Sept. 15, 1953

K. M. McLELLAN

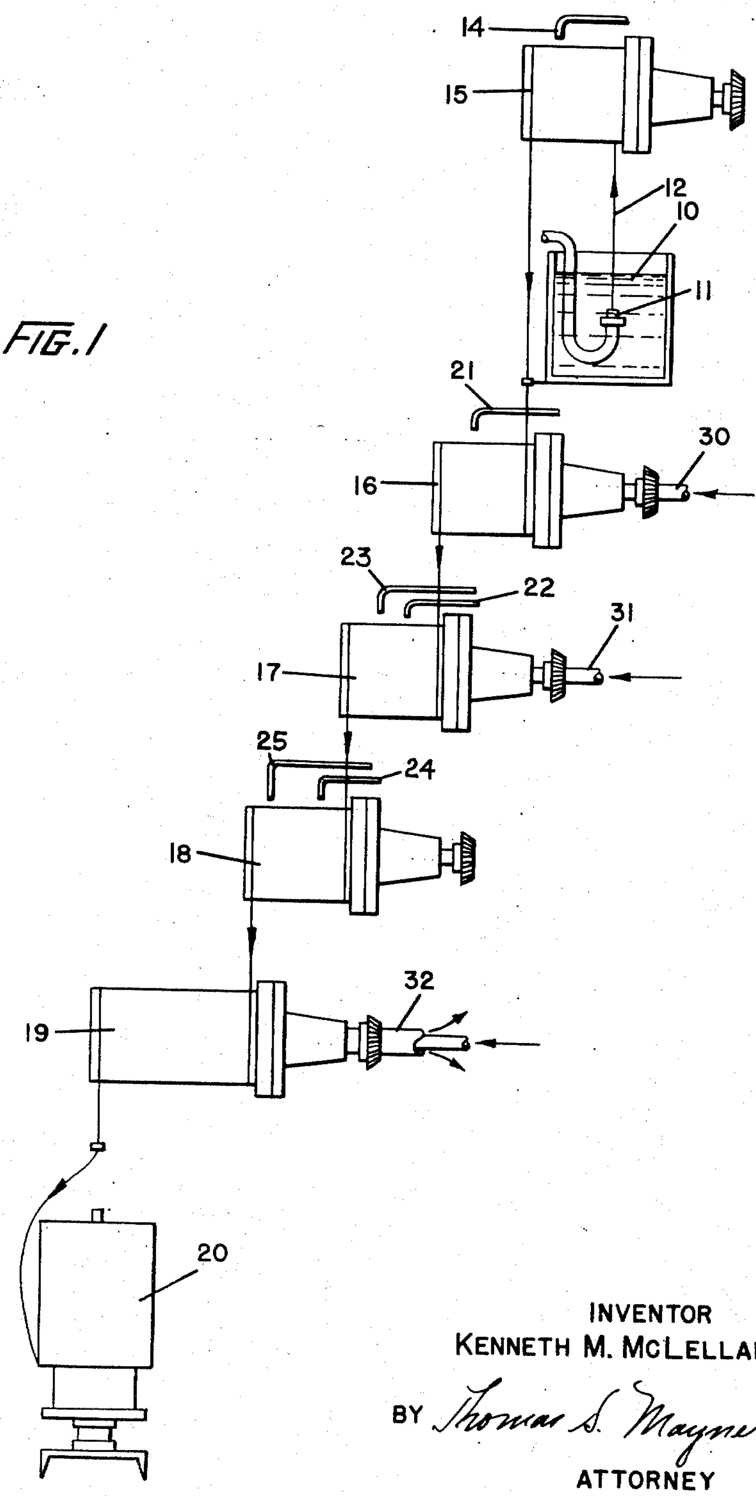
METHOD OF AFTER-TREATING VISCOSE THREADS ADVANCING IN A HELICAL PATH

2,652,311

Filed April 20, 1951

2 Sheets-Sheet 1

FIG. 1



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2 Sheets-Sheet 2

