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MIST SPINNING

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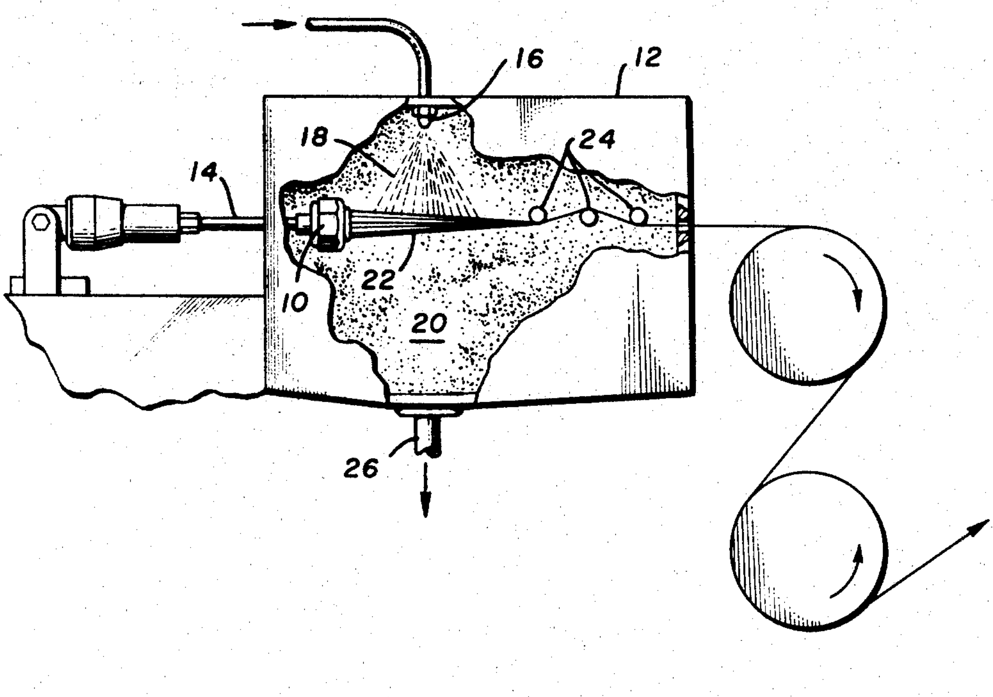

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MIST SPINNING

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ABSTRACT OF THE DISCLOSURE

A process for removing solvent from a solution of filament-forming material and solvent and for collecting the removed solvent includes the contacting of the solution while in an extruded filament-form with an atomized liquid which condenses on the filament-forms to diffuse the solvent therefrom, the stripping of the condensate from the filament-forms and collecting of the stripped condensate.

The present invention relates to the production of textile filaments. More particularly, this invention relates to an improved spinning process for producing filaments wherein a mist coagulant is utilized to form better quality filaments from a solution of fiber-forming materials dissolved in a suitable organic solvent.

In the production of filamments by the typical wet-spinning processes, the solution of fiber-forming materials is extruded into a coagulating bath which contains a liquid coagulant for the fiber-forming materials. The filaments are formed as the solvent diffuses out into the coagulation bath. This diffusion is generally accompanied by the penetration of the coagulating bath liquid into the filament. Therefore, a clean bath must be maintained to prevent contamination of the filaments. One of the major problems with preparing filaments by these processes is the fragility of the newly formed extrusions. Because of this initial fragileness, the filaments may be damaged easily and various precautions are necessary if good quality filaments are to be obtained. For example, the spinning speed must be reduced in order to avoid breaks and damage to the fragile filaments because of the density of the liquid coagulating bath. Also, the coagulating bath must be regulated closely to insure that the extracted solvent is removed while maintaining minimum turbulence in the bath to avoid contaminating or damaging the newly formed filaments. Thus, a recirculating system capable of maintaining accurately controlled conditions is essential.

