

Oct. 9, 1962

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3,057,038

WET SPUN CELLULOSE TRIACETATE

Filed April 21, 1958

3 Sheets-Sheet 1

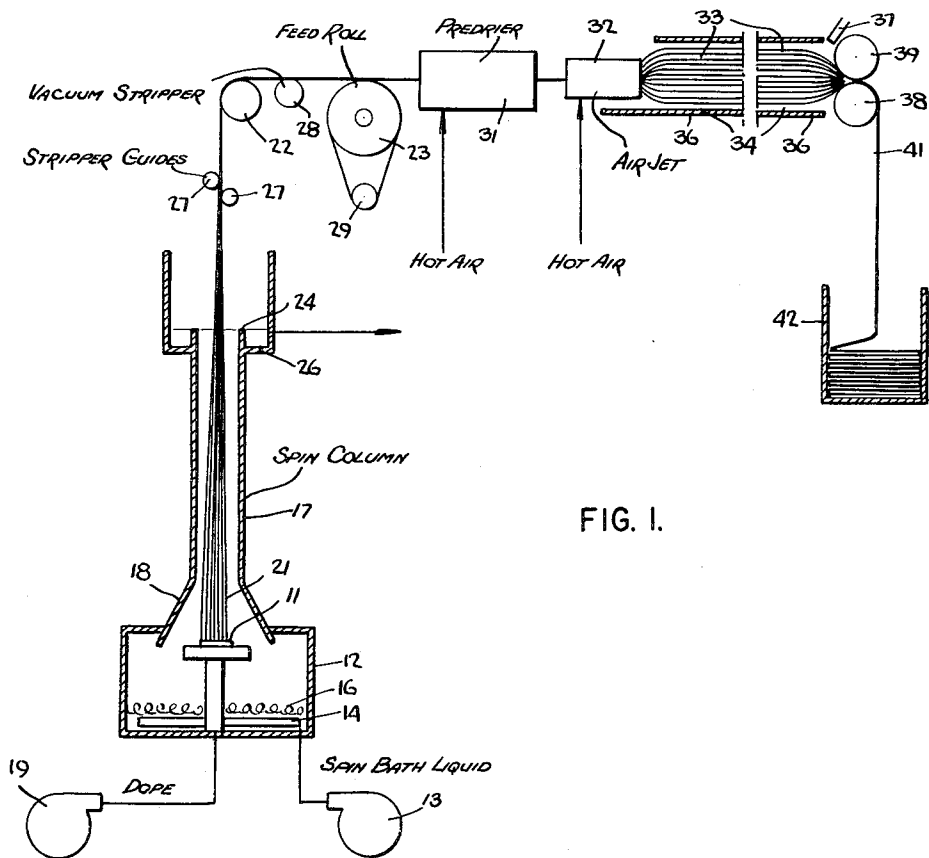


FIG. 1.

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3 Sheets-Sheet 2

FIG. 4.

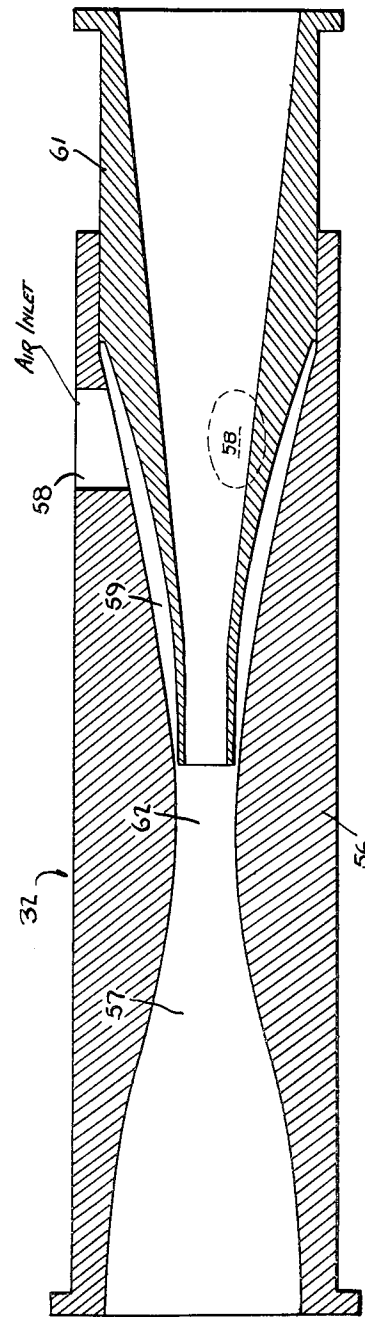


FIG. 3.