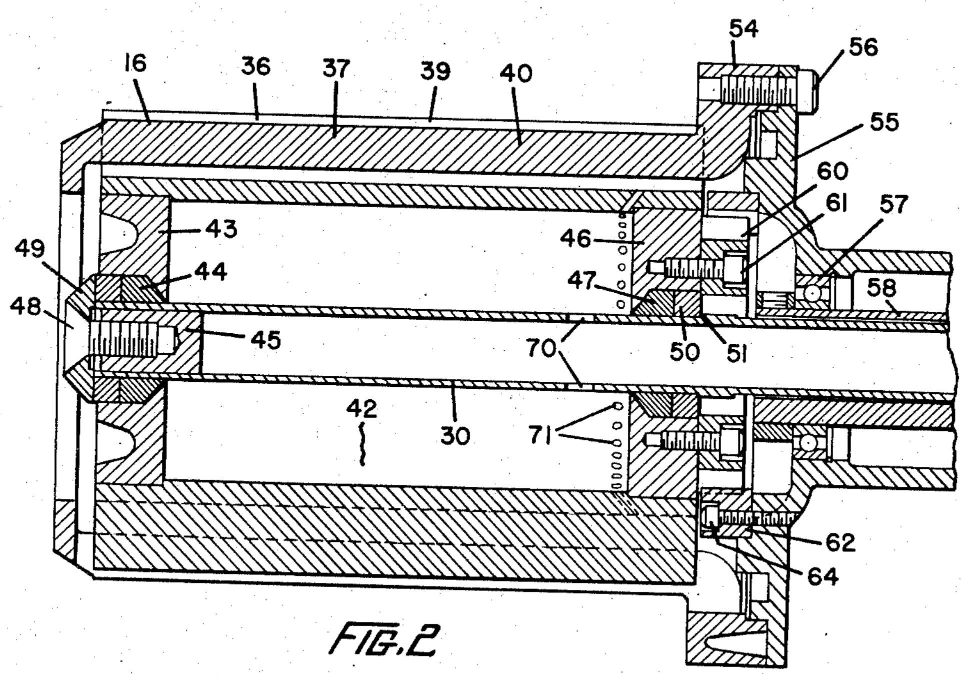

FIG. 2

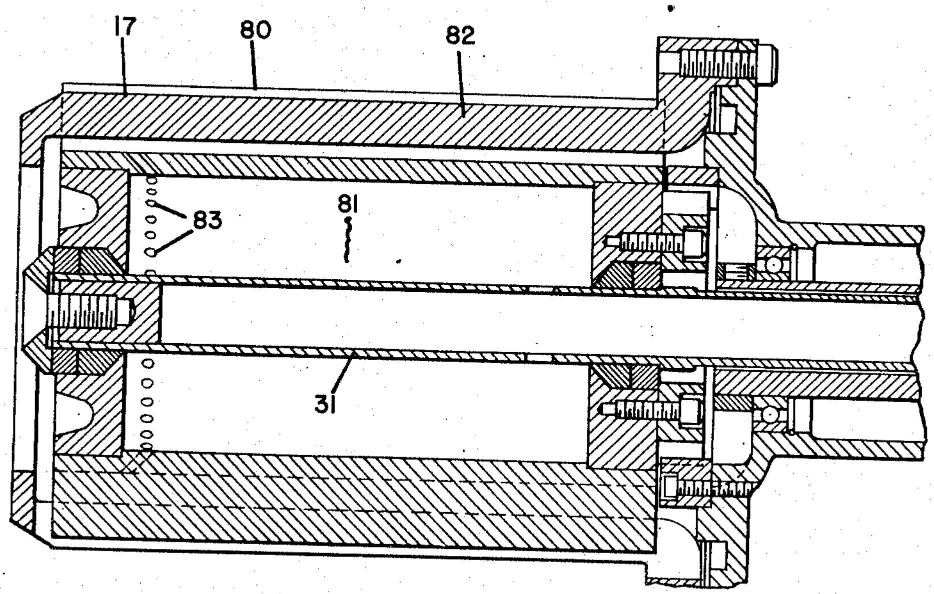

FIG. 3

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METHOD OF AFTER-TREATING VISCOSE THREADS ADVANCING IN A HELICAL PATH

