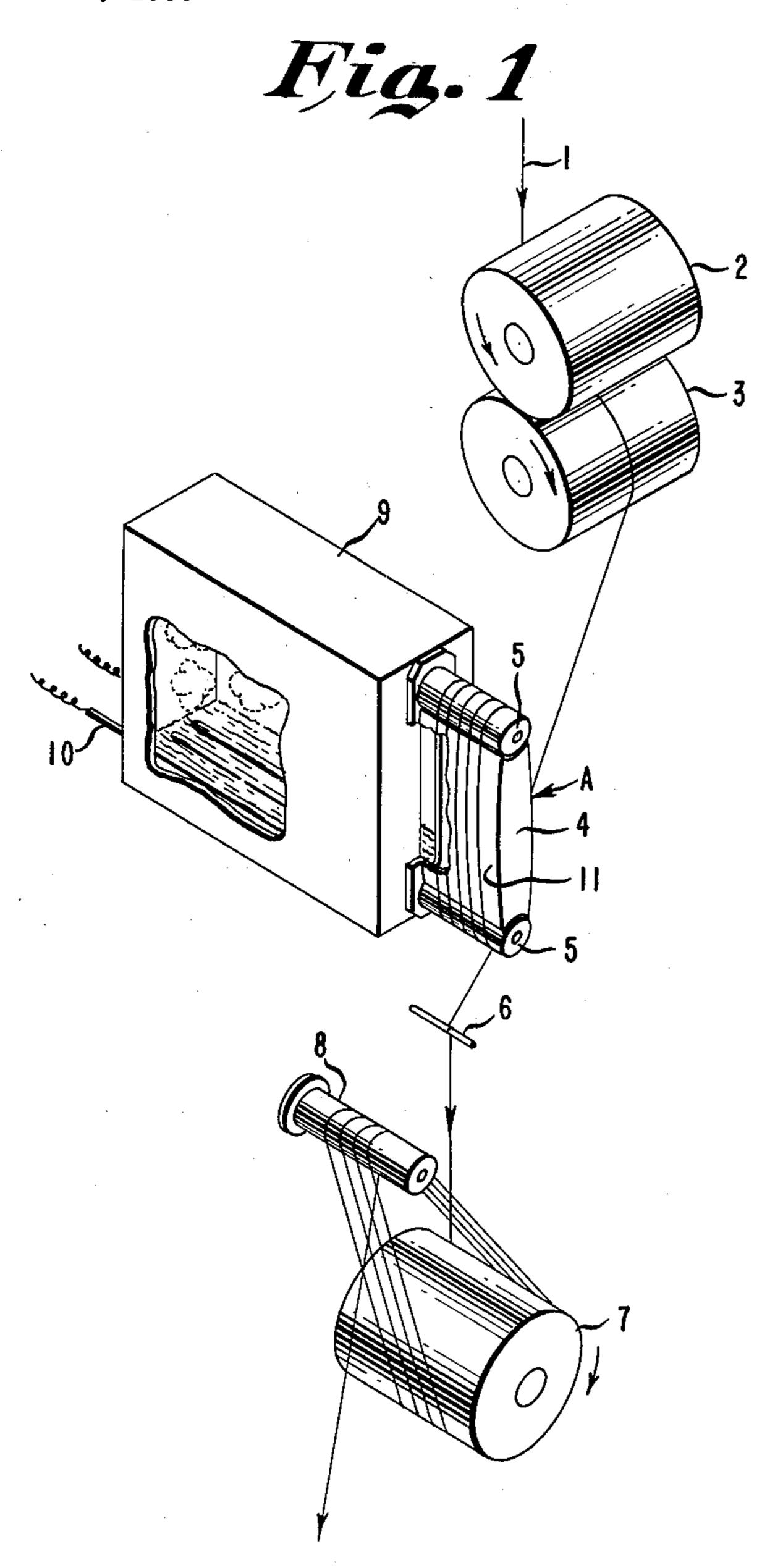
3,101,990

PROCESS OF DRAWING FILAMENTARY STRUCTURES

Filed Oct. 13, 1960

2 Sheets-Sheet 1

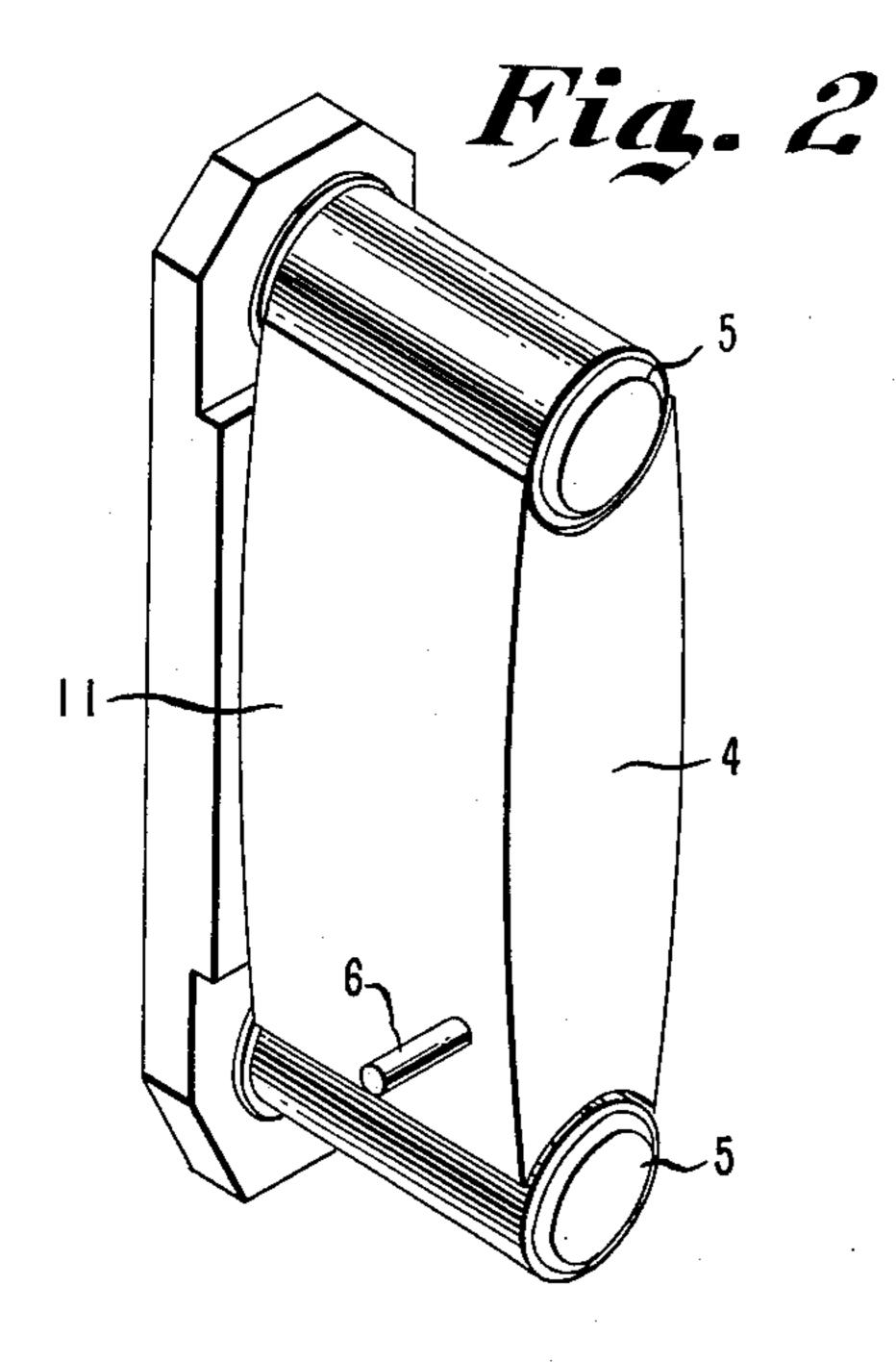


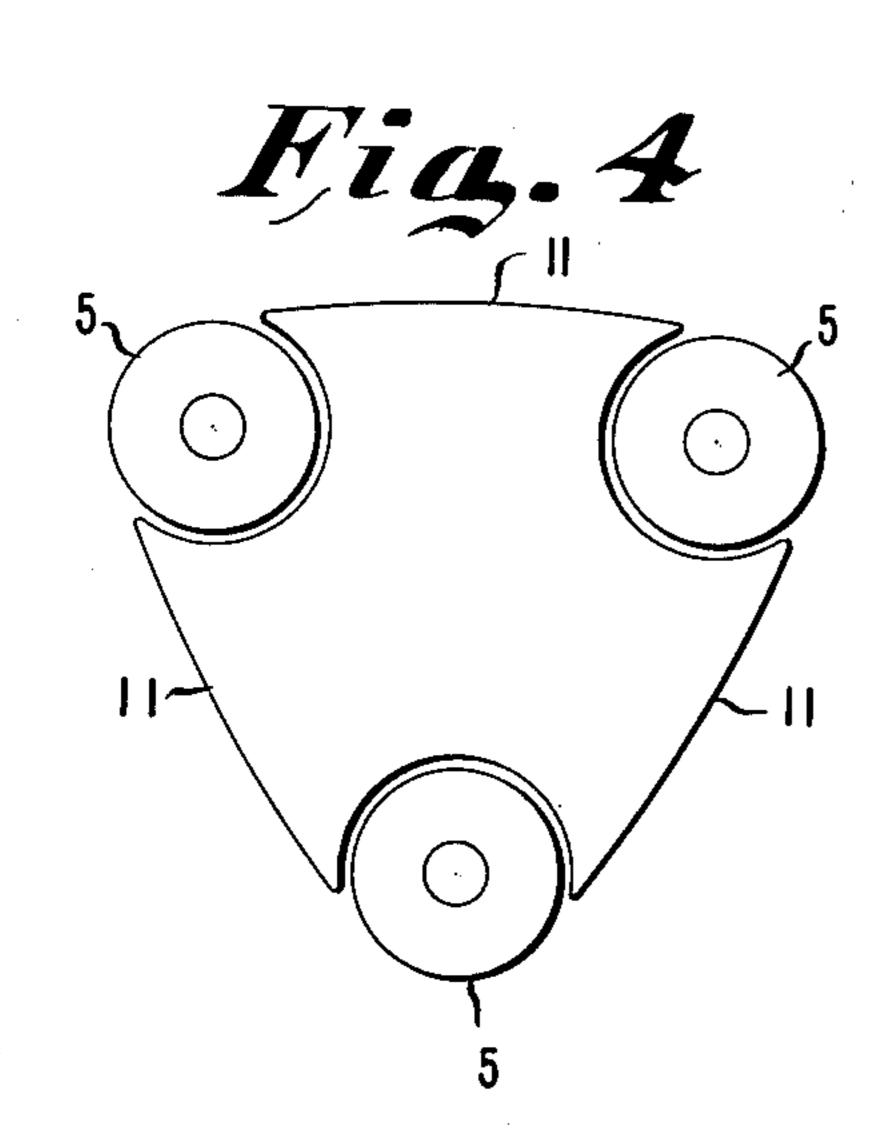
INVENTOR HAROLD H. HEIGHTON

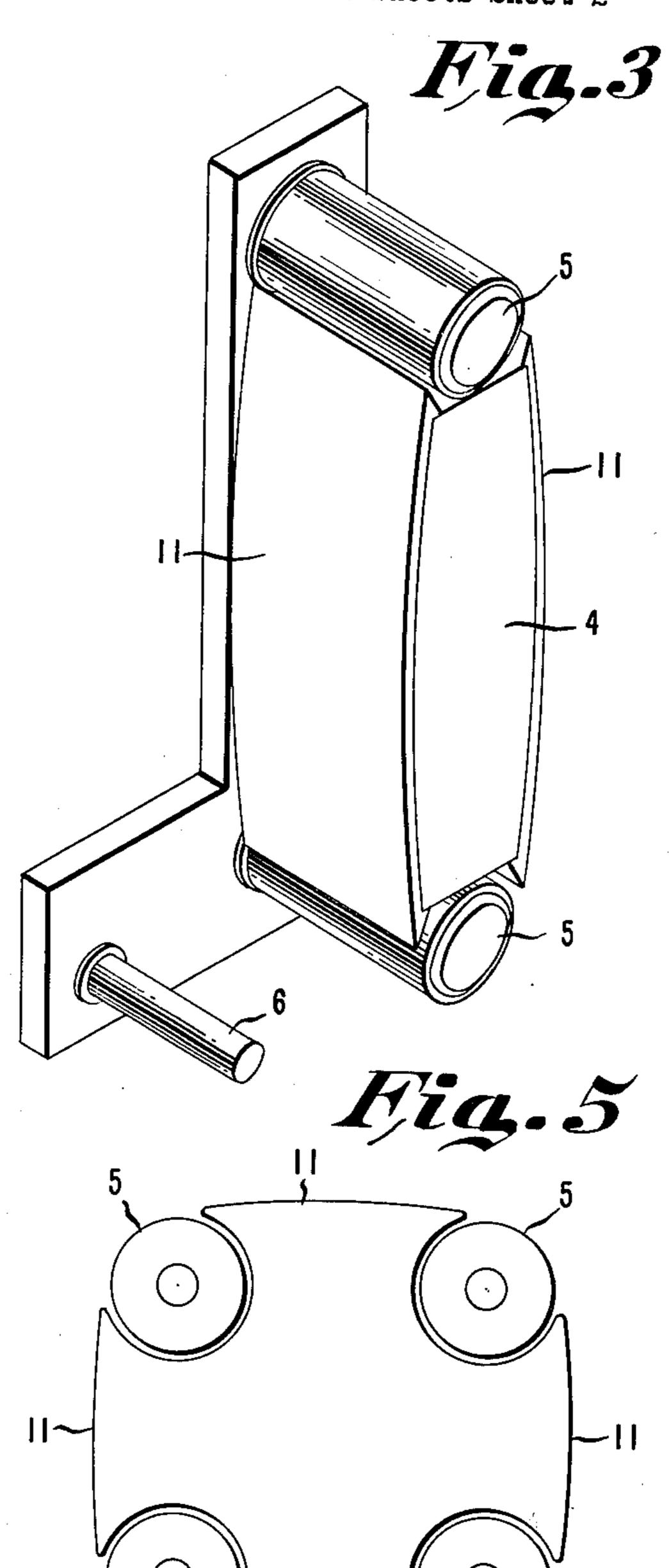
BY Frank C. Hillerg ATTORNEY PROCESS OF DRAWING FILAMENTARY STRUCTURES

Filed Oct. 13, 1960

2 Sheets-Sheet 2







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3,101,990 PROCESS OF DRAWING FILAMENTARY STRUCTURES

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This invention relates to the manufacture of filamentary 10 structures made from linear superpolymers. More particularly it relates to a new and improved method for drawing filaments, yarns, threads, and the like made from synthetic linear polyesters.