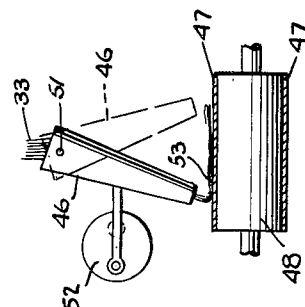
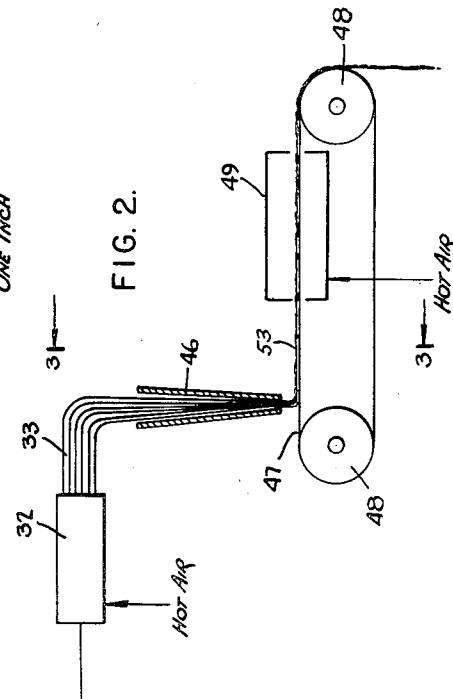


FIG. 2.



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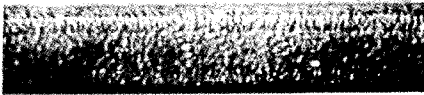
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FIG. 5.

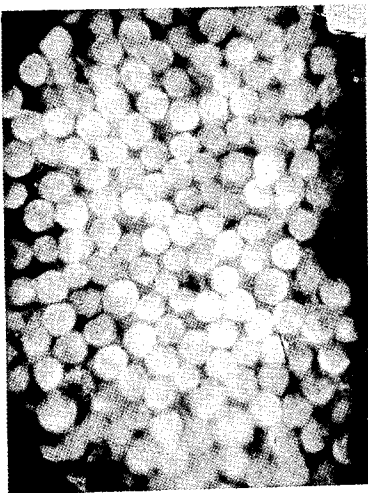
individual filament

FIG. 7.



tow

FIG. 6.



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WET SPUN CELLULOSE TRIACETATE

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Filed Apr. 21, 1958, Ser. No. 729,980

5 Claims. (Cl. 28—82)

This application is a continuation-in-part of copending application Serial No. 663,815 filed June 5, 1957.

This invention relates to the production of filamentary materials.

It is an object of this invention to provide a new and improved method for making crimped continuous artificial filaments.

Another object of this invention is the provision of a new and crimped artificial filamentary material.

Other objects of this invention will be apparent from the following detailed description and claims. In this description and claims all proportions are by weight, unless otherwise indicated.

In accordance with one aspect of this invention a solution of a filament-forming material in a liquid organic solvent is extruded through a multi-orificed spinnerette into a liquid coagulant to form a running bundle of parallel filaments swollen with liquid. The bundle is drawn through and out of the coagulating liquid, whereupon the filaments cling tightly together under the action of tension in the bundle and the action of surface tension of entrained liquid. The individual filaments of the bundles are then separated from each other and the liquid which swelled the filaments is removed therefrom while the filaments are maintained in substantially separated and tensionless condition. In accomplishing this, it is most convenient to pass the bundle of swollen parallel continuous filaments through an appropriate jet of air or other evaporative gas in such a manner that the continuous filaments are separated but remain substantially parallel, and then to remove the liquid from the separated parallel filaments by evaporation while these filaments are under substantially no tension. During the removal of the liquid, without tension, spontaneous crimping of the filaments occurs.

Preferably, the coagulant is a mixture of a solvent and a non-solvent for the filament-forming material.

The present invention has shown its greatest utility in the wet-spinning of cellulose triacetate from solutions thereof in methylene chloride or mixtures of methylene chloride and small amounts of an alcohol such as methanol, using a cellulose triacetate having an acetyl value of at least 60%, preferably above 61%, calculated as acetic acid. The cellulose triacetate spinning solution may be extruded into a bath of coagulant, or "spin-bath," comprising methylene chloride mixed with methanol or other lower aliphatic alcohol in accordance with the teachings of the copending application of Jesse L. Riley, entitled Spinning, Serial No. 638,414, filed February 5, 1957; the entire disclosure of said application of Riley is hereby incorporated, by reference, into the present application. As disclosed in said Riley application, the resulting filaments are stretched in the spin-bath, which has a considerable swelling power for the cellulose triacetate, the stretching being effected by taking up the swollen filaments at a higher linear speed than the linear speed at which they are extruded. The stretching force is most conveniently applied by means of a positively driven feed roll, such as a godet roll, over which the filaments pass, as a bundle, after they leave the spin bath. The bundle of swollen filaments leaving the feed roll generally carries a considerable proportion of liquid, for example 200% or more of liquid based on the weight of the cellulose triacetate.