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Application April 20, 1951, Serial No. 222,009

4 Claims. (Cl. 8—151.1)

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This invention relates to the treatment of filamentary material such as yarn, thread and the like (hereinafter referred to as thread) at elevated temperatures while such thread is continuously stored and advanced on a thread-advancing, thread-storage device in a plurality of generally helical turns. More particularly, it relates to the treatment of thread such as viscose rayon thread with aqueous fluids at elevated temperatures to assist the regeneration and processing of such thread.

In general, a chemical or physical treatment of thread is more thorough and more efficient if it is performed at an elevated temperature. This is particularly true, for example, in the manufacture of viscose rayon thread, and especially in the continuous processing systems where a series of coordinated thread regeneration steps are performed on a series of thread-storage, thread-advancing devices. Among the regeneration steps involved are, for example, treatments with aqueous acid solutions, aqueous desulfurizing solutions, aqueous bleaching solutions, and washing treatments with water between any two or more of the chemical treatments mentioned. One or more of the individual treatment steps mentioned may be performed on each of the thread-advancing devices.

The present invention advantageously provides a process whereby the processing time for the regeneration of viscose rayon in a continuous system is materially reduced. In addition, the present invention makes possible a continuous process apparatus which is more simple and smaller in size and cost. In accordance, then, with the present invention this is accomplished generally by temporarily storing and advancing a thread on a thread-storage, thread-advancing device in a plurality of generally helical turns, heating the advancing thread by circulating or passing steam through the interior of the device, and by-passing at least a portion of the steam from the interior of the device to the exterior thereof, and into contact with the advancing thread. Advantageously, at least a portion of the steam employed is condensed and the advancing thread subjected to the action of both the condensate and steam. Condensation of steam may take place within the advancing device or it may take place at the thread-bearing surface thereof, or at both places. Particular advantages, however, are derived when a substantial proportion of the condensate is formed within the device and then by-passed, or diverted together with the steam, to the exterior thread-helix.

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The steam condensate or steam, or both, in addition to their function as the source of heat for heating the advancing device and thread thereon also function as thread-washing and leaching media. Moreover, such hot aqueous fluids may also serve to dilute the chemical treating agents that are present on or applied to the advancing thread. For instance, the advancing thread being contacted by the diverted hot aqueous fluids may already contain one or more of the chemical treating agents previously mentioned. Such agents may be present on the thread as the result of a prior chemical treatment, or with advantage, such chemical treating agents may be applied to the heated thread-helix and the thread then subjected to the washing or diluting action of the diverted heated fluids. If desired, as illustrated hereinafter in the examples, the washing or leaching-out action may be performed by the diverted condensate and steam at a plurality of locations on a single thread-helix, i. e., both before and after an application of a chemical treating agent to such thread-helix. The dilution of chemical treating agents such as sulfuric acid, desulfurizing or bleaching solutions that may be present on or applied to the thread, may be accomplished advantageously by slightly tilting the thread-advancing device so that the condensate-wash liquid is permitted to flow along the thread-helix countercurrently of the advancing thread.

The emission of steam and water vapor in accordance with the present invention from the exterior surface of the heated thread-advancing device to the atmosphere surrounding the device performs still another very advantageous function. During the regeneration of viscose rayon thread, substantial amounts of salting out takes place on the various mechanical parts and devices such as guides, etc. associated with, or in the vicinity of, the thread-advancing devices. The aqueous liquids discharged by the thread and by the rotating devices form salt crystals and deposits where they contact such mechanical parts. Such deposits in turn cause breaks in the running thread. In a process such as that of the present invention where elevated treatment temperatures are employed, there is a greater rate of salt formation due to the higher rate of evaporation of the discharged liquids. By the process of the present invention, however, the steam and water vapor present in the atmosphere surrounding the steam-heated advancing device condense into a mist which settles on and continuously wets out the associated parts referred to, thus avoiding or minimizing the damage caused by the salt deposits.

