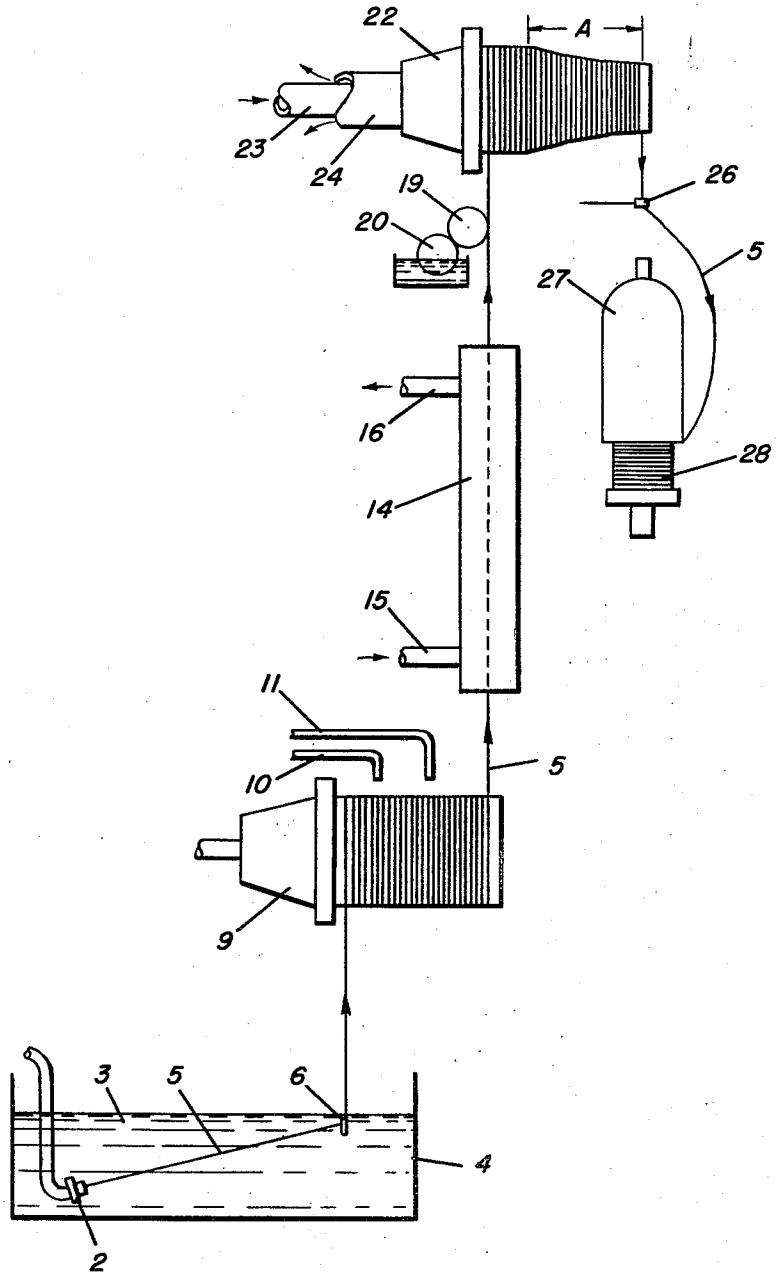
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WET SPINNING OF ACRYLONITRILE POLYMERS

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WET SPINNING OF ACRYLONITRILE POLYMERS

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This invention relates to the production of wet-spun acrylonitrile polymer fibers having stabilized and uniform whiteness and luster. More particularly, it is concerned with the treatment of opaque fibers made from ethylene carbonate spinning solutions of polymers and copolymers of acrylonitrile containing in the polymer molecule at least 80% by weight acrylonitrile. More specifically, this invention involves a process for treating such wet-spun opaque, low-luster fibers to increase the luster thereof, to reduce the opacity and porosity thereof, and to impart to them a permanent stability against further relustering in the presence of heat.

It is generally known that the highly lustrous, transparent, colorless fibers made from acrylonitrile polymers such as those produced by dry spinning possess an inherent discoloration or off-whiteness ranging from a slight yellowish shade to a golden yellow. It has also been found that such inherent discoloration may be eliminated by producing a less lustrous fiber such as, for example, by wet-spinning under appropriate aqueous conditions. However, it has been found that most of the wet spinning methods heretofore proposed are not capable of producing a fiber of reduced luster having satisfactory strength and satisfactory stability against further relustering in the presence of heat. Thus, for example, fibers made by such methods may be either excessively delustered and too porous resulting in a low strength, chalky, brittle fiber; or, if a reduced-luster fiber of suitable strength is obtained, it may undergo further relustering before or after dyeing, thus changing the entire appearance of the fiber or fabric made therefrom. When an unstabilized, reduced-luster fiber or fabric is contacted with a hot surface such as during ironing and pressing in the presence of moisture, it will undergo further relustering and will exhibit a completely different intensity of whiteness or depth of dye.