2

This bundle may comprise several thousand individual filaments.

In accordance with this invention, the running bundle of parallel swollen cellulose triacetate filaments leaving the feed roll is passed through a zone where the filaments are separated from each other. As previously stated, a suitable gas jet may be used for this purpose, the construction of the jet, and the pressure of and temperature of the gas delivered thereto, being such that the filaments leaving the jet are not looped or tangled but are parallel and separate. It is preferable to remove a substantial portion of the liquid from the bundle of filaments before it reaches the jet. This may be done by the use of suitable arrangements of liquid-stripping guides, sucking devices engaging the bundle before or after it passes over the feed roll, and heated evaporative zones through which the bundle passes on its way to the jet. It is important, however, not to remove too much of the liquid before the bundle enters the jet, since unless the separated filaments emerging from the jet are still in swollen condition they will not crimp. Furthermore, the swollen filaments in the bundle leaving the feed roll are in an adhesive condition so that if too much of the solvent is evaporated before the bundle reaches the jet the swollen parallel filaments will tend to stick to each other and will not be separated in the jet. It is therefore preferable that the amount of preliminary liquid removal be such that the filaments of the bundle entering the jet are wet with liquid, that is, they carry free liquid on their surfaces. On the other hand, when the bundle entering the gas jet carries too much liquid, it is found that it is very difficult to separate the filaments in the jet, presumably because of the effect of the surface tension of the liquid; if in this case the flow of gas through the jet is increased in an attempt to increase the separating force on the filaments, the force of the gas blast generally tends to tangle the filaments. The proportion of liquid in the filaments entering the jet is preferably within the range of about 100 to 250%, based on the weight of filament-forming material.

Regarding the gas jet, very good results have been obtained by the use of a commercial venturi type water-aspirator jet; i.e. a device which is usually employed for creating a vacuum by sucking air or other gas into a stream of water passing through a venturi. In the practice of the present invention, this jet is, of course, used in an entirely different manner. Thus, the bundle of filaments is passed axially through the jet in the same direction as the air usually travels, the cross-sectional area of the bundle being, however, considerably less than the cross-sectional area of the throat of the venturi, while the gas is delivered under pressure to that inlet of the jet into which the water is usually fed. The details of design of one suitable jet are shown in the drawings and discussed below.

For best results, the gas fed to the jet should be heated to increase the evaporation of swelling liquid which occurs as the bundle passes through and beyond the jet. However, since in most jets the filaments will be under tension at some point in the jet, due to the force of the gas stream, and since crimping takes place when the swelling liquid is removed from the filaments while they are in substantially separated, tensionless condition, too great a degree of evaporation in the jet may interfere with the crimping tendency. Generally, it is desirable that the filaments emerge from the jet in an uncrimped, swollen condition, and that crimping take place after the filaments have left the jet.

The further evaporation of the swelling liquid, and the resulting crimping of the separated untensioned continuous filaments, may be effected continuously by conveying the parallel separated filaments in a stream of

heated air or other gas and in substantially tensionless condition, until sufficient swelling liquid is removed. In this case, the crimping occurs while the separated filaments are floating in the gas stream. This crimping is not dependent on any turbulent flow of the carrying stream, and, in fact, gross turbulence, which would tend to entangle filaments or cause snarls or loopiness, is undesirable and is preferably avoided by suitable shielding to avoid drafts or other outside influences during drying. Thus, the stream carrying the filaments may pass through a tunnel or a long chute so constructed as to minimize turbulence. After the filaments have dried sufficiently to set the crimp firmly therein, they are brought together on a suitable collection device. Thus, they may pass through the nip of a pair of driven take-up rolls for delivery to a packaging device, such as the tow-packaging device shown in the copending application of Weber et al. Serial No. 262,856, filed December 21, 1951, now Patent No. 2,798,348. Since both evaporation and crimping and the degree to which the fibers of the bundle are disarranged from a parallel orientation result in a differential reduction of the length of the filament bundle, the take-up rolls should be driven at such a speed that the rate of movement of the filaments at said rolls is less than the peripheral speed of the feed roll.