In practicing the present invention a thread-advancing device is employed having an enclosed chamber into which the steam is introduced and advantageously circulated prior to diverting desired quantities of the condensate and steam to the exterior to the thread-helix. The chamber walls of the advancing device are provided with circumferentially spaced peripheral openings so that the heated aqueous fluids can flow therethrough in generally predetermined quantities to selected areas of the reel periphery.

Among the thread-storage, thread-advancing devices that may be employed in accordance with this invention are, generally, any that are adapted to advance thread in general helical turns of thread. Particularly advantageous for the purpose of this invention are the devices on which the traveling yarn describes a helix which is almost circular such as, for example, a thread-advancing reel that operates on the principles disclosed in the Knebusch Patent No. 2,210,914 and in the Corey Patent No. 2,413,217 and of the type illustrated in Figures 2 and 3 of the drawing. In general, this type of thread-advancing reel consists of two reel members each having a periphery of a plurality of longitudinally extending bar members, and each reel member being mounted for rotation on axes that are offset and askew relative to each other. The reel can have an enlarged chamber for condensing steam, or the reel shaft may comprise the condensing area. The chamber is contained in one of the reel members and embodies a fluid tight cylindrical chamber positioned about a hollow reel shaft through which steam is conducted. In the periphery of the cylindrical chamber there are bored a plurality of openings extending to the atmosphere having diameters which permit a predetermined flow of the heated aqueous fluid, e. g., steam condensate and steam to the periphery of the reel. These passages may be positioned circumferentially at a desired place along the length of the reel so as to provide for one or more hot aqueous fluid treating zones, e. g., hot-water washing or leaching zones, at any desired position along the reel surface. Such positions or zones may be after, before or both before and after, the point of application of a chemical treating agent, e. g., acid, desulfurizing, bleaching, etc. solutions.

This invention will be more specifically shown and described in the accompanying specification and drawing where:

Figure 1 is a schematic end view of a continuous process rayon spinning machine;

Figure 2 is a cross-section of a thread processing reel of this invention; and

Figure 3 is a further modification in cross-section of a thread processing reel of this invention.

Referring to Figure 1 of the drawing there is shown in schematic arrangement a viscose spinning apparatus in which a viscose solution is extruded through a spinneret **11** into a sulfuric acid coagulating bath **10** to form a rayon thread **12**. The thread **12** is withdrawn from the acid bath **10** by a reel **15** positioned above the bath. The thread may be merely stored thereon while containing entrained acid bath liquor or, if desired, it may be treated with a dilute aqueous acid applied through the distributor tube **14**. The thread **12**, containing acid, is forwarded by the reel **15** in a downward direction to reel **16** which is heated internally with steam supplied by hollow reel shaft **30**.

On this reel the thread is subjected to a washing treatment followed by a dilute aqueous acid treatment to further its regeneration. The washing treatment is performed as follows. A major part of the steam supplied through the hollow reel shaft **30** condenses within the reel and the condensate flows outwardly through peripheral openings in the reel to the reel surface, thus subjecting the advancing thread to a condensate-wash treatment. The steam pressure within the reel, because of the openings, is slightly above or nearly atmospheric, thus assisting the steam condensate formed in the reel to flow out to the reel surface together with the uncondensed steam. The subsequent dilute acid treatment on the heated reel **15** may be accomplished by applying such acid through tube **21**, or a dilute acid condition can be formed on the thread-helix by diluting the acid present on or entrained by the thread entering the reel **16**. The thread **12** discharged from the reel **16** is conducted to reel **17** which is heated by steam supplied through a hollow reel shaft **31**.