It has now been discovered, however, that wet-spun opaque fibers can be relustered into white colorless fibers which are permanently stabilized against changes in luster and color in the presence of heat and moisture. Moreover, it has been found that by the process and conditions of the present invention such relustering, or opacity-stabilizing, treatment is not only imparted uniformly, but is advantageously carried to the point where heat stability of the opacity is achieved with only a partial loss of the whiteness of the original unrelustered fiber. This may be accomplished generally as follows: An opaque, low-luster white fiber is first formed by extruding

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an ethylene carbonate spinning solution of an acrylonitrile polymer through a spinneret and into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water. The acrylonitrile polymer employed contains in the polymer molecule at least about 80% by weight acrylonitrile. The resulting opaque fiber is then stretched and thereafter, while it contains at least about 3% by weight of water based upon the weight of the dry fiber, is conducted to a thread-storing, thread-advancing device which is heated to a temperature above 120° C. The fiber is then advanced in generally helical turns on the thread-advancing device, which device is heated above about 120° C. The fiber is temporarily stored on the heated device at least until substantially all of the water is removed from the fiber.

In general, the low-luster, opaque fibers of acrylonitrile polymers which are subject to the treatment in accordance with this invention may be produced by wet spinning methods in which at least a portion of the coagulation of the extruded material is performed in the presence of water. The coagulating action may be performed completely by an aqueous medium or part of the coagulating action may be performed by a non-aqueous medium, e. g., a polyhydric alcohol such as glycerol, followed by the coagulating action of an aqueous medium such as water. Among the aqueous cogulants that have been proposed and which are capable of producing non-lustrous opaque fibers of suitable strength may be mentioned water baths containing calcium chloride, sodium thiosulfate, calcium nitrate, ethylene carbonate, etc. In general, in order to produce opaque non-lustrous fibers of suitable strength, highly porous and extremely low density fibers should be avoided. This may be accomplished with facility, for example, by adjusting the various coagulating conditions, e. g., bath temperature, coagulant proportions, water content, bath-travel, etc., so that a fiber having the highest luster and transparency under the particular conditions is first formed and thereafter reversing one or more of such conditions until the fiber upon completion of its coagulation assumes a milky white, non-lustrous, opaque appearance. The coagulating conditions are adjusted so that they are maintained with advantage in the vicinity of the transition point preferred, i. e., transparent to milky white. One method of producing such opaque fibers, which can be treated in accordance with the present

process, is described in the Gilman S. Hooper and Thomas S. Solomon application Serial No. 248,561, filed September 27, 1951. In this method a non-aqueous liquid coagulating medium comprising certain water-soluble polyalkylene glycols and ethylene carbonate is employed as the initial coagulant. The resulting fiber formed therein is highly lustrous and transparent and is withdrawn therefrom while it is still in an incompletely coagulated state. It is then subjected to the coagulating action of water which completes the coagulation thereof and imparts a low-luster opaque appearance. Among the polyalkylene glycols that may be employed as the initial coagulants in the spinning process just described may be mentioned dipropylene glycol, tripropylene glycol, triethylene glycol, and tetraethylene glycol.

The resulting opaque fiber is thereafter stretched to any desired degree, e. g. 3 to 10 times. The fiber may be stretched in the presence of a hot fluid medium such as water or other inert liquids or gases such as hot air or steam. It is found particularly advantageous, however, to stretch the fiber while it is in direct contact with steam. It is also found advantageous to wash the fiber substantially free of solvent and coagulant prior to stretching, i. e., less than about 4% ethylene carbonate and preferably less than 1%.

In accordance with the present invention, the stretched fiber is then conducted to a thread-advancing device which is heated to and maintained at a temperature above 120° C. The fiber being conducted to the heated advancing device should contain at least about 3% by weight of water based upon the weight of the dry fiber. If a water-containing stretched fiber has been dried after the stretching step, e. g., by stretching in non-aqueous fluids or on hot surfaces, etc., it is necessary that the fiber be rewetted such as by applying water thereto or by running or dipping the fiber continuously into a water trough prior to the relustering treatment on the heated advancing device. On the other hand, greater advantages are derived if the fiber is conducted to the heated device in a previously undried and water-swollen, gelled state, e. g., by conducting a steam-stretched fiber directly to the heated advancing device. The water-containing fiber being advanced on the heated device is stored thereon for a sufficient time (or a sufficient number of helical thread turns) to permit substantially all of the water to be removed from the fiber. It is believed that the contact of the water-containing fiber with the heated thread-bearing periphery of the advancing device relusters the fiber by causing the pores in the opaque fiber to collapse and close due to the plasticizing action of the water under these conditions.