In another method of evaporating the swelling agent with the filaments in substantially tensionless, separated condition, the separated filaments leaving the air jet are allowed to fall freely onto a slowly moving porous belt or other conveying means which is passed through a drying zone. The filaments should be deposited on the belt, which may be in the form of an endless screen, in such a manner that there is a minimum of contact or crossing-over of filaments and therefore a minimum of interaction between adjacent filaments during the period when the filaments are crimping spontaneously, though some contact is permissible and generally unavoidable. It is convenient to lay the voluminous bundle of filaments onto belt back-and-forth across the belt, in a sinuous configuration. In this case, at the edges of the belt, in the sections where the bundle of filaments changes direction, the filaments tend to develop slightly less crimp than in the intermediate portions of the belt where the bundle of filaments is generally straight and where the bundle is looser. This effect may be reduced by directing the hot air, used for drying the filaments, upwardly in a gentle stream through the pores of the belt; this makes for a more effective relaxation and separation of the filaments on the belt. Preferably the force of the stream of hot air is not great enough to actually lift the bundle of filaments from the belt.

The extent of separation of the filaments need not be great so long as they are kept substantially independent of each other. Thus, in one case, in which a bundle of 1400 swollen filaments, the average denier per filament being 3 (on a dry basis), were passed through an air jet to separate the filaments, the diameter of the bundle leaving the air jet was $\frac{3}{8}$ inch; in this case, by calculation, the average distance between adjacent filaments was on the order of 700-800 microns.

The extent of removal of liquid from the separated, untensioned filaments should be such that the filaments are brought to a substantially unswollen condition. For example, in the application of this invention to wet-spinning of cellulose triacetate in a methylene chloride-methanol spin bath, it is preferable to continue the drying, while the filaments are separated and untensioned, until the liquid content is reduced to about 10% or less, based on the weight of the cellulose triacetate.

The crimping obtained in accordance with this invention is not dependent on the use of high temperatures during removal of the liquid. Thus, in the preferred form of the invention (involving wet-spinning of cellulose triacetate in a methylene chloride-methanol spin bath), it is desirable to feed air at a temperature in the range of

about 70 to 140° C. to the jet, but good results have been obtained using air at room temperature for this purpose. Even when hot air at 140° C. is fed to the jet, the temperature of the air leaving the jet with the filaments is probably in the neighborhood of 50° C., due to cooling resulting from evaporation of methanol and methylene chloride. In the same process, air at a temperature of about 80 to 90° C. has been supplied to the drying zone where the crimping of the filaments occurs, but here again the actual temperature of the filaments during the time crimping takes place is probably less than 65° C.

It is advantageous to apply a textile lubricant to the filaments. This lubricant is best applied after the removal of liquid from the filaments, that is, when the filaments are in non-swollen condition. The lubricant, which may, for example, be that described in United States Patent No. 2,805,992, issued to Fortress et al. on September 10, 1957, is conveniently supplied by passing the unswollen filaments over a roller, carrying a film of lubricant, before the filaments are engaged in the nip of the take-up rolls. More desirably, a mist of the lubricant may be applied by a suitable spraying device at this point. The application of lubricant in the form of a mist or spray results in a more uniform distribution of lubricant throughout the bundle and avoids the development of tension which would result from passage of the bundle over a lubricating roller or other surface under such conditions as to secure uniformity of application.

The process of this invention is especially valuable for the production of a crimped tow, made up of a thousand or more continuous filaments and having a total denier of 3000 or more, the denier per filament being about 1 to 6. It has previously been the general practice to crimp tow mechanically, as by passing the tow through a suitable heated stuffing box type of crimper. Such mechanical crimping damages the individual filaments of the tow, particularly when a highly crimped material, having five or more crimps per inch, is produced. In addition, when mechanical crimping is employed, the crimps in all the filaments of the tow are aligned and more or less in the same plane. In contrast, when the method of the present invention is used, the filaments of the tow are not damaged at all. The crimps in adjacent filaments are randomly arranged, out of alignment; the crimp being three-dimensional, either helically or randomly three-dimensional, and not substantially in a single plane. Thus, the tow is much more voluminous or lofty than the conventional tows. When the tow produced in accordance with this invention is cut into staple fiber lengths and then processed in the conventional manner to produce staple fiber yarn, much less breakage of filaments and formation of undesirable short filaments or "fly" takes place. Also, because of the voluminous character of the material, much less mechanical processing is necessary to "open" or separate the staple fibers before they are formed into yarns.