Reel **17** is constructed in a manner similar to reel **16** so as to permit steam and condensate formed within the reel to be diverted to the thread-helix thereon. To the thread-helix on reel **17** may be applied a desulphurizing solution through tube **22**. If desired, prior to such desulphurizing treatment the thread-helix may be subjected to a condensate-wash treatment by means of condensate diverted from the interior of the reel in a manner similar to reel **16**. Further, if desired, the desulphurizing treatment may be followed by still another condensate-wash treatment on the same reel **17**. Thread **12** discharged from reel **17** is conducted to reel **18** and treated with a mild bleaching solution supplied by delivery tube **24** and the resulting bleach-treated thread then advanced to a wash zone provided by an aqueous washing liquid delivered by tube **25**.

Further, if desired, both the desulphurizing treatment and the bleaching treatment may be performed on the reel **17** by applying such chemical agents in sequence through tubes **22** and **23**. Further, if desired, an aqueous oil emulsion may be applied to the thread-helix on reel **18** by tube **25** following the bleaching treatment supplied by tube **24**. In this manner the oil emulsion from tube **25** may serve the dual function of washing off residual bleach from the thread and also oiling the thread prior to drying on steam heated dryer reel **19**.

In contradistinction to the previously described steam heated reels **16** and **17**, the steam introduced into dryer reel **19** through hollow reel shaft **32** is recycled and the condensate formed therein is trapped and also recycled. The oiled and dried thread discharged from reel **19** is then collected by a cap spinning take-up apparatus **20**.

In general, the temperature of the heated aqueous fluids, e. g., steam and steam condensate present within the reel or undergoing diversion to the reel surface is in the neighborhood of 100° C. On the other hand, the temperature of the treating liquid and advancing thread on the reel surface may vary depending upon a number of factors such as, for example, the quantity and rate of steam introduced into the reel; the quantity and rate of steam and steam condensate diverted to the thread-helix; the size and number of the orifices employed to divert the steam and condensate; and the volume and temperature of the treating liquids delivered to the thread-helix. It is found particularly advantageous, however, to adjust and to control such conditions so as to achieve thread treatment temperatures on the reel surface at above about 75° C. and preferably between about 80° and 100° C.

Particular advantages are derived if the factors above mentioned are adjusted and controlled as follows. The steam pressure within the reel may be maintained from about atmospheric pressure to about 5 pounds and preferably at about 1 or 2 pounds. The treating liquids applied to the thread-helix advancing on the heated reel advantageously may be delivered at about room temperature at the rate of 15 to 100 cc. per minute from each delivery tube; the size of the orifices for the diversion of steam and steam condensate may range from about $\frac{1}{32}$ inch to ¼ inch depending upon the location thereof and upon the total number thereof in the steam chamber of the reel; the above factors in turn being adjusted so that a major portion of the steam introduced into the reel is condensed on the thread-helix and before it contacts the thread-helix. Thus by adjusting one or more of these factors, higher or lower thread treating temperatures may be achieved and greater or lesser quantities of condensate may be produced on and within the helix so as to provide whatever volume of condensate-wash is necessary to perform the amount of washing or diluting action desired.

The internal construction of the reels **16** or **17** is generally shown in Figure 2 of the drawing. As there shown, such a thread-advancing reel **16** advantageously comprises two reel members **36** and **37** each having a periphery of a plurality of longitudinally extending bar members **39** and **40** respectively. The bar members of the one are interleaved with those of the other. Reel member **36** is secured to the tubular shaft **30** and sealed thereto by means of fluid tight seals **44** and **47** positioned in the end enclosing sections **43** and **46**. The seals **44**, **47** are drawn fluid tight by the stud **48** turned into a thread bore in the solid front section **45** of the tubular shaft **30**. The rear seal **47** is compressed against the shaft by being forced against a collar **50** which abuts a shoulder **51** on the tubular shaft **30** and the front seal **44** is compressed against the shaft by the collar **49**.