In the manufacture of filamentary material from syn- 15 thetic linear polyesters, such as the polyethylene terephthalates disclosed by Whinfield and Dickson in U.S. Patent 2,465,319, it is customary to extrude molten polymer through a spinneret, solidify the filaments thus formed by cooling, and then cold draw the filaments to 20 several times their original length in order to enhance their physical properties such as tenacity, elongation, and shrinkage. In order to carry out the drawing of polyester yarns and filaments it is usual to heat the yarns and filaments to an elevated temperature, and several 25 procedures have been proposed for carrying out this operation. For example, polyester yarns have been drawn by using a heated feed roll or a heated draw roll, or by inserting a hot plate or a hot pin between cold feed roll and cold draw roll. Polyester yarns also have been 30 drawn in two stages as, for example, by first drawing over a hot pin and then further drawing over a hotter plate.

In the commercial manufacture of polyester textile yarns the drawing step is extremely critical since non-uniform drawing leads to non-uniform dyeing, which in 35 turn leads to commercially unacceptable dyed fabrics. Previously known drawing processes have not successfully overcome this obstacle. Furthermore, previously known processes have been found to be limited in speed of operation. A substantial increase in speed has always 40 resulted in either a markedly less uniform product, excessive broken filaments, or a complete break down of the threadline.

An object of this invention therefore is to provide an improved method for the drawing of synthetic linear 45 polyester filamentary structures.

Another object is the provision of a drawing process for producing polyester yarns and filaments with greatly improved uniformity of dye receptivity.

Another object is to provide a process for drawing 50 polyester yarns at higher drawing speeds.

Still another object is the provision of means for a more uniform application of heat to polyester yarns in the drawing process. These and other objects will more clearly appear from the detailed description which follows.

These objects are realized by the present invention which comprehends a process for drawing synthetic linear polyester filamentary structures in which the undrawn filamentary structure, protected by a lubricating finish, 60 is passed at a uniform rate to and over a hot surface maintained at a temperature between the crystalline second order transition temperature and about 45° above the crystalline second order transition temperature, heated on the hot surface for a period of at least about 0.06 sec. 65 and not more than about 3 sec. while being subjected to a stretching tension in the range between about 10% and about 40% of the natural drawing tension at the temperature of the hot surface without significant drawing, and then drawn by passing it to and around a snubbing 70 element where drawing tension is applied.

This process may be carried out on a yarn drawing

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apparatus comprising a means for supplying yarn at a uniform rate; a multisided yarn heating shoe having at least two arcuate surfaces and with low drag means for carrying yarn from one arcuate surface to a second arcuate surface; means for heating the shoe; a snubbing element; means for applying drawing tension to the yarn; and means for winding up the drawn yarn.

The invention may be more easily understood by referring to the accompanying drawings wherein FIGURE 1 is a plan view showing one arrangement of elements combined in accordance with the principles of this invention, FIGURES 2 and 3 are isometric views which illustrate alternative arrangements of the heating and snubbing elements, and FIGURES 4 and 5 are diagrammatic sectional views which illustrate further alternate modifications in the form of the heating element. In all figures the numerals refer to similar elements.

The description of the apparatus and method of carrying out the invention may be illustrated simultaneously by reference to FIG. 1. The undrawn synthetic linear polyester filamentary structure indicated as 1, which is protected by a lubricating finish, is fed at a constant rate by feed rolls 2 and 3 to and around (several wraps) a heating assembly A consisting essentially of a hot twosided shoe 4 fitted at each end with integrally mounted freely rotating idler rolls 5 and outwardly arched heating surfaces 11, the whole assembly being heated to a suitable temperature by vapor from boiler 9 which is heated by electrical heaters 10, or by any other known means, then around a small stationary snubbing pin 6 where drawing is accomplished, then around draw roll 7 with its accompanying idler roll 8, and then to a yarn take-up device, not shown. The hot vapor is transferred directly to the heating assembly A as shown.

The term "synthetic linear polyester" used above refers to and includes fiber-forming linear polyesters in which at least 85% of the recurring structural units are units of the formula

wherein G represents the hydrocarbon residue of a glycol containing from 2 to 10 carbon atoms which is attached to the adjacent oxygen atoms by saturated carbon atoms. Thus, the radical -G— is a hydrocarbon radical of 2–10 carbon atoms and may be a polymethylene radical, $-(CH_2)_n$ —, where n is a number from 2 to 10, a radical such as $-(CH_2)_m$ —A— $(CH_2)_m$, where m and m' are 1 or 2 and -A— represents a divalent cycloaliphatic radical, or the like.