Typical crimped filaments produced in accordance with this invention contain 8 or more e.g. 8 to 12, fine crimps per inch, the amplitude of the crimp being irregular but generally being on the order of 1 mm. and the percent crimp, based on the straightened length, being above about 4%. Percent crimp is defined as

$$\frac{\text{Straightened length} - \text{crimped length}}{\text{Straightened length}} \times 100$$

The individual filaments are generally substantially circular in cross-section, in contrast to the bulbous or potato-shaped cross-section of dry-spun filaments. Also, stress-strain curves for the wet-spun cellulose triacetate filaments show a higher modulus of elasticity and higher resistance to deformation throughout the whole range of strain, as compared with dry-spun cellulose triacetate filaments.

As taught in the copending application of Riley, re-

ferred to above, the use of certain critical proportions of methylene chloride in the spin bath results in the production of filamentary materials of outstanding physical properties (e.g. tenacity of at least 1.8 grams per denier and elongation of at least 18%). In the practice of the present invention it is preferable to use proportions of methylene chloride within the scope of the Riley application. It is found however, that if the proportion of methylene chloride in the spin bath is decreased below the critical level and the proportion of methanol increased (for example by 2%, based on the weight of the bath, above the critical level), there is a greater tendency for the formation of crimped filaments, though the physical properties of the filaments are not as good. Preferably, the proportions of methylene chloride in the spin bath are within the range defined by the formula

$$C=75\frac{1}{4}-T\pm 5$$

where C is the concentration in percent and T is the temperature in °C.

By the process of this invention there are obtained crimped cellulose triacetate filamentary materials whose mechanical properties are superior to those of any crimped filaments obtained by the wet-, dry- or melt-spinning of cellulose triacetate. Thus, there are obtained crimped filamentary materials whose tenacity is at least 1.8, usually above 2, grams per denier and whose elongation is at least 18%, usually above 20%, even for filaments whose denier is in the range of 1.5 to 4. The energy of rupture, i.e. the area under the stress-strain curve from zero stretch to break, is high. These crimped filamentary materials are characterized by radial uniformity. This can be determined by treating the filamentary materials with a saponifying agent which deesterifies the surface portions of the filamentary material, forming a cellulose skin which is then removed with a solvent for cellulose. When subjected to this treatment a filamentary material which is non-uniform exhibits different properties as compared with the original material, while a radially uniform material has the same properties as before.

In testing for radial uniformity the surface removal can be effected, for example, by wetting the filaments to be tested in cold water containing 0.1 gram per liter of "Triton X-100" (iso-octyl phenyl ether of polyethylene glycol) then immersing them in 1000 times their weight of a 50 grams per liter solution of sodium hydroxide at 95° C. for 30 seconds to 3 minutes, and quickly transferring them to cold running water for 5 minutes. The filaments are then soured in acetic acid for 15 minutes and again rinsed in running water for 15 minutes. After drying in air, the filaments are immersed at room temperature for 3 minutes in a solution made up of equal weights of cupriethylene diamine and water to dissolve the cellulose skin formed by the saponification. The filaments are then rinsed, soured, rinsed and dried as before.

The foregoing treatments of course reduce the filament denier but the tenacity in grams per denier is not changed. The percent elongation also remains unchanged. X-ray diffraction patterns, microscopic observations and other properties are also the same for the starting material and for specimens from which surface layers of different thickness are removed. The safe-ironing temperature following heat treatment is also the same whether or not the filament is de-surfaced.

Whereas surface removal of dry spun cellulose triacetate effects a marked increase in the rate of dyeing, surface removal of crimped cellulose triacetate filamentary material wet spun in accordance with the present invention does not similarly affect the dyeing rate. This is demonstrated as follows: Dry spun cellulose triacetate filaments of 3.75 denier when immersed in a dyebath took up 0.18% of their weight of dyestuff after being immersed in the dyebath for 5 minutes and 0.22% of their weight after 15 minutes immersion. If these fila-

ments are first treated as described to remove a surface layer 44×10^{-6} cm. thick, the filaments under identical dyeing conditions will pick up 0.22% by weight of dyestuff in 5 minutes and 0.30% in 15 minutes. This appreciable increase in pick up evidences radial heterogeneity in the filaments.

