substantial length. More specifically, the invention comprehends the forming of metal filaments and the like having a diameter of approximately 10 microns more or less and down to under 1 micron if desired. Thus, the invention may be employed in the formation of fine filaments including whisker-type filaments wherein substantially single crystal diameters are provided. Further, the invention comprehends the provision of such fine filaments in substantial lengths such as approximately 50 feet and over whereas heretofore whiskers have been limited to relatively short lengths due to the presence of fracture points and the like occurring in the known methods of formation thereof.

The present invention comprehends the forming of such fine filaments by constriction of a plurality of wires and more specifically by successive drawing operations. In the illustrated embodiment a wire 13 having a relatively large diameter such as over .05 inch is firstly encased in a sheath 14 of suitable matrix material. As shown in FIG. 2, the sheath may have an internal diameter slightly larger than the external diameter of the wire 13 to permit facilitated coaxial assembly thereof. As indicated briefly above, the filaments 11 may comprise metal filaments. Examples of material of which the wire 13, and thus the filaments 11 may be formed by the present process, comprise niobium, stainless steel, nickel, tungsten, iron, aluminum, carbon steel, and chrome nickel alloys, and other suitable drawable materials. The wire 13 may be suitably

formed to have an originally small diameter by any suitable method including melt forming, foil slitting, electrodeposition, vapor phase deposition, chemical deposition, powder forging, and suitable conventional wire forming processes. It is preferable that the wire 13 be relatively free of occlusions and the like to preclude formation of fracture points in the wire in the drawing process. The wire may have any suitable cross-section including the circular cross-section illustrated in FIG. 1. Further, the wire may be longitudinally uniform in cross-section or may vary as desired.

The sheath 14 may be formed of a suitable matrix material which will act generally as a fluid medium under the pressures induced at the locality of that drawing dies. Examples of such matrix material are metals such as copper and iron.

As shown in FIG. 3, the sheath 14 is firstly constricted onto the wire 13 to make a tight physical bond between the sheath and the wire so that in subsequent drawing steps the sheath 14 remains fixed relative to the wire 13 and does not stretch thereafter. The assembly 15 of the wire 13 and thusly reduced sheath 14 is next drawn down through a suitable die such as die 16 illustrated in FIG. 11. The assembly 15 is forced through the die by suitable pulling means diagrammatically illustrated at 17 in FIG. 11. The resultant reduced-diameter sheathed wire generally designated 18 is illustrated in FIG. 4.

A plurality of sheathed wires 18 are next disposed within a sheath 19 formed of a suitable matrix material which may, but need not necessarily, comprise the same material as sheath 14. As shown in FIG. 5, the sheathed wires 18 may be uniformly distributed within the sheath 19 where it is desired to obtain filaments 11 of generally uniform cross-section.

The bundle 20 of sheathed wires 18 in sheath 19 is then drawn down to define a reduced-diameter bundle generally designated 21 as shown in FIG. 6. The plurality of the reduced diameter bundles 21 may then be disposed within a further sheath 22 as shown in FIG. 7 to define a further bundle generally designated 23. The bundle 23 may then be drawn down to define a reduced-diameter bundle generally designated 24 as shown in FIG. 8. A plurality of the reduced-diameter bundles 24 may then be disposed within a further sheath 25 as shown in FIG. 9 to define a further bundle generally designated 26. The bundle 26 may then be drawn down, as shown in FIG. 10, to define a final reduced-diameter bundle generally designated 27.

The number of wires and bundles disposed within the bundling sheaths and the number of drawing steps may be varied as desired to obtain the desired resultant filament diameter; for facilitated illustration of invention we have shown three bundling and subsequent drawing steps with seven sheathed wires and bundles being disposed within the respective bundling sheaths, it being understood that more or less wires, bundles, and steps may be employed as desired.

The individual filaments 11 are obtained from the final bundle 27 by removing the matrix material which comprises the various sheaths employed in the drawing operation. As illustrated in the drawing, the respective constricting operations effected by the drawing steps cause the sheath material to substantially completely fill the voids between the wires so as to form a matrix extending substantially continuously in cross-section whereby each of the wires in the respective bundles is firmly and positively supported by the matrix material during the drawing thereof through the drawing die 16. As indicated briefly above, the matrix material preferably comprises a material capable of acting in the manner of a fluid under the pressure induced at the drawing die so as to provide improved support of the wires during the drawing operation and thereby effectively preclude the formation of discontinuities in the respective wires.