Several techniques have been devised in attempts to overcome the problems experienced with the known wet-spinning processes. In some instances, it has been recommended that spinning tubes be used to minimize turbulence in the bath flow. Others have recommended that the bath flow at the same rate as the extrusion rate so that no drag or tension will be exerted on the newly formed filaments. Perhaps these methods tend to reduce, to some extent, the problems imposed by the liquid coagulating bath, but none of them effectively eliminate all such problems because of the inherent physical conditions established by the bath itself. Furthermore, the liquid coagulating bath must be maintained at elevated temperatures which cause slight color formation of some polymers.

Although the problems experienced in wet spinning and dry spinning processes are not similar, there are however, some inherent disadvantages encountered with the typical dry-spinning processes. These include the limitations such as the low number of ends and the small deniers which the spinning processes must be limited in order to avoid fiber coalescence or married fibers. Also, the elevated temperatures employed to facilitate drying and solvent removal cause yellowing of acrylic polymers.

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It is an object of fthe present invention to provide an improved spinning process wherein the filaments are coagulated by a mist coagulating medium.

Another object of the present invention is to provide an improved spinning process wherein the newly formed filaments are not contaminated as heretofore by impurities that normally occur in the liquid spin baths which collect on the filaments.

A further object of the invention is tot provide a process which facilitates faster spinning rates and improved fiber qualities.

Still another object of the invention is to provide a process for coagulating filament-forming materials with a mist coagulant at approximately ambient temperature to prevent color formation normally caused by elevated temperatures.

Yet another object of the invention is to provide a process which facilitates uniform solvent removal to produce cleaner fibers.

Another object of the invention is to provide an improved process wherein the coagulating zone is enclosed and free of collected impurities.

Other objects and advantages of the present invention will become apparent from the detailed description and drawing.

The objects of this invention have been accomplished by extruding a solution of filament-forming polymers through a spinneret located in a generally enclosed chamber filled with a suitable coagulant introduced therein as a mist or fine spray which diffuses or causes a chemical reaction to remove the polymer solvent from the extruded solution and coagulates the remaining filament-forming polymers to form filaments that are withdrawn from the mist-filled chamber and then advanced through successive washing, stretching, and drying operations. Subsequently, the mist spinning process contemplated by the present invention is distinguishable from the known spinning processes by the manner in which coagulation is achieved. Instead of extruding the filaments into a liquid bath or heated chamber to accomplish coagulation, the extrusion is made into an enclosure where the coagulant employed is in the form of a fine mist at approximately 25° C. which instantly saturates the emerging extrusions and transports the diffused solvent out of contact therewith to produce clean filaments having lower solvent content that must be removed later by washing than regular wet-spun filaments. Thus, the solvent is uniformly extracted in a substantialy frictionless coagulating environment at ambient temperatures wherein increased spinning speeds can be employed to produce higher quality fibers more cheaply than by normal wet-spinning processes known heretofore.

By "mist coagulant," as employed herein, is meant a liquid reduced to ultra-fine particles by a pressurized fluid which is a nonsolvent for the fiber-forming polymers but a solvent for the polymer solvent.

The present invention will be more clearly understood by reference to the accompanying drawing in which a single figure illustrates a side elevation, partly in cross-section, of suitable apparatus for accomplishing the objects set forth.

Referring specifically to the individual elements and their arrangement as illustrated by the drawing, there is shown a spinneret or jet **10** located in a chamber **12**. The spinneret is positioned for horizontal extrusion of filament-forming materials supplied through rounder arm **14** from a source, not shown. A conventional spray nozzle **16** is disposed in the upper portion of chamber **12** for introducing a mist or spray **18** into the area enclosed by the chamber to produce a vaporized atmosphere which functions as a coagulating zone **20**. The mist or spray is vaporized by pressurized gas such as air, and may be comprised of water, but a mixture of water and a suitable solvent for the filament-forming materials has been found to be preferable. As the extrusions emerge from the spinneret **10**, they are coagulated by the mist or spray coagulant **18** to form a ribbon of filaments **22**. The filaments are withdrawn from the chamber **12** through a group of horizontally aligned bars **24** to strip or remove excessive amounts of water and solvent condensate from the filamentary structures. A drain **26** is provided for removing the mixture of water and solvents from the chamber **12** thereby preventing contamination of the filaments with the diffused solvents and removing deleterious drag normally imposed by a liquid bath.