By this procedure, the desired extent of relustering of the opaque fiber can be achieved for stabilizing such fiber against subsequent changes in luster. In addition, the resulting relustered colorless fiber or fabric possesses a greater degree of whiteness than is obtainable if the fiber were coagulated initially in a fully lustered form.

In general, thread-storing, thread-advancing devices that may be employed in the present process may comprise a plurality of drums, rollers, rods, bars, and the like on which the advancing thread is stored in a plurality of turns in the form of an almost true circular helix or a distorted oval-like helical configuration. Particularly suitable and advantageous are those advancing devices, e. g., reels on which the thread

is substantially continuously in contact with the heated thread-bearing periphery such as, for example, those on which the traveling stored yarn described an almost true circular helix and on which a large number of turns per inch may be stored. Devices of this type are described in the Knebusch Patent No. 2,210,914 and particularly in the Bergmann Patent No. 2,294,866 illustrating a thread-advancing reel which can be heated internally and maintained at the desired temperatures with hot fluids such as water, steam, hot gases, etc.

It is found especially advantageous to heat the thread-advancing device internally although, if desired, external heating such as by infrared ray lamps, electric heating coils, etc., may also be employed. Particularly suitable is the introduction and circulation within the device of steam at gauge pressures between about 15 and 130 pounds per square inch depending upon the structural material of the device and the design thereof.

It is also found advantageous to perform the heat-relaxing step of the oriented fiber simultaneously with the relustering step of this invention. The heat-relaxing treatment is usually employed to enhance the physical properties such as tenacity and elongation of an oriented fiber. In general, this operation is performed on a dried fiber such as in skein form. The presence of moisture, however, is not necessary in such operations. In the present process, however, the two operations could with advantage be performed continuously on the same heated advancing device. This can be accomplished by employing a thread-advancing device having at least a section thereof of progressively diminishing diameter in the direction of thread-advance so that the tension of the advancing fiber is reduced while it is being heated, thereby permitting the fiber to be heat-relaxed. Thus, for example, when employing an internally heated reel of the circular-helix type previously described but having a tapered section to allow shrinkage under controlled tension, a water-containing fiber can be relustered and also heat-relaxed by storing the fiber thereon for at least about 3 seconds, and preferably less than 10 seconds.

The process of the present invention will be further described in conjunction with the drawing which illustrates a form of apparatus suitable for practicing the invention. In the drawing, a polymer spinning solution of the type previously described is extruded through a multihole spinneret **2** into a coagulant **3** contained in a spin-bath trough **4**. The filamentary material **5** formed in the bath is led through a bath-submerged guide **6** which can be adjusted by a slight rotation to act as a resistance or snubbing guide to aid stretching. After emerging from the coagulant, the thread **5** is conducted to a positively driven thread-storing, thread-advancing reel **9** around which it is wrapped a sufficient number of helical turns to store the same thereon for between about 2 and 6 minutes. To a thread-helix on reel **9**, water may be delivered by tubes **10** and **11**, respectively, at the total rate of between about 200 and 600 cubic centimeters per minute depending upon the extent of washing desired and the speed and denier of the yarn being washed. The thread **5** discharged from reel **9** is then conducted to a steam tube **14** having a steam inlet opening **15** and an outlet opening **16** whereby the thread passing through the tube is brought into direct contact

with the steam. The stretched thread leaving the steam tube 14 is passed over a water-applying roller 19 fed by a roller 20. The thread is pulled through the steam tube 14 and over roller 19 by a positively driven, internally heated, thread-advancing reel 22 driven at a peripheral speed greater than that of reel 9 whereby the desired amount of stretch may be imparted. Reel 22 is heated by introducing a heated fluid such as, for example, steam, through the shaft 23 at sufficient pressure to maintain the reel at between about 120° and 175° C. but preferably, at about 150° C. After circulation of the steam within the reel, it is exhausted through the concentric shaft 24. Reel 22 is constructed with a tapered section "A" having a progressively diminishing thread-bearing periphery in the direction of thread-advance so that the advancing thread thereon may be heat treated under relaxed tension. For treating threads having deniers between 60 and 300, there is about a 5% taper for the first 12 meters of thread and about a 1% taper for the next 18 meters of thread. The thread 5 discharged by the heated relustering and relaxing reel 22 is conducted around a guide 26 and is then collected by a cap twisting device 27 onto the package 28.