The respective filaments 11 are subsequently made to comprise a filament tow by the removal of the matrix

material in a subsequent step of the forming process. The present invention comprehends the removal of the matrix material by suitably acting on the final bundle 27 to eliminate the matrix material while allowing the filaments to remain. Thus, the invention comprehends the use of a matrix material which differs in physical characteristics from the wire material from which the filaments are formed in such a manner as to permit the ready removal of the matrix material without substantially affecting the filaments. For this purpose, the sheath-matrix material may comprise, as indicated above, copper where the filament material is stainless steel permitting the copper to be removed by treatment with suitable copper-dissolving acid, such as nitric acid, which leaves the stainless steel filaments substantially unaffected. Other methods of removal of the matrix may be employed with suitable matrix materials permitting the removal thereof such as by electrolysis, shock, melting, physical break-up as by chopping and the like.

In the illustrative example of matrix removal step, as shown in FIG. 12, the bundle 27 is disposed in a suitable tank 28 containing a body 29 of solubilizing fluid such as nitric acid, the matrix material of bundle 27 illustrated therein being copper and the filaments being stainless steel. Thus upon complete removal of the copper matrix material, the individual filaments 11 define a tow 10 of stainless steel filaments as shown in FIG. 13, each filament being separate of the other filaments and of pre-selected small diameter.

Specific examples of filament forming processes embodying the invention are as follows:

Example 1.—A wire 13 of type 302 hard drawn stainless steel having a diameter of .081 inch is inserted into a copper tube sheath 14 having a .125 inch outer diameter and a wall thickness of .020 inch. In the first step the sheath is drawn down to an outer diameter of .109 inch. The resultant sheathed wire 18 is then annealed at a temperature of approximately 1800° F. The reduced sheathed wire 18 is then subsequently drawn seriatim in a number of similar drawing and annealing steps until the final outer diameter of the sheath wire 18 is approximately .016 inch.

The .016 inch diameter sheathed wire 18 is then cut into 19 pieces and inserted into a copper sheath 19 having an outer diameter of approximately .125 inch and a wall thickness of approximately .015 inch. The sheath is then drawn down to an outer diameter of .109 inch and the assembly annealed at approximately 1800° F. The resultant bundle 20 is then drawn down in successive steps including interposed annealing steps to an ultimate diameter of .016 inch. The resultant reduced-diameter bundle 24 is then cut into 19 pieces and inserted in a copper sheath 26 similar to sheath 19. The above steps are then repeated to again reduce the bundle to a final diameter of .040 inch wherein the individual wires have been reduced in diameter to define filaments having a diameter of approximately .0005 inch. Alternatively, the final draw may be to a diameter of .032 inch to produce filaments of approximately .0004 inch diameter, or to a diameter of .028 inch to produce filaments of approximately .00032 inch (8 microns) diameter. The matrix copper material is then dissolved in tank 28 with the nitric acid 29 being maintained at a temperature of approximately 120° F.

Example 2.—A stainless steel wire 13 having a diameter of .083 inch is inserted into a copper tube sheath 14 having a .125 inch outer diameter and a wall thickness of .020 inch. In the first step the sheath is drawn down to an outer diameter of .109 inch. The resultant sheathed wire 18 is then annealed at a temperature of approximately 1800° F. The reduced sheathed wire 18 is then subsequently drawn seriatim in a number of similar drawing and annealing steps until the final outer diameter of the sheathed wire 18 is approximately .016 inch.