Although the filaments prepared in accordance with the present invention are cleaner than filaments prepared by typical wet-spinning processes, the solvent is not always entirely diffused or reacted from the filaments by the mist coagulant **18**. Therefore, it is desirable to advance the withdrawn filaments through a wash bath to remove any remaining solvent.

The invention will be further illustrated but is not intended to be limited by the following examples which define parts and percentages by weight unless otherwise specified. The tests for color indicative of approaching whiteness used throughout the examples consist of measurements of brightness and purity as calculated from the tristimulus values determined on a General Electric Spectrophotometer in accordance with the methods recommended by the Standard Observer and Coordinate System of the International Commission on Illumination as fully set forth in the Handbook of Colorimetry published by the Technology Press, Massachusetts Institute of Technology 1935. The test results are given in figures which indicate a greater whiteness or purity as they decrease towards zero.

Example I

A slurry of 250 grams of a copolymer of 93 percent acrylonitrile-7 percent vinylacetate in 750 grams of dimethylacetamide was prepared by chilling the dimethylacetamide to −10° C., adding the copolymer to the chilled slurry and mixing the slurry for 10 minutes. The slurry was then heated to 75° C. with continuous agitation. Heating and stirring was continued at this temperature for two hours to form a homogeneous solution. The polymer solution was extruded through a 10-hole, 3.5-mil diameter spinneret into a coagulation bath containing 55 percent dimethylacetamide and 45 percent water at a temperature of 55° C. The solution was converted into fiber in the coagulation bath and removed from the bath, after an immersion of 1.5 inches, by advancing rolls. The fiber was next passed through a boiling water bath where it was stretched 5.0 times, the stretch being imparted by another set of advancing rolls traveling at a faster speed than the first set of advancing rolls. The oriented fibers were then passed through an aqueous bath where a conventional antistatic finish was applied. The fiber was then dried by a final set of rolls, traveling at 71 f.p.m., which were steam heated to 105° C. The dried fiber had a denier per filament of 12.5, an elongation of 17 percent, and a tenacity of 2.9 grams per denier. The brightness, purity, and dominant wave length of this fiber were 88.7, 7.9, and 574 mμ respectively.

Example II

The procedure followed in Example I was repeated with the exception that the spinneret was positioned 0.25 inch above the spin bath, the fibers extruded vertical to the bath surface, and passed around a ceramic guide to permit removal from the bath after a total immersion of 1.5 inches. The denier per filament was 12.54, elongation was 19 percent and the tenacity was 3.10 grams per denier.

The brightness, purity and dominant wave length were 87.8, 7.5, and 574 mμ, respectively.

Example III

The procedure followed in Example I was repeated with the exception that the bath employed in the two previous examples was replaced with a chamber into which was sprayed a coagulating mist which consisted of a mixture of air and water at room temperature (25° C.). The nozzle employed gave a hollow cone spray from a 0.028-inch orifice diameter and at 25 p.s.i.g. delivered 2.4 gallons of water per hour at room temperature. The nozzle was located approximately 2 inches below and 0.5 inch in front of the spinneret. The chamber employed was 24 inches wide, 25 inches deep, and 30 inches in height and was constructed of Plexiglas with three sides and a top to permit viewing of the spinning process and with one side open to permit removal of the fibers. The chamber was set in a stainless steel pan fitted with a drain to collect any mist condensate which formed during spinning. The fiber, on passing from the open end of the mist chamber, was passed over a ceramic guide which stripped condensate from the fibers. The fiber had a denier per filament of 15.29, a tenacity of 2.93 grams per denier and an elongation of 15 percent. The brightness, purity, and dominate wave length were 87.8, 6.9, and 573 mμ, respectively. A skin core effect was observed in the fiber cross-section.

Example IV

The procedure followed in Example III was repeated with the exception that the mist employed was a 50:50 mixture of water and dimethylacetamide. The fibers obtained were identical to those obtained in Example III with the exception that the skin-core effect was reduced and the fibers were more uniform in cross-section.