The reel member **37** also comprises a plurality of longitudinally extending bar members **40** which interdigitate with the bar members **39** of the reel member **36**. The bar members **40** at their rearward portions merge into the flange **54** which is secured to the reel member **37** supporting flange **55** by means of studs **56**. The supporting flange **55** is adapted to rotate on spaced bearings **57** (only one set being shown) positioned about a sleeve **58** having an offset and askew axis relative to the reel shaft. The reel member **36**, because it is secured to the shaft **30**, drives the reel member **37**. The driving, however, is through the gear **60** that is attached to the end closure **46** by the studs **61**. The gear **60** is in mesh with an annular gear **62** that is attached to the supporting flange **55** also by means of studs **64**.

The hollow shaft **30** serves as a conduit for the steam. The steam flows from the shaft within the reel through openings **70** into a chamber **42** in the reel member **36**. Passages **71** are provided in the peripheral walls of reel member **36** for the flow of steam condensate and steam to the thread on the reel surface. The passages **71** are bored of any desired size so that all of the steam introduced into the reel flows through them as condensate and steam. The passages **71** open into the spaces between the bar members of the reel member **36** which spaces are occupied by the interdigitating bar members **40**. Steam at a desired line pressure up to about 5 pounds is forced into the reel chamber **42** where at least a major portion condenses because of heat given up both to the reel and to the yarn and treating liquids thereon. In this manner a sufficient amount of condensate is provided for the various aqueous treatments of the thread passing over the reel.

Where a chemical treatment is performed on the thread-helix at the take-up or flange end of a thread-advancing reel and a subsequent wash treatment of the thread-helix is desired, then condensate passages may advantageously be positioned in that part of the reel selected as the washing zone. If a flow of the condensate is desired over a greater number of thread turns, then a plurality of parallel circumferential openings can be provided so as to spread the condensate over a larger area.

There is shown in Figure 3 the heated processing reel **17** having heated-fluid passages **83** in the forward or thread discharge end of the heating chamber **81**. The reel member **80** containing the heating chamber **81**, and the reel member **82** are similar in construction and function to the reel members **36**, **37** comprising reel **16** of Figure 2.

The following examples are more specifically illustrative of preferred methods for treating thread with heated aqueous liquids while such thread is being stored and advanced on steam heated processing reels.

Example I

A thread formed of 150 denier, 40 filament viscose rayon is produced as follows. A viscose spinning solution containing about 6.5% sodium hydroxide and about 8% cellulose is prepared in the conventional manner. Upon being ripened to an index of approximately 4.5 (sodium chloride) the viscose is extruded through spinneret **11** positioned in the coagulating bath **10**, bath **10** being maintained at about approximately 45° C. and containing approximately, by weight, 12% sulfuric acid, 22% sodium sulfate, 2% zinc sulfate, and 0.1% of a cation-active agent.

The spinning solution is extruded into this bath and the newly formed yarn **14** is drawn from the bath liquid at the rate of about 90 meters per minute by means of the reel **15** on which the thread is stored and advanced while containing entrained bath liquid. The thread **12**, while still in an incompletely regenerated state and while wet with the acid bath liquid is then transferred from the reel **15** to reel **16** to which steam is metered at about 4 pounds per hour. All of the reels **15**, **16**, **17** and **18** employed in this example are tilted upwardly about 5° at their unsupported ends.

The reel **16**, as shown in Figure 2 of the drawing, is provided with openings **71** of about ⅛ inches diameter between each pair of bars and located at substantially the flange or the supported end of the reel. The reel **16** is driven at a peripheral speed of about 108 meters per minute so as to stretch the thread about 20% between reels **15** and **16**. The thread is initially wound on the reel at a point ahead of the openings **71**, so as to subject this portion of the helix to the washing action of the condensate and steam emitted from said openings. The washing action in this zone removes acid bath liquid together with various salts and thiocarbonate impurities which are present in the thread. To a subsequent portion of the helix on reel 16 there is delivered by tube 21 about 30 cc. per minute of a 2% aqueous sulfuric acid solution free of sodium sulfate and at room temperature.