The .016 inch diameter sheathed wire 18 is then cut into 7 pieces and inserted into a copper sheath 19 having an outer diameter of approximately .072 inch and a wall

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thickness of approximately .009 inch. The sheath is then drawn down to an outer diameter of .065 inch and the assembly is annealed at approximately 1800° F. The resultant bundle 20 is then drawn down in successive steps, including interposed annealing steps, to an ultimate diameter of .016 inch. The resultant reduced-diameter bundle 24 is then cut into 7 pieces and inserted in a suitable copper sheath 26 similar to sheath 19. The above steps are then repeated to reduce the bundle to a final diameter of .016 inch wherein the individual wires have been reduced in diameter to define filaments having a diameter of approximately .00032 inch. Alternatively, the final draw may be to a diameter of .032 inch to produce filaments of approximately .00047 inch diameter, or to a diameter of .025 inch to produce filaments of approximately .0004 inch (10 microns) diameter. The matrix copper material is then dissolved in tank 28 with the nitric acid 29 being maintained at a temperature of approximately 120° F.

Example 3.—A wire 13 of type 302 hard drawn stainless steel wire having a diameter of .083 inch is inserted into a copper tube sheath 14 having a .125 inch outer diameter and a wall thickness of .020 inch. In the first step the sheath is drawn down to an outer diameter of .109 inch. The resultant sheathed wire 18 is then annealed at a temperature of approximately 1800° F. The reduced sheathed wire 18 is then subsequently drawn seriatim in a number of similar drawing and annealing steps until the final outer diameter of the sheathed wire 18 is approximately .025 inch.

The .025 inch diameter sheathed wire 18 is then cut into 37 pieces and inserted into a copper sheath 19 having an outer diameter of approximately .250 inch and a wall thickness of approximately .030 inch. The sheath is then drawn down to an outer diameter of .225 inch and the assembly is annealed at approximately 1800° F. The resultant bundle 20 is then drawn down in successive steps, including interposed annealing steps, to an ultimate diameter of .025 inch. The resultant reduced diameter bundle 24 is then cut into 37 pieces and inserted in a suitable copper sheath 26 similar to sheath 19. The above steps are then repeated to reduce the bundle to a final diameter of .049 inch wherein the individual wires have been reduced in diameter to define filaments having a diameter of approximately .0005 inch. Alternatively the final draw may be to a diameter of .035 inch to produce filaments of approximately .004 inch diameter, or to a diameter of .028 inch to produce filaments of approximately .0003 inch (7½ microns) diameter. The matrix copper material is then dissolved in tank 28 with the nitric acid 29 being maintained at a temperature of approximately 120° F.

Example 4.—A wire 13 of alloy 270 soft nickel having a diameter of .063 inch is inserted into a copper tube sheath 14 having a .095 inch outer diameter and a wall thickness of .015 inch. The sheathed wire is drawn seriatim in a number of drawing and annealing steps until the final outer diameter of the sheathed wire 18 is approximately .016 inch.

The .016 inch diameter sheathed wire is then cut into 19 pieces and inserted into a copper sheath 19 having an outer diameter of approximately .125 inch and a wall thickness of approximately .015 inch. The bundle 20 is drawn down in successive steps to an ultimate diameter of .018 inch. The reduced diameter bundle is then cut into 19 pieces and inserted in a suitable copper sheath 26 similar to sheath 19. The above steps are then repeated to again reduce the bundle to a final diameter of .028 inch wherein the individual wires have been reduced in diameter to define filaments having a diameter of approximately .0004 inch (10 microns). The matrix copper material is then dissolved in tank 28 with the nitric acid 29 being maintained at a temperature of approximately 120° F.

Example 5.—A wire of type 304 hard drawn stainless

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steel having a diameter of .062 inch is inserted into a copper tube sheath 14 having a .095 inch outer diameter and a wall thickness of .015 inch. The sheathed wire 18 is then subsequently drawn seriatim in a number of similar drawing and annealing steps until the final outer diameter of the sheathed wire 18 is approximately .016 inch.