By way of comparison, 2.5 denier filaments produced in accordance with the invention pick up 0.24% by weight of dyestuff after being immersed 5 minutes in the dyebath previously set forth and 0.34% after 15 minutes. Removal of a surface layer 47×10^{-6} cm. thick does not increase the dyeing rate. Actually there is a slight decrease to 0.23% and 0.31% in 5 and 15 minutes, respectively, i.e. approximately the same rate as de-surfaced dry spun cellulose triacetate filaments. This slight decrease in the dyestuff pick up rate of de-surfaced wet spun filaments produced in accordance with the present invention as opposed to wet spun filaments which have not been de-surfaced is due to the fact that the wet spun filaments initially have a slightly pebbled surface which is smoothed out upon de-surfacing thereby reducing slightly the surface to volume ratio of the filaments.

In the above tests the dyebath was water containing 50 grams per liter of dispersed "Amacel Red 2B" (a red cellulose acetate dye), 1 gram per liter of "Igepon T-51" (a dispersing agent) and 1 gram per liter of sodium hexametaphosphate; the bath was maintained at 95° C.

The filamentary materials of this invention show a relatively high overall birefringence after complete saponification (according to the technique described below) of said materials. The overall birefringence of the saponified material is above about 0.031, typical values being in the range of about 0.034 to 0.037. This overall birefringence is the sum of the birefringences through the fiber and is measured, in conventional manner, by a transmission technique. In the complete saponification method employed for this purpose, the filamentary material is saponified completely by immersion for at least 30 minutes in 100 times its weight of a solution containing, by weight, 5 parts of sodium hydroxide, 12 parts of sodium acetate, 10 parts of dimethylsulfoxide and 73 parts of water, at 80° C. Completion of saponification can be checked by wetting the filamentary material with 1-N cupriethylene diamine solution; if, as viewed under a microscope, the filamentary material dissolves completely in 30 seconds, saponification is complete; if not complete, the time of immersion in the saponifying liquor can be increased. When it has been determined that saponification is complete, the filamentary material is rinsed with distilled water until the rinse water is neutral. The saponified material is air dried. The treatment does not cause shrinkage or loss of strength. The overall birefringence, as opposed to merely surface birefringence, is determined in customary manner, as with a Berek compensator using polarized light.

Cellulose triacetate filamentary materials produced in accordance with this invention exhibit definite rubbery properties at elevated temperatures. This is demonstrated in the following manner: A 125 denier 40 filament yarn is held at constant length (e.g. 10 inches) and heated to a temperature of 220° C. at a just perceptible initial tension (about 0.03 g.). The temperature is then cycled between 217° C. and 223° C. It will be found that the tension on the filament increases as the temperature increases and decreases very perceptibly as the temperature decreases, typical of a rubber. By way of comparison, if the temperature of the filament is cycled between 162° C. and 168° C., the tension will be found to decrease as the temperature increases, typical of a glass.

Like other cellulose triacetate filamentary material, the cellulose triacetate filamentary material obtained in accordance with this invention may be heat treated to raise

the safe ironing temperature of fabrics produced therefrom and to improve the dimensional stability, resistance to creasing, permanence of pleating, and the like. However, the filamentary material of this invention shows substantially no shrinkage or decrease of tenacity on such heat treatment. In fact, the tenacity may even increase. For example, a filament produced in accordance with this invention and having an original tenacity of 2.15 grams per denier, when heat treated in air at 210° C. for 5 minutes shrinks less than 1% and has a final tenacity of 2.37 g./den.

Cellulose triacetate filamentary material produced in accordance with the invention is also characterized by resistance to creep at elevated temperature. This is demonstrated as follows: One end of a filament is anchored within a horizontal heating tube. 10 inches from the anchored end, the filament is knotted to a glass filament which extends outside the tube and runs over a pulley. A weight is suspended from the protruding end of the glass filament. With various size weights suspended from the glass filament the tube is heated and the displacement of the weight with change in temperature is noted. Cellulose triacetate filaments produced by dry spinning the initial solutions begin to creep at about 168° C. The instant filamentary materials do not creep comparably below about 178–183° C. The rate and amount of creep for dry-spun filaments under a load of 0.033 gram per denier are only reached for the instant filamentary materials at a load equal to or in excess of 0.067 gram per denier.

While the invention has been described in connection with cellulose triacetate as the filament-forming material, it may be applied to other organic-solvent soluble filament-forming materials; e.g. to cellulose nitrate or other cellulose esters.

In the accompanying drawing, which illustrates some aspects of this invention,

FIG. 1 illustrates, diagrammatically, the process of this invention.

FIGS. 2 and 3 are two views of a modification of the drying and crimping stage of the process,

FIG. 4 is a cross-sectional view of an air jet, drawn to scale, as indicated,

FIG. 5 is an enlarged photograph of a crimped tow produced in accordance with this invention, and also showing an individual crimped filament, alongside said tow.