Example V

The procedure followed in Example I was repeated with the exception that the maximum continuous spinning speed, as measured at the drier rolls, was established as 337 f.p.m. This speed is the speed which can be maintained continuously without breaking a filament. The speed is determined by successively increasing the speed of each roll until a filament breaks and then setting the speed at 85 percent of the breaking speed.

Example VI

The experiment described in Example V was repeated with the exception that the wet spinning coagulation bath was replaced with the mist spinning apparatus described in Example III. The maximum continuous spinning speed was 420 f.p.m., an increase of 25.4 percent over the speeds obtained in Example V.

Example VII

The procedure described in Example III was repeated with the exception that the concentration of polymer in solution was decreased from 25 percent by weight to 20 percent by weight. The spinning speed was increased to 220 f.p.m. at the drier roll. The fiber obtained had a denier per filament of 4.14; tenacity of 2.9 grams per denier and an elongation of 10.0 percent.

Example VIII

The procedure described in Example III was repeated with the exception that a 500-hole, 3-mil diameter spinneret with the holes randomly distributed over the spinneret's 1-inch face was employed. It was observed that with a single jet for mist formation coagulation was incomplete and it was necessary to install a second jet, identical to the first but located above the spinneret in order to obtain continuous, trouble-free spinning. The fibers obtained with the dual jet system were equivalent to those obtained in Example III.

Example IX

A 30 percent by weight solution of poly(vinyltrifluoroacetate) in acetone was prepared by adding 300 grams of the polymer, prepared as described in the copending application Ser. No. 358,243 filed Apr. 8, 1964, to 700 grams of acetone which had been previously chilled to 0° C., slurrying the mixture until the polymer was thoroughly wet and then allowing the mixture to warm to room temperature. Agitation was continued for two hours to assure that the polymer was completely dissolved. The solution was spun into fiber as described in Example III with the exception that the finish bath was replaced with a 37 percent by weight solution of ammonium hydroxide at 26° C. The poly(vinyltrifluoroacetate) was continuously hydrolyzed in the ammonium hydroxide bath to poly(vinyl alcohol). The fibers obtained were clear, flexible and had a denier per filament of 11.9, tenacity of 4.9 grams per denier, and an elongation of 22 percent.

Example X

A solution containing 100 grams of poly(vinyl chloride) and 60 grams of N,N-dimethyl lauramide in 240 grams of dimethylformamide was prepared by first dissolving the amide in the solvent, slurrying this mixture at 5° C. with the poly(vinyl chloride) and then heating the mixture to 85° C. for 30 minutes. The clear viscous solution was then extruded through a 10-hole, 5-mil diameter spinneret into a mist chamber, such as that described in Example III, into a mist of 10 percent by volume dimethylformamide and 90 percent by volume water. The mist forming nozzle was positioned 8 inches in front of and 2 inches below the spinneret. The remaining spinning conditions were identical to those employed in Example III. The fibers had a denier per filament of 736, a tenacity of 0.14 gram per denier, an elongation of 231 percent, a tenacity at 50 percent extension of 0.03 gram per denier, a tensile recovery after stretching and releasing tension of 94.8 percent, and a stress decay of 12.0 percent on the second extension.

Example XI

A solution of 35 percent by weight nylon 6 in formic acid was prepared by heating the polymer having a relative viscosity of 72, at 50° C. for two hours with gentle stirring. The clear homogeneous solution which resulted was spun according to the procedure described in Example III with the exception that a 10-hole, 2-mil diameter spinneret was employed and the mist was generated by spraying a 10 percent by weight sodium hydroxide solution through the mist forming orifice. The fibers obtained were clear translucent fibers having a denier per filament of 3.5, a tenacity of 3.4 grams per denier, and an elongation of 28 percent.

Example XII

A solution of an isocyanate modified dihydroxy elastomer with a viscosity of approximately 30,000 cps., prepared as described in copending application Ser. No. 369,019, was spun by the process described in Example III to produce elastomeric fibers having improved color. By carrying out the coagulation in a mist atmosphere the deleterious drag of spinning solutions is eliminated and the problems associated with broken filaments is removed.