The acid-containing thread discharged from reel 16 is conducted to steam heated reel 17 which is similar to reel 16 but is driven at a peripheral speed of about 112 meters per minute so as to stretch the thread another 5%. The stretched thread is then subjected to the following treatments on reel 17. To the beginning portion of the thread-helix there is applied through tube 22 about 30 cc. per minute of water at about room temperature. To a subsequent portion of the thread-helix there is applied about 30 cc. per minute of 0.2% by weight aqueous alkaline sodium sulfide solution at room temperature. Following this desulphurizing zone on the thread-helix there is formed a washing zone at the thread discharge end of the reel. This washing zone is supplied by the condensate and steam being emitted from the openings 83 as illustrated in Figure 3 of the drawing. The washed thread 12 discharged from reel 17 is then conducted to reel 18 also driven at about 112 meters per minute. The thread advancing on reel 18 is first treated with a dilute aqueous sodium hypochlorite solution having a pH of about 10 and an available chlorine content of about 0.1% and is delivered at room temperature by tube 24 at the rate of 30 cc. per minute. To a subsequent portion of the thread-helix on reel 18 there is delivered by tube 25 at 30 cc. per minute an aqueous oil emulsion containing about 1.0% of a yarn finishing oil. The resulting washed and oiled thread 12 discharged from reel 18 is transmitted to the steam heated dryer reel 19 and the resulting dried yarn after being discharged from the reel 19 is collected on the cap spinning take-up apparatus 20.

Example II

The procedure of this example is the same as Example I with the exception that a 5% aqueous sulfuric acid solution at about room temperature is delivered to the advancing thread on reel 15 at the rate of about 20 cc. per minute by delivery tube 14.

Example III

The procedure of this example is the same as Example I with the following exceptions. The sodium sulfide solution is delivered by tube 22 instead of tube 23; the hypochlorite bleach solution is delivered to the thread-helix on reel 17 by the delivery tube 23 instead of delivering such solution to reel 18; and tube 24 is set to deliver about 30 cc. per minute of water instead of the bleach solution.

Example IV

The procedure of this example is the same as Example III with the following exceptions and additions which provide for additional condensate-wash treatments on heated reel 17. Reel 17 is modified so that a set of openings or passages, similar to openings 83 of Figure 3, are positioned at about the center of the chamber 81 at a point between the delivery tubes 22 and 23 and another similar set of openings positioned at the thread take-up end of the chamber 81 in a manner and location similar to the openings 71 in Figure 2. The flow of condensate and steam from these additional openings provide wash zones which precede and also follow the desulphurizing treatment zone supplied by tube 22.

Example V

The procedure of this example is the same as Example III with the exception that an additional steam and condensate-wash zone is provided on reel 16 after the treatment of the thread-helix thereon with the 2% sulfuric acid solution supplied by tube 21.

As indicated heretofore the viscose rayon regenerating liquids which are employed in accordance with the present invention in association with the steam and steam-condensate treatment for the processing of rayon thread may comprise one or more of a number of various chemical treating liquids. Thus, the aqueous acid solutions applied to the thread undergoing regeneration on the thread-advancing devices may be of various types and may have a wide range of concentrations. For example, acids such as sulfuric, phosphoric, nitric or hydrochloric and the like may be employed, if desired, sulfuric acid being preferred. If desired, the acid solutions may have substantially the same composition as the acid coagulating bath or with advantage may consist of an acid coagulating bath composition which has been suitably diluted with water. Where oxidizing acids such as nitric are employed, however, care must be taken to maintain concentrations that will not degrade the yarn. In general aqueous sulfuric acid solutions having less dissolved salts than the coagulating bath or preferably substantially free of salts may be employed with advantage. Acid concentrations corresponding to between 0.05% and 15% by weight, and preferably between about 1% and 5% sulfuric acid are found especially advantageous.