The .016 inch diameter sheathed wire 18 is then cut into 19 pieces and inserted into a copper sheath 19 having an outer diameter of approximately .125 inch and a wall thickness of approximately .015 inch. The sheath is then drawn down to an outer diameter of .109 inch and the assembly is annealed at approximately 1800° F. The resultant bundle 20 is then drawn down in successive steps, including interposed annealing steps, to an ultimate diameter of .016 inch. The resultant reduced-diameter bundle 24 is then cut into 19 pieces and inserted in a suitable copper sheath 26 similar to sheath 19. The above steps are then repeated to reduce the bundle to a diameter of .028 inch. The reduced bundle is then cut into 7 pieces and inserted into a copper sheath having an outer diameter of .125 inch and a wall thickness of .015 inch. The bundle is then drawn down by successive steps to a diameter of .016 inch. This reduced-diameter bundle is then cut into 19 pieces and inserted into a copper sheath having an outer diameter of .125 inch and a wall thickness of .015 inch. This bundle is then drawn down by successive steps to a final diameter of .032 inch wherein the filaments 11 have a diameter of approximately .00010 to .00012 inch (2½ to 3 microns).

Example 6.—A wire 13 of type 304 stainless steel having a diameter of .062 inch is inserted into a low carbon steel tubular sheath 14 having an outer diameter of .125 inch and a wall thickness of .023 inch. The sheathed wire is drawn seriatim in a number of drawing and annealing steps until the final outer diameter thereof is approximately .020 inch.

The .020 inch sheathed wire 18 is then cut into 19 pieces and inserted into a low carbon steel sheath 19 having an outer diameter of approximately 1.56 inches and a wall thickness of approximately .023 inch. The bundle 20 is drawn down in successive steps to an ultimate diameter of .020 inch. The reduced-diameter bundle is then cut into 19 pieces and inserted into a copper sheath 26 similar to sheath 19. The above steps are then repeated to reduce the bundle to a final diameter of .025 inch wherein the individual filaments have a diameter of approximately .0004 inch (10 microns).

The resultant filaments 11 by virtue of their extremely small diameters have textile characteristics in that they are highly compliant (i.e. they will bend around their own diameter without a permanent set), are flexible, and may be used in conventional textile machinery for forming fabrics and the like. The filaments may be formed in substantial lengths such as over 50 feet. Such continuous filaments are highly desirable in fabric formation as compared to the short staple fibers obtainable in other filament forming processes such as cold and hot drawing, cold and hot swaging, cold and hot rolling, and cold and hot extrusion processes.

The tows 10 may be provided with the individual filaments 11 therein having a preselected twist by suitably twisting the bundles during the drawing steps. By suitably annealing the twisted drawn bundle, the twisted arrangement of the filament may be permanently set therein. Thus, by suitably twisting the individual bundles of the multiple bundles 23 and 26 substantially complete elimination of twisting forces in the composite multiple bundle may be obtained. Still further, by providing the wires 13 with varying diameter in the longitudinal direction, the resultant filaments may correspondingly have varying diameters along their longitudinal extent as desired. As the resistivity of the wires is a function of the cross-section diameter of the wires controlled resistivity may be obtained. As the wires 13 are twisted about the longitudinal axis of the bundle, the resultant filaments are formed in

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spaced concentric helices wherein the matrix material maintains the spacing thereof until removed in the leaching step.

As indicated briefly above, the inventive concept comprehends a limiting of the constriction of the filaments to have at least one crystal thickness to provide improved high strength filaments.

While we have shown and described certain embodiments of our invention, it is to be understood that it is capable of many modifications. Changes, therefore, in the construction and arrangement may be made without departing from the spirit and scope of the invention as defined in the appended claims.

We claim:

1. A tow of metal filaments each having a maximum cross-section of less than approximately 10 microns and a length of greater than approximately 50 feet and having a trace amount of a different material diffused in the outer surface thereof.

2. The tow of filaments of claim 1 wherein the filaments are substantially free of surface burnishing.

3. The tow of filaments of claim 1 wherein the filaments are of substantially one crystal thickness throughout the length thereof.

4. A tow of filaments as set forth in claim 1 wherein

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the filaments are substantially identically cold worked at each transverse cross-section of the tow.

5. A tow of filaments as set forth in claim 1 wherein the filaments have a substantially uniform total cross-sectional area in different planes taken at positions spaced axially along the tow.

6. A tow of filaments as set forth in claim 1 wherein the filaments are formed in spaced concentric helices.

7. The tow of metal filaments of claim 1 wherein said different material comprises a metallic material.

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