The linear terephthalate polyesters may be prepared by reacting terephthalic acid or an ester forming derivative thereof with a glycol, G(OH)₂, where —G— is a radical as defined above, to form the bisglycol ester of terephthalic acid, followed by polycondensation at elevated temperature and reduced pressure with elimination of excess glycol. Examples of suitable glycols include ethylene glycol, butylene glycol, decamethylene glycol, and trans bis - 1,4 - (hydroxymethyl)cyclohexane. Isophthalic acid and similar acids which form linear fiberforming polyesters with these glycols may also be used in making the filamentary structures.

The "filamentary structures" referred to above are best exemplified by multifilament textile denier yarns having a yarn denier, after drawing, in the range 20 to 250 (2.2 to 28 Tex.).

The term "lubricating finish" refers to a coating of a suitable textile lubricant, usually 0.4 to 2.5% by weight, such as a mineral, vegetable, or animal oil as, for example, a light mineral oil, olive oil, or sperm oil, or a processed oil such as a sulfonated castor oil, or a synthetic ma-

terial such as a silicone oil. The lubricant may be combined with an anti-static agent in accordance with principles known to the art. Undrawn yarn entering the drawing process of this invention must be protected by a lubricating finish, otherwise the threadline breaks down 5 completely or is characterized by numerous broken filaments.

The term "second-order transition temperature" used above is defined by Pace (U.S. 2,556,295) and is found, for example, to be above about 57° C. for crystalline polyethylene terephthalate having an intrinsic viscosity above 0.30, and to be about 80° C. for intrinsic viscosities above about 0.50. The temperature of the heating step in the process of this invention must fall in the range between the second-order transition temperature and about 45° C. above said transition temperature. Temperatures below this range result in non-uniform drawing and numerous broken filaments, while temperatures appreciably above this range give operability difficulties because of the sticking of the polyester structure to the heating surfaces.

The term "natural drawing tension" used above means the tension in the threadline at the draw point when a yarn is being drawn at its natural draw ratio under steady state conditions. Natural draw ratio as used herein is defined by Marshall and Thompson, J. Applied Chemistry 4, 25 145 (1954).

The heating assembly in the preferred embodiment of the invention comprises a heated shoe fitted at two ends with freely rotating idler rolls and having two exposed outwardly facing convex surfaces 11 for contacting and 30 heating the yarn. The assembly is arranged in such a way that a major portion of the change of direction in the yarn path around the heating assembly occurs on the idler rolls and only a minor portion occurs on the heated shoe surface. The heated shoe is best made of a ma- 35 terial with good heat transfer qualities as, for example, aluminum, copper, or brass. The shoe may be heated by the insertion of electrical heaters directly into the interior of the shoe, or more advantageously the shoe may be hollow and connected directly to a boiler as shown in FIG. 1 so that it is heated by vapors of a boiling liquid. The outer surface of the shoe engaging in sliding contact with the yarn should combine good heat transfer properties with low frictional properties, e.g. as may be obtained by a matte finished chrome-plated surface.

For continuous commercial operations the heated shoe is advantageously fitted with replaceable yarn contacting surfaces 11 as, for example, by brazing to the sides of the shoe thin formed copper sheets which are sand-blasted and chrome-plated on the exposed surfaces to give the desired matte finish. Such an arrangement is illustrated in FIGURE 3 wherein the number 11 indicates the replaceable surface. The eroding qualities of a fast moving threadline are well known and it is obviously an advantage to be able to replace only a thin section of worn surface instead of the complete shoe.

The idler rolls 5 of the heating assembly A (FIGURE 1) are mounted on extensions of the base of the heated shoe 4 so that the rolls are heated by direct conduction through the mounting assembly as well as by contact with the heated yarn. In operation the temperature of the idler rolls is substantially the same as that of the heated shoe. The axes of rotation of the idler rolls are not parallel but are canted in such a manner that successive wraps of yarn around the heating assembly are kept in separate paths thereby preventing overlapping and resultant fouling of the threadline. The idler roll mounting arrangement may be such as to allow adjustment of the angle of cant, thereby allowing an adjustment in the distance between adjacent yarn wraps on the heating assembly.

The idler rolls themselves must satisfy certain critical requirements as to the amount of frictional drag imposed on the threadline. For example, in one test for suitable idler rolls a pair of rolls inserted in a loop of a threadline 75

moving at 750 y.p.m. must not cause a tension drop of more than about .086 gm. per denier in the threadline. Thus the maximum allowable tension drop may vary from about 12 grams to about 45 grams for undrawn yarn deniers in the range 140 to 525 (16 to 60 Tex.). In another test for suitable idler rolls the unbraked stopping time from a speed of 9000 r.p.m. must be no less than about 0.40 minute for undrawn yarn of 140 denier (16 Tex.), and about 0.25 minute for undrawn yarn of 525 denier (60 Tex.).