The alkaline desulphurizing solutions may comprise the alkali metal sulfides such as sodium or potassium sulfide, trisodium phosphate, sodium sulfite, etc.

The aqueous bleaching solutions employed in the process of the present invention may comprise any of the common oxidizing materials that are used for the oxidative bleaching of cellulosic materials. They may be of acid, neutral or alkaline character and may be delivered to a thread-helix in either dilute aqueous or relatively concentrated form provided, however, that the oxidizing conditions, i. e., concentration, time of treatment, etc. that are maintained in the aqueous oxidizing zone on the thread-helix are mild enough to prevent oxidative degradation of the thread. Where the bleaching treatment is performed at elevated temperatures such as, for example, on a steam heated reel even milder conditions should be employed.

Among the bleaching agents that may be employed with advantage are the aqueous oxyhalogen type such as, for example, hypohalites and halites. Among the hypohalites may be mentioned the hypohalous acids such as, for example, hypochlorous acid, etc., or the soluble alkali metal and alkaline earth metal salts of such hypohalous acids. Typical examples of such hypohalites are chlorine water and sodium or potassium hypochlorite. Aqueous hypochlorite solutions may be employed advantageously having a pH less than about 12 and with particular advantage having a pH between about 5 and 10. They may have an available chlorine content between about 0.005 and 0.3%, and with particular advantage between about 0.05 and 0.1%.

As examples of the halites may be mentioned chlorous acids, etc. and the soluble alkali metal and alkaline earth metal salts thereof. Typical of these metal salts are sodium or potassium chlorite. For example, aqueous chlorite solutions having a pH less than about 9 and preferably between about 1 and 8 may be employed with advantage. They may have an available chlorine content between about 0.005% and 0.3% and preferably between 0.05 and 0.1%.

In practicing the present invention it is found advantageous to incorporate various surface-active compounds together with oleaginous materials into one or more of the aqueous treating liquids present on the heated thread-helix. This practice is found to be effective in scouring and removing sulfur impurities and by-products from the thread undergoing regeneration and washing. Particularly advantageous results are obtained where such compounds are incorporated into a washing medium and near-boiling treatment temperatures are employed. The presence of such compounds may also be effective in maintaining the advancing device free of harmful incrustations. Such surface-active compounds may be cationic, anionic, or even non-ionogenic in character. They may, for example, consist of sulfonated aryl, long chain alkyl, or combination alkyl-aryl substituted compounds; solubilized amines or amides containing aryl or long chain alkyl radicals; highly polymerized ethylene oxide or long chain hydrocarbons modified with polyethylene oxide radicals, etc. It was found particularly advantageous to incorporate relatively small amounts of oleaginous materials with or without surface-active agents into the steam which is introduced into the thread-advancing reels. In this manner the oleaginous material becomes dispersed in the steam and condensate and is thereafter also diverted to the exterior reel surface and thread-helix.

If desired, the surface-active compounds may be employed to disperse or emulsify various thread lubricants or other oleaginous materials

in the aqueous liquids applied to the advancing thread. The thread lubricants may, for example, consist of mineral, vegetable or animal waxes and oils. Concentrations of the order of 0.01% to 4%, by weight, or greater of the lubricant in water may be employed with advantage.

I claim:

1. In the continuous aftertreatment of viscose rayon thread including the step where the thread is heated while being advanced in the form of a helix by circulating steam therethrough the further treatment during this processing step comprising, continually heating the said helix while simultaneously substantially condensing the circulating steam inside the said helix, meanwhile continuously applying a treating liquid to a portion of said helix, continuously immediately radially passing said condensate as a wash to a subsequent portion of the said same helix.

2. In the continuous aftertreatment of viscose rayon thread of claim 1 in which the applied treating liquid is an acid regenerating liquid.

3. In the continous aftertreatment of viscose rayon thread of claim 1 in which the applied treating liquid is an aqueous bleaching solution.

4. In the continuous aftertreatment of viscose rayon thread of claim 1 in which the applied treating liquid is an aqueous desulphurizing liquid.

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