The invention will be more fully described by the following examples, it being understood that the invention is not intended to be limited by the illustrative conditions therein. In these examples "parts" and "percent" of materials is intended to mean parts and percent by weight.

Example I

20 parts of polyacrylonitrile (average molecular weight about 42,000) is dissolved in 80 parts ethylene carbonate. This solution after filtering and deaerating under vacuum, is heated to 120° C. and extruded at the rate of 15 grams per minute through a spinneret having 40 holes (0.003 inch diameter) into a coagulating bath maintained at a concentration of about 80 parts dipropylene glycol and 20 parts ethylene carbonate. The bath temperature is maintained at 130° C. while the freshly formed filaments are drawn through the bath for a distance of about 40 inches and then through air for about 12 inches by means of the thread-advancing reel 9 of the drawing traveling at a peripheral speed of about 42 meters per minute. Under these conditions the total time the freshly formed filaments remain in the coagulating bath corresponds to about 1½ seconds. The withdrawn thread is stored on the thread-advancing reel 9 for about 2¼ minutes while being treated with a total of about 200 cubic centimeters per minute water at about 60° C. delivered by tubes 10 and 11. Immediately after its first contact with water, the thread changes in appearance from a lustrous transparent fiber to a white, milky, opaque fiber. The washed thread discharged from the reel 9 is thereafter pulled through the steam tube 14 by the internally heated, tapered thread-advancing reel 22 traveling at the rate of about 400 meters per minute thereby stretching the thread over nine times. While traveling from the steam tube to the reel 14, the thread is wetted with water by the roller 19. The relustering and relaxing reel 22 is maintained at a temperature of about 140° C. by circulating steam therein at about 40 pounds per square inch gauge pressure. The thread is then stored thereon for about five seconds and is discharged therefrom as a dried thread and collected by the cap twister 27. The resulting heat-treated thread and also the fabric made therefrom are fully stabilized against further relustering and will not change in color or appearance when contacted with a flatiron at 175° C. in the presence of moisture. Moreover, it retains a very desirable whiteness and is relatively free of any off-white yellowish shade and can be dyed with acetate dyes. It has a tenacity of about 4 grams per denier and an elongation of 18%.

Example II

12 parts of a copolymer containing 94% acrylonitrile, 5% 2-vinylpyridine and 1% vinyl acetate (average molecular weight about 45,000) is dissolved in 80 parts ethylene carbonate and while maintaining the resulting solution at a temperature of about 50° C., there is added eight parts of water. The resulting spinning solution is extruded at the rate of 19 grams per minute through a spinneret having 40 holes (0.003 inch diameter) into a coagulating bath maintained at a concentration of about 20 parts ethylene carbonate and 80 parts water. The bath temperature is maintained at 65° C. while the freshly formed filaments are drawn through the bath for a distance of about 40 inches. The thread is withdrawn from the bath at a speed of about 41 meters per minute and onto the thread-advancing, thread-storage reel 9 on which it is washed with a total of about 160 cubic centimeters per minute of water at about 60° C. delivered by tubes 10 and 11. In contrast to Example I, the fiber assumes a milky, opaque appearance immediately after its initial coagulation in the aqueous coagulating bath.

The washed thread leaving the reel 9 is thereafter pulled through the steam tube 14 by the heated, tapered reel 22, the thread being conducted directly thereto without any intermediate application of water. Reel 22 is driven at the rate of about 410 meters per minute thereby augmenting the stretch to a total stretch of about ten times. The heated reel 22 is maintained at a temperature of about 140° C. by circulating steam therein at about 40 pounds per square inch gauge pressure while the thread is stored thereon for about five seconds. The resulting heat-treated thread discharged from the reel and collected by cap twister 27 possesses a desirable whiteness and is permanently stabilized against further relustering and discoloration under the influence of hot, wet ironing. It has a tenacity of about 2.3 grams per denier and an elongation of 10%.

The process of spinning fibers as set forth in Example II above is the subject matter of John M. Terpay application Serial No. 245,697, filed September 8, 1951.