The frictional drag imposed by the heating assembly on the threadline must be low enough so that the tension in the threadline between the heating assembly and the snubbing pin is never more than about 40% of the natural drawing tension for the yarn at the temperature of the heating assembly. Higher tensions lead to a non-uniform product. Furthermore, the tension in the zone between the feed rolls and the heater must be at least 10% of the drawing tension to prevent yarn wraps on the feed rolls.

Birefringence measurements made on yarn samples taken from the zone between heating assembly and draw pin will be substantially the same as those made on the supply yarn, indicating that no appreciable change in orientation occurs in the yarn as it traverses the heating assembly.

The size of the heating assembly and the number of wraps taken around it should be adjusted so that the heating time of the yarn is sufficient to heat the yarn uniformly, thereby avoiding uneven drawing and the resulting increase in dye streaks in fabric prepared from such yarns. Under nearly optimum process conditions the heating time will vary with the temperature of the heating assembly according to the following formula:

$$T^{\circ} = \frac{4.94648 - \log(100H)}{0.03335} \pm 9$$

where T is the heating assembly temperature in degrees centigrade, and H is the heating time in seconds for the yarn on the heating assembly.

Preferably, it is desirable to use higher temperatures and lower heating times for the lighter denier yarns. Heating times and heating assembly temperatures found particularly suitable were as follows:

	De	enier	Drawing Speed,	Heating Time,	Shoe Temp.,		
	Drawn	Undrawn	yards/ min.	Seconds	w./yarn, C.		
•	70 40 70 140 150 220	245 120 245 616 660 1, 179	1, 200 750 750 545 545 302	0. 20 0. 40 0. 47 0. 80 0. 80 1. 80	105 100 97 94 90 80		

The exact location of the snubbing pin 6 of FIGURE 1 is a matter of choice. It may be mounted several inches away from the heating assembly A, or if desired it may be mounted directly in the face of the heated shoe 4 as shown in FIGURE 2. Separate means of heating the snubbing pin may be necessary to obtain optimum temperature conditions if the pin is not mounted directly in the shoe. The snubbing pin may be made of any one of several materials disclosed by Babcock in U.S. 2,289,-232. However, it is necessary that the surface of the pin be of such a character that the coefficient-of-friction falls in the range from about 0.10 to 0.40, depending somewhat upon the denier of the yarn being drawn, the operating speed, and the pin diameter. In a preferred embodiment of this invention the snubbing pin is a bonded TiO₂ pin with a fine matte surface. If desired, the single pin of FIGURE 1 may be replaced by a set of two or more pins acting in concert, or by other equivalent snubbing elements.

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The coefficient of friction of a snubbing pin surface, as used herein, is measured by pulling drawn yarn around the pin in a 180° turn at a speed of 225 y.p.m. Input tension is held constant at 9 grams with a suitable tensioning device, and tension drop across the pin is measured with a Shirley General Purpose friction tester (Longworth Scientific Instruments Co., England), which reads directly in terms of coefficient-of-friction.

Some variation in final yarn properties may be made by adjusting snubbing pin diameter. For preferred low 10 shrinkage (8 to 15%) no-crimp polyester yarn it is necessary to maintain pin diameter in the range from about 3/16 inch to about 3/4 inch. A pin appreciably smaller than 3/16 inch, e.g. 1/8 inch, may be used to produce a

yarn with a spontaneous crimp.

The draw ratios used in the process of this invention, which are determined by the relative speeds of the feed rolls and draw rolls, are substantially equivalent to those disclosed in the prior art and will depend upon the preorientation in the undrawn yarn which in turn depends in 20 part on the spinning speed used in preparing the undrawn yarn. Good operability has been obtained with undrawn yarns having a measured birefringence value as high as .0115.

The following examples are illustrative of the practice 25 of this invention.

EXAMPLE I

Apparatus for drawing yarn is set up as shown in FIG-URE 1. The hot shoe 4, made from a copper block, has two outwardly facing heating surfaces 11/4 inches wide (at right angles to the yarn path) and 2.75 inches long (parallel to the yarn path). These heating surfaces are sections of a cylinder having a radius of 18 inches and are spaced apart so that lines extrapolated from the surface parallel to the yarn path are tangent to idler rolls 5. The yarn contacting surfaces of the hot shoe are prepared by blasting with alumina grit followed by plating with 0.005 inch of industrial hard chrome to obtain a surface roughness measurement of about 115 micro-inches average deviation in surface radial contour. The shoe 4 has a large internal cavity which is connected through an opening in the mounting plate at one side of the shoe to a boiler assembly 9, containing dichlorodifluoromethane, which is fitted with electrical heaters 10 and a pressure controller for regulating temperatures. The mounting plate of the hot shoe has extensions suitable for mounting rolls 5. Rolls 5 are 1/8-inch diameter separator rolls having carefully chosen low friction bearings such that a pair of these rolls inserted in a loop of yarn moving at 750 y.p.m. causes a tension drop of only 8 grams. The rotational axes of the rolls are canted toward each other at an angle of 9°.