Example XIII

A slurry of 2,500 grams of a copolymer of 93 percent acrylonitrile-7 percent vinylacetate in 7.50 grams of dimethylacetamide was prepared by chilling the dimethylacetamide to −10° C., adding the copolymer and mixing the chilled slurry for 30 minutes. The slurry was then heated to 65° C. and mixed for two hours to form a clear homogeneous solution. Attempts were then made to dry spin the solution into fibers using the apparatus described in the patent U.S. 2,636,217 to W. H. Hammond et al., using a 10-hole, 3-mil diameter spinneret. At pump speeds calculated to produce a 12.5 denier per filament fiber the filaments contacted each other before the solidification process could be completed to produce an unsatisfactory bundle of married filaments.

Example XIV

The experiment described in Example XIII was repeated with the exception that a fine mist of water was introduced into the tower at room temperature. The mist was produced by introducing nitrogen at 25 p.s.i.g. and water at 2.4 gallons per hour into a hollow cone spray with a 0.028-inch orifice diameter. After advancing a distance of less than one foot from the spinneret face the fibers were sufficiently solidified to permit their removal from the apparatus for further processing. The apparatus was modified to permit the removal of the fiber after one foot of vertical travel and the arrangements for heating the apparatus were eliminated. The fibers were passed over a ceramic guide and advanced horizontally to advancing rolls and then stretched 5 times in a boiling water bath by a second set of rolls. The fibers were thoroughly washed with 60° C. water on the second rolls, passed through an aqueous finished bath containing an antistatic finish agent, dried on a final set of steam heated rolls at 105° C., and collected at 68 f.p.m. on a conventional take-up machine. The collected fibers were free of married filaments and after exposure to boiling water for 5 minutes had a denier per filament of 17.4, a tenacity of 2.57 grams per denier and an elongation of 27.6 percent.

Example XV

The procedure described in Example XIV was repeated with the exceptions that a 500-hole, 3-mil diameter spinneret was used and three nozzles located in a plane 0.5 inch below the spinneret at 120° intervals and at a distance of 2 inches from the extruded fibers were employed. The pumping rate was increased 50 times to accommodate the additional number of holes in the spinneret. The properties of the spun fiber were essentially identical to those obtained in Example XIV.

Example XVI

The procedure described in Example XIV was repeated with the exception that the copolymer employed in Example XIV was replaced with a blend of 88 percent by weight of a copolymer of 97 percent acrylonitrile and 3 percent vinyl acetate with 12 percent by weight of a copolymer of 50 percent acrylontrile and 50 percent 2-methyl-5-vinylpyridine and the concentration of the solvent was increased to 82 percent by weight. The fibers obtained after exposure to boiling water for 5 minutes had a denier per filament of 15.4, a tenacity of 1.9 gram per denier, and an elongation of 39 percent.

Example XVII

A solution of approximately 23 percent isocyanate modified dihydroxy elastomer polymer, such as the polymer described in copending application Ser. No. 369,019 filed May 20, 1964, mixed in dimethylformamide solvent was spun by the process described in Example XIV to give married elastomeric fibers having improved color and by a more economical process. By either increasing the number of mist forming nozzles or by varying the point of removal from the spinning apparatus it was found that unmarried fibers could be produced. Also, by employing lower spinning temperatures, a product having improved color was obtained.