FIG. 6 is a photograph showing the cross-section of a crimped tow produced according to this invention, showing the circular cross-sections of the individual filaments.

FIG. 7 is a photomicrograph of the single filament of FIG. 5, the filament being tensioned sufficiently to pull out the crimp temporarily and being shadowed with chromium to show the pebbled surface configuration. Like reference numerals designate like parts in the several views of the drawing.

In the drawing reference numeral 11 designates a spinnerette mounted in suitable vessel 12 filled with spin bath liquid which is supplied to said vessel by a pump 13. The spin bath liquid enters the vessel through a sparger 14, passing through a layer of metallic wool 16 to reduce turbulence. The spin bath also fills a vertical spin column 17 which communicates with the vessel 12 and whose tapered lower end 18 is positioned directly above and surrounds the spinnerette 11. A spinning solution, or "dope," is forced by the action of a pump 19 through the apertures of the spinnerette 11 and into the spin bath to form filaments 21, which are drawn upward through the spin column 17, and over a pulley 22, by the action of a driven feed roll 23. The spin bath, which moves slowly up the spin column 17, flows over a weir 24 to a catch basin 26, from which it is withdrawn and, after purification, recirculated to the vessel 12 through the pump 13. Some of the spin bath liquid adheres to the filaments leaving the column 17; part of this adherent liquid is removed mechanically by suitable devices such as stripper guides 27 and vacuum strippers 28 engaged

by the filaments as they pass to the feed roll 23. The still wet filaments, in the form of a bundle or "tow" make several wraps around the feed roll 23 and the usual idler roll 29 associated therewith and then are fed to a predrier 31, supplied with hot air. From the predrier 31 the tow passes through a horizontal air jet 32, where it is subjected to a stream of heated air and from which it emerges as a horizontal stream 33 of parallel separated filaments, supported in substantially tensionless condition in a stream of heated air passing through an enclosed drying zone 34. A long smooth horizontal chute 36, mounted just below the stream of air and out of contact with the filaments helps to control the direction of air flow and to support the filaments in the air stream. The filaments, now thoroughly dry and crimped, are passed under a sprayer 37, where a mist of textile lubricant is applied, and are then taken up as a continuous bundle by means of a pair of driven rolls 38 and 39. This bundle, or tow 41, is collected in any suitable manner, as in a container 42.

In the embodiment illustrated in FIGS. 2 and 3, the separated filaments 33 emerging from the jet 32 fall, under their own weight, through a pivoted traverse tube 46, to an endless moving screen 47, driven through rollers 48, which screen carries them through a drying oven 49. The traverse tube 46 is rocked on its pivot 51, by suitable mechanism 52, to distribute the filaments back and forth across the width of the moving screen 47, and the rate of movement of the traverse tube and screen are so coordinated the loose bundle of filaments 53 is laid back and forth across the belt, successive traverses of the bundle being side by side and not one on top of another.

As shown in FIG. 4 the jet 32 comprises a main body 56 having an axial venturi passage 57 and three circular side inlets 58, spaced evenly around the axis, for the introduction of compressed air into the entrance end 59 of said passage. Fitted axially within said entrance end 59 is a tapered yarn inlet tube 61, which terminates just before the throat 62 of said venturi passage 57. The yarn passes axially through the tube 61 while the air travels around said tube, the yarn and air moving together from the throat 61 to the wider discharge end of the venturi passage, and then out into the atmosphere. The jet 32 is symmetrical about its axis, all cross-sections at right angles to the axis being circular, except, of course, for the side inlets 58. FIG. 4 is drawn precisely to the scale shown thereon.

The following examples are given to illustrate this invention further.

Example I

A solution containing 21.5% of cellulose triacetate of 61.5% acetyl value, calculated as acetic acid, and of intrinsic viscosity 2.0 (measured on cellulose regenerated, without degradation, from said cellulose triacetate) dissolved in a mixture of 90 parts of methylene chloride and 10 parts of methanol, is extruded upward from a spinnerette having 1396 circular holes each 0.10 mm. in diameter into a spin bath containing 43.0% methylene chloride and 57.0% methanol at a temperature of 32° C. By the action of a feed roll mounted outside the spin bath the resulting filaments are drawn upward through a spin tube having a height of 110 cm. and filled with the spin bath, the latter moving upward at the rate of about 14 meters per minute. The filaments emerging from the top of the spin column carry about 300–400% of liquid, based on the weight of cellulose triacetate. The composition of this liquid is substantially the same as that of the spin-bath. On their way to the feed roll the wet filaments are brought together to form a tow and are passed successively over stripper guides, a pulley and a sucking roll, which remove some of the liquid, so that their liquid content is about 250% at the feed roll. The feed roll moves the wet tow at the rate of 50 meters per minute under a tension of 186 grams (measured at the