Among the polymers and copolymers containing at least 80% acrylonitrile which may be employed in accordance with the process of this invention may be mentioned polyacrylonitrile and copolymers of acrylonitrile with the following monomeric compounds: vinyl esters (vinyl acetate, vinyl formate, vinyl benzoate), vinyl ethers, and vinyl ketones; acrylic acid and its esters and amides; methacrylic acid and its esters, amides, and nitrile; maleic, itaconic, fumaric, crotonic acids and their esters, amides and nitriles; allyl alcohol and its esters; styrene and nuclear substituted styrenes, e. g. chloro- and dichloro styrene; halogenated monoethylenic compounds such as vinyl chloride, vinyl fluoride, and vinylidene chloride; N-vinyl compounds such as N-vinyl pyrrolidone, N-vinyl succinimide, N-vinyl carbazole, 2- and 4-vinylpyridine; and the like.

For various purposes it may be desirable to chemically and physically modify the polymeric compositions of this invention by the presence of other materials such as, for example, pigments, dyes, plasticizers, color stabilizers, spinning agents, etc.

The acrylonitrile polymers may be prepared by any suitable polymerization method such as, for example, polymerization with oxygen-yielding catalysts, e. g., benzoyl peroxide, hydrogen peroxide, tertiary butyl hydroperoxide, potassium or ammonium persulfate, etc. Redox polymerization systems employing oxygen-yielding catalysts such as the above in combination with reducing agents such as sodium bisulfate, sodium hydrosulfite, sodium formaldehyde sulfoxylate, etc., may be used with advantage. Generally, after completion of the polymerization reaction, the resulting polymer is washed with water to remove any remaining impurities, and preferably, distilled or demineralized water is employed so as to achieve a minimum of impurities in the final polymer.

The molecular weights of the polymeric materials are preferably within the range of 10,000 and 250,000, or even higher, although polymers having molecular weights between 30,000 and 100,000 may be used with particular advantage.

In general, the spinning solutions may be prepared by heating a mixture of the finely divided acrylonitrile polymer or copolymer with the ethylene carbonate solvent until the polymer is dissolved. Advantageously, the spinning solution may be maintained, prior to extrusion, at temperatures between about 60° and 150° C., and preferably between about 80° and 120° C. These spinning solutions may have with advantage a solids content between about 10% and 30% solids depending upon the molecular weight of the polymer, and preferably between about 18% and 25%.

We claim:

1. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.

2. A process for producing a wet-spun opaque fiber having a stabilized luster from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device until substantially all of the water is removed from the fiber.

3. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with an aqueous medium; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device for at least about 3 seconds.

4. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with an aqueous medium; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; shrinking said fiber on said device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device until substantially all of the water is removed from the fiber.

5. A process according to claim 4 in which the washing step and the stretching step are performed together in the presence of a heated aqueous medium.

6. A process for producing a wet-spun opaque

fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with water; stretching the resulting fiber in the presence of steam; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber is in a water-swollen state and contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; shrinking said fiber on said device by progressively diminishing the thread-bearing periphery of said device in the direction of thread-advance; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device until substantially all of the water is removed from the fiber.

7. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into a liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with water; stretching the resulting fiber in the presence of steam; continuously conducting said stretched fiber to a thread-storing, thread-advancing reel while said fiber is in a water-swollen state and contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing reel; shrinking said fiber on said reel by progressively diminishing the thread-bearing periphery of said reel in the direction of thread-advance; heating said thread-advancing reel and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated reel between about 3 and 10 seconds.

8. A process according to claim 7 in which the yarn produced has a denier between about 30 and 500.

9. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into an initial liquid coagulating medium comprising a polyhydric alcohol compound under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with an aqueous medium; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device for at least about 3 seconds.

10. A process for producing a wet-spun opaque fiber having a stabilized luster made from a polymer of acrylonitrile containing in the polymer molecule at least about 80% by weight acrylonitrile, the steps comprising, forming an opaque white fiber by extruding a polymeric spinning solution comprising said acrylonitrile polymer and ethylene carbonate as a solvent for said polymer, through a spinneret into an aqueous liquid coagulating medium under conditions such that at least a portion of the fiber coagulation takes place in the presence of water; washing substantially all of the ethylene carbonate from the fiber with an aqueous medium; stretching the resulting fiber; continuously conducting said stretched fiber to a thread-storing, thread-advancing device while said fiber contains at least about 3% by weight of water, based upon the weight of the dry fiber; imparting all of the fiber orientation prior to conducting said fiber to said thread-advancing device; advancing said fiber in generally helical turns on said thread-advancing device; heating said thread-advancing device and maintaining it at a temperature above about 120° C.; storing said advancing fiber on said heated device for at least about 3 seconds.

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