The snubbing pin 6 is mounted 134 inches from the heating assembly and consists of a 3/16-inch diameter bonded TiO₂ pin having a fine matte surface with a coefficient-of-friction of .22.

Polyethylene terephthalate polymer having an intrinsic viscosity of 0.65 is melt spun into a yarn having 34 filaments with an average birefringence of 0.0090 and a total undrawn denier of 245 (27 Tex.). The yarn, protected by a finish consisting mainly of a light mineral oil, is fed by feed rolls 2 and 3 of FIGURE 1 at a speed of 214 y.p.m. to heating assembly A maintained at 100° C. The yarn makes 5 wraps around the heating assembly and then makes one wrap around snubbing pin 6 before proceeding to draw roll 7 which is operating at a surface speed of 750 y.p.m. Four wraps are made around draw roll 7 and its accompanying idler roll 8 in order to prevent slippage. The drawn yarn then proceeds to a yarn take-up device. Examination of the thread-line during the operation of the drawing process reveals that the yarn remains undrawn until it reaches the snubbing pin. The tension in the threadline is found to be about 8 grams between feed roll and hot shoe, about 20 grams between hot 75 6

shoe and snubbing pin, and about 55 grams between snubbing pin and draw roll. The drawn yarn has a denier of 70.0 (7.8 Tex.), an elongation of 18%, and a tenacity of 4.6 g.p.d.

Yarn prepared as described above is woven in a 40 inch satin fabric. The fabric is scoured ½ hour at 160° F., heat set 8 seconds at 325° F., and dyed with Celanthrene Brilliant Blue FFS (C. I. 61,505) for 1 hour at 205° F. The resulting fabric is remarkably free of short length dyeing non-uniformities.

Substantially equivalent results are obtained when drawing is carried out with the heating assembly maintained at temperatures of 86° C. and 105° C. Temperatures higher than about 125° C. give difficulty because of yarn sticking to the hot shoe surface. Temperatures lower than about 80° C. result in non-uniform dyeing properties.

Substantially equivalent results are obtained when the number of yarn wraps on the heating assembly is 3 or 7, and also when the wind-up speed in the drawing operation is 200 y.p.m. or 1300 y.p.m. (at constant draw ratio).

EXAMPLE II

Apparatus for drawing yarn is set up as shown in FIG-URE 1 except that the snubbing pin is inserted in the face of the hot shoe as shown in FIGURE 2. The hot shoe, made from a chrome plated copper block, has heating surfaces 1½ inches wide and 2¼ inches long with a curvature equivalent to that of a cylinder having a radius of 9 inches. The heating surfaces are blasted with alumina grit and chrome plated until a measurement of surface roughness indicates an average deviation of about 40 microinches in surface radial contour. The 5/8-inch diameter rolls 5 mounted on the base plate of the hot shoe have carefully chosen low friction bearings such that a pair of these rolls inserted in a loop of yarn moving at 750 y.p.m. causes a tension drop of only 6 grams in the threadline. The rotational axes of the rolls are canted toward each other at an angle of 8°. The snubbing pin mounted in the face of the hot shoe is a 3/16-inch diameter bonded TiO2 pin having a matte finished surface characterized by a coefficient-of-friction of 0.26 as measured by the test for coefficient-of-friction described herein.

A polyester of polyethylene terephthalate incorporating 2 mol percent sodium 3,5-di(carbomethoxy)-benzene sulfonate and having an intrinsic viscosity of 0.48 is melt spun to give a yarn having a denier of 230 (26 Tex.), a birefringence of 0.0075 and a crystallinity level of about 0%. This undrawn yarn, protected by a lubricating finish consisting mainly of a sperm oil, is fed by feed rolls 2 and 3 of the drawing apparatus at a speed of 395 y.p.m. to the heating assembly maintained at 95° C. The yarn makes six wraps around the hot shoe/separator roll assembly and then makes one wrap around the snubbing pin 6 before proceeding to draw roll 7 which is operating at a surface speed of 1300 y.p.m. Four wraps are made around draw roll 7 and its accompanying idler roll 8 in order to prevent slippage. The drawn yarn then proceeds to a yarn take-up device. Examination of the threadline during the operation of the drawing process reveals that the yarn remains undrawn until it reaches the snubbing pin. The product yarn has a denier of 70.1 (7.8 Tex.), an elongation of 21%, and a tenacity of 4.1 g.p.d.

Yarn prepared as described above is woven into a 48-inch taffeta fabric. The fabric is scoured ½ hour at 160° 65 F., heat set 8 seconds at 325° F., and then dyed with Celanthrene Brilliant Blue FFS (C.I. 61,505) for 1 hour at 205° F. Inspection of the dyed fabric reveals that it is completely free of short length dyeing non-uniformities.

An attempt to draw the same yarn using a heated pin but no preheating plate at a speed of 1300 y.p.m. was completely unsuccessful. Operated at 545 y.p.m. the hot-pin drawing process gave yarn which exhibited visible short length dyeing defects when woven into fabric and dyed as before.