From the data set forth in the foregoing examples, it is apparent that filaments spun in accordance with the present invention have several improved qualities over filaments spun in accordance with the procedure of Examples I and II which are typical of the conventional wet-spinning processes and Example XIII which represents a typical dry-spinning process. The spinning speeds for the wet-spinning processes are directly related to the amount of drag imposed on the newly formed filaments. Therefore, by accomplishing the fiber coagulation in a mist atmosphere, the deleterious drag of spinning solutions normally encountered will be eliminated and the accompanying problems associated with broken filaments will be removed. In addition to faster spinning speeds, the mist atmosphere facilitates the preparation of fibers having improved whiteness. For example, where the spinneret is submerged in a liquid bath, the temperature must be elevated somewhat to avoid lowering the temperature of the spinneret, which changes the viscosity of the spinning solution. Similarly, in the dry spinning processes elevated temperatures are employed to facilitate fiber formation and solvent removal. The elevated temperatures tend to cause color formation of some polymers such as the acrylics unless special precautions are observed. Since the mist does not remove a substantial amount of heat from the spinneret, and the solvent is removed by said mist, the coagulating medium can be maintained at a much lower temperature while optimum spinning conditions are observed. In addition to the improved fiber qualities, coagulation is accomplished in a hooded area which reduces solvent evaporation and thereby reduces costs.

Generally, this process is applicable to the spinning of a broad range of synthetic fiber-forming polymers and polymer blends which may be solution spun by methods well known in the art. Included among these synthetic materials are acrylic polymers, notably homopolymers and interpolymers of acrylonitrile. The acrylonitrile polymers containing at least 85 percent of acrylonitrile, interpolymerized with one or more ethylenically unsaturated monomers copolymerized therewith, such as methyl acrylate, acrylic acid, methacrylic acid, vinyl acetate, vinyl pyridine, vinyl chloride, vinyl bromide, styrene and the like are particularly preferred. Other fiber-forming polymers such as polyamides, polyesters, polyurethanes, vinyl polymers and the like may be solution spun by the process of this invention.

The particular solvent employed to dissolve the polymer will, of course, depend on the identity of the polymer in each particular instance as is well known in the art. Where the above-described polymers of acrylonitrile are spun according to this invention, it is most desirable to employ organic polar solvents such as dimethylformamide, dimethylacetamide, dimethyl sulfoxide, butyrolactone, ethylene carbonate, propylene carbonate, and the like. However, other known solvents such as aqueous salt solutions may likewise be employed for the acrylic polymers.

It has been found that conditions in the spinning chamber are not generally critical except that the mist or spray must be reduced to ultra-fine particles which are present in abundance to coagulate the fiber-forming polymers. Thus, the particles must be small enough to impinge upon the freshly extruded threadlines without breaking the fine denier filaments. Also, as described elsewhere herein, it has been found to be particularly advantageous to maintain the temperature level in the spinning chamber at approximately ambient temperature when spinning acrylonitrile, polyurethane, etc. to prevent yellowing of the fibers.

The foregoing detailed description has been given for clearness of understanding only, and unnecessary limitations are not to be construed therefrom. The invention is not to be limited to the exact details shown and described since obvious modifications will occur to those skilled in the art, and any departure from the description herein that conforms to the present invention is intended to be included within the scope of the claims.

What is claimed:

1. A process for removing solvent from a solvent comprised of a polymeric filament-forming material and a solvent for said polymeric material and for collecting said removed solvent, comprising:

(a) extruding said solution into an enclosed chamber in filament-form, having a plurality of openings, said filament-forms being guided along a path through said chamber and out a first one of said openings;

(b) injecting into said chamber through a second one of said openings, said atomized liquid being a solvent for said polymeric material solvent and being inert to said polymeric material, said atomized liquid contacting said filament-forms and condensing thereon whereupon portions of said polymeric material solvent is diffused from said filament-forms to coagulate the same;

(c) stripping said condensate from said coagulated filament-forms, said condensate being comprised of said atomized liquid and said polymeric material solvent; and

(d) collecting said stripped condensate in said chamber at a point removed from said filament-forms path.

2. The process of claim **1** wherein said atomized liquid is water.

3. The process of claim **1** wherein said atomized liquid is comprised of water and an organic polar solvent for said filament-forming material.

4. The process of claim **1** wherein said atomized liquid is comprised of an aqueous salt solution.

5. The process of claim **1** wherein said path along which said filament-forms are guided is horizontally disposed in said chamber.

6. The process of claim **1** wherein said collected condensate is removed from said chamber by means of a drain forming a third one of said chamber openings.

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