top of the column after the tow passes the stripper guides), the draw-down ratio being 6.6:1. The total denier (on a dry basis) of this 1396 filament tow is 4188. From the feed roll the tow passes through a predryer in the form of an air jet through which the tow passes axially and which is supplied with air at a temperature of 120° C. and a pressure of 50 p.s.i.g. at the rate of 7.6 s.c.f.m. (i.e. measured at 14.7 p.s.i.g. and 62° F.). This effects a partial separation of the filaments and decreases the liquid content thereof to 210%. The still wet swollen filaments emerging from the predryer pass directly to an air aspirator, constructed as shown in FIG. 4 and supplied with air at a temperature of 120° C. and an inlet pressure of 90 p.s.i.g. at the rate of 8.8 s.c.f.m. The resulting separated, parallel filaments containing 70% liquid, based on the weight of cellulose triacetate, emerge horizontally from the jet and then fall onto an endless moving metallic screen, where they are dried in substantially tensionless and separated condition in air, the air being at a temperature of 70° C. and moving upwardly through the screen, until their liquid content is reduced to less than 1%, after which a mist of lubricant is applied uniformly to the filaments. The resulting tow is much more voluminous than mechanically crimped tow. The individual filaments have 3-dimensional, irregular and sinusoidal crimps and have an amplitude of about 1 mm. and a frequency of about 10 crimps per inch. The percent crimp, based on the straightened length, is about 5.1%. The crimp is retained even when the filaments are strained past their yield point, with appreciable crimp being retained even after a strain of 5% is applied. The crimp is retained after washing in hot water (e.g. 70° C.). The individual filaments, which are substantially circular in cross-section, have a tenacity at break of 1.8 grams per denier and an elongation at break (based on their uncrimped length) of 22%. When the tow is cut to form staple fibers 2 inches in length and the resulting fibers are processed in the usual manner to form yarn, a strong yarn is formed easily, with practically no formation of "fly."

Example II

A solution containing 21.5% of cellulose triacetate of acetyl value 61.5%, based on acetic acid, dissolved in a mixture of 90 parts methylene chloride and 10 parts of methanol, is forced through a spinnerette, having 40 circular holes each 0.1 mm. in diameter, into a spin bath containing 40% of methylene chloride and 60% methanol at a temperature of 33° C. and is taken up on a feed roll at the rate of 50 meters per minute, at a draw-down ratio of 6.6:1. The resulting wet 40-filament yarn, having a total denier (on a dry basis) of 120 is passed through an air aspirator supplied with air at 20 p.s.i.g. and at a temperature of 22° C. The separated filaments emerging from the jet float substantially horizontally on

the air stream for a distance of 3 feet under substantially no tension until they are dry and are then taken up as a voluminous yarn, by means of a pair of driven rollers which move said yarn at the rate of 50 meters per minute. The individual filaments of this yarn are almost perfect helices, the amplitude of the crimp being about 1 mm. at a frequency of 10 crimps per inch, and the resulting yarn is voluminous. The filaments are substantially circular in cross-section.

It is to be understood that the foregoing detailed description is merely given by way of illustration and that many variations may be made therein without departing from the spirit of my invention.

Having described my invention, what I desire to secure by Letters Patent is:

1. A crimped filamentary material of cellulose triacetate having a tenacity of at least 1.8 grams per denier, an elongation of at least 18% and a pebbled surface.

2. A crimped cellulose triacetate filament exhibiting a tenacity of at least 1.8 grams per denier, an elongation of at least 18%, a pebbled surface, radial uniformity, an overall birefringence above about 0.031 when completely saponified, rubbery properties at 220° C., resistance to creep at 168° C., and substantially no shrinkage on heat treatment.

3. A crimped filamentary material of cellulose triacetate having a tenacity of about 1.8 grams per denier, an elongation of about 22% and a pebbled surface.

4. A crimped filamentary material of cellulose triacetate having a tenacity of about 1.8 to 2.15 grams per denier and an elongation of about 18% to about 22% and a pebbled surface.

5. A crimped filamentary material of cellulose triacetate having a denier below about 4, a tenacity of at least 1.8 grams per denier, an elongation of at least 18% and a pebbled surface.

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