In like manner the process of this example gives a uni-

formly dyeable yarn at speeds up to 1000 y.p.m. from 34-filament yarn having a drawn denier of 150 (17 Tex.) whereas the hot pin drawing process is limited to a speed of 350 y.p.m. or below.

EXAMPLE III

Apparatus for drawing yarn is set up as in Example I except that the hot-shoe 4, made from a copper block, has heating surfaces 63/8" long with a radius of curvature of 51". The heating surfaces are blasted with alumina grit and plated with about 0.005 inch of industrial hard chrome until a measurement of surface roughness with a Surfindicator indicates an average deviation of about 115 microinches in surface radial contour. The shoe is heated with six 50-watt electrical cartridge heaters controlled by a 15 bimetallic thermo-regulator.

Melt spun polyethylene terephthalate yarn consisting of 34 filaments protected by finish consisting mainly of a light mineral oil is drawn on the above apparatus at a wind-up speed of 1200 y.p.m. under the conditions summarized in the following table (Table I). Samples are dyed as in Example I. It is obvious from the data in the table that heating times of 0.14 second and below are not sufficient to give the desired dyeing uniformity for this particular set of process conditions.

An attempt to operate this process with a hot shoe having a mirror-finished surface or with a surface having an average deviation in contour of less than 25 microinches resulted in failure because of the high friction developed between yarn and shoe surface.

Table I

Undrawn denier	249 (28) 105 3 0.2 3/16 71.6 (8.0) 23.3 4.5	249 (28) 105 2 0.14 3/16 71.5 (8.0) 17.8 4.1 Excessive	350 (39) 100 4 0. 28 1/4 100. 7 (11) 23. 3 4. 3	350 (39) 100 2 0.14 100.8 (11) 18.5 4.1 Excessive	4
No. wraps on heater Heating time, seconds Pin diameter, inches Drawn denier (Drawn Tex.) Elongation	0.2 $\frac{3}{16}$ 71.6 (8.0) 23.3	0. 14 3/16 71. 5 (8. 0) 17. 8 4. 1 Exces-	0. 28 1/4 100. 7 (11) 23. 3 4. 3	100. 8 (11) 18. 4 Exces	13 13 1-

Synthetic linear polyester yarns drawn according to the process of this invention are found to be remarkably free from dyeing non-uniformities and in particular those short length dyeing non-uniformities stemming from the drawing process which have plagued the development of polyester yarns since their original discovery. Furthermore, the process of this invention may be operated at wind-up speeds at least as high as 1300 y.p.m. for many yarn deniers without loss of uniform dyeing properties, whereas commercial drawing processes have been limited to much lower speeds; i.e. usually at speeds about ½ as great but frequently even lower speeds.

The two outstanding advantages of the form of the heating assembly described above are the attainment of relatively long heating times in a small amount of equipment space, and the ease of maintaining temperature uniformity over short heating surfaces as compared with the 60

difficulty of obtaining such uniformity over a long hot plate for example.

It will be apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, and therefore it is not intended to be limited except as indicated in the appended claims.

This application is a continuation-in-part of my copending application Ser. No. 785,734, filed January 8, 1959, now abandoned.

I claim:

1. The process of drawing filamentary structures made from synthetic linear polymers which comprises passing the said structure, having a lubricating finish, at a uniform rate over a hot surface, maintained at a temperature between the crystalline second order transition temperature of the polymer and about 45° C. above the said transition temperature for a period of time from about 0.06 second to about 3.0 seconds, and subjecting the said structure to a stretching tension while passing it over the said hot surface in the range between about 10% and about 40% of the natural drawing tension at the temperature of the hot surface without significant drawing, and then passing it through a snubbing zone where drawing tension is applied.

2. The process of claim 1 in which the synthetic linear polymer is a polyester containing at least 85% of recurring structural units of the formula:

in which G is an organic radical containing from 2 to 10 carbon atoms.

3. The process of claim 2 in which the polyester is a glycol terephthalate.

4. The process of claim 3 in which the said polyester has a second order transition temperature of about 57° C. and an intrinsic viscosity of above about 0.30.

5. A rapid hot draw process for a multi-filament yarn of synthetic fiber-forming polyester which comprises passing a previously undrawn yarn of the polyester over feed and draw rolls, heating the yarn under low tension and without significant drawing as it passes between the said rolls by slip contacting it with a curved convex surface maintained at a temperature substantially equal in effect to the second-order transition temperature of the polyester, and then drawing the yarn at a temperature between the second-order transition temperature and 45° C. above by passing the yarn over a snubbing pin and draw roll.

References Cited in the file of this patent UNITED STATES PATENTS

2,611,923	Hume	Sept.	30,	1952
2,767,427	Covington et al	_		
2,794,700	Cheney	_ June	e 4,	1957
2,821,458	Evans	Jan.	28,	1958
2,859,472	Wincklhofer	Nov.	. 11,	1958