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MANUFACTURE OF FIBERS

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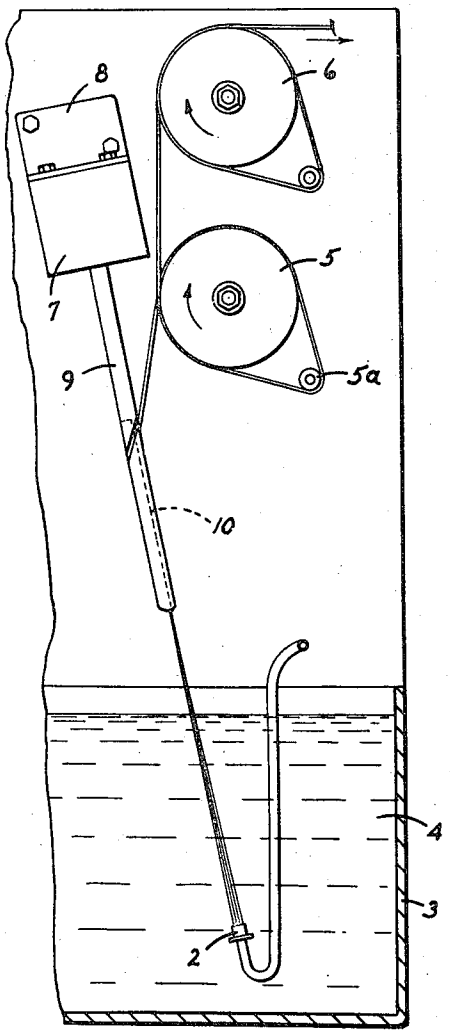


Fig. 1

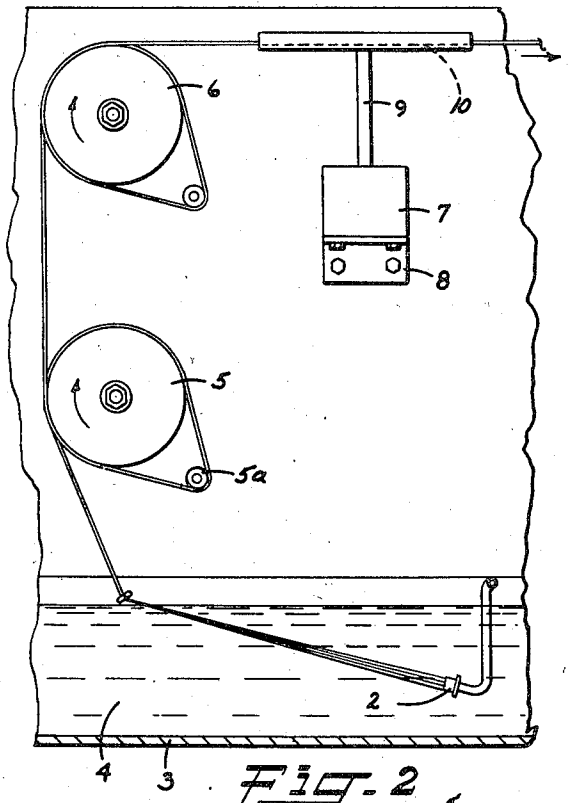


Fig. 2

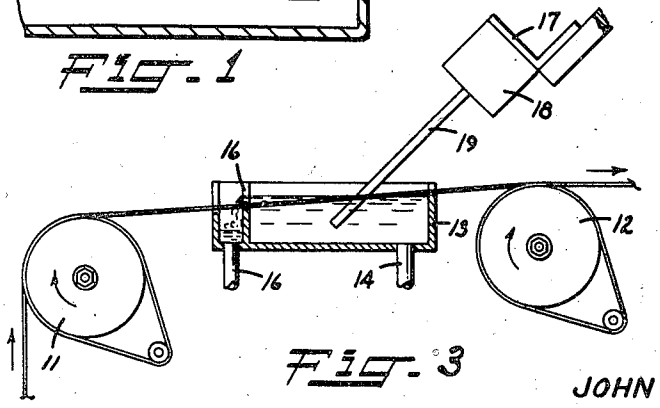


Fig. 3

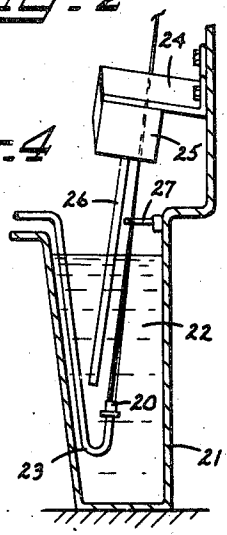


Fig. 4

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MANUFACTURE OF FIBERS

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This invention relates to improvements in the manufacture of artificial fibers from solutions of fiber-forming materials.

While the invention may be practiced with advantage in connection with the manufacture of artificial fibers from solutions of fiber-forming materials of various types, it will be described in detail in connection with the manufacture of regenerated cellulose fibers from viscose by the wet spinning method, by way of specific exemplification.

Cellulose, as shown by Meyer (Brochemische Zeitschrift, 214,253–281, 1929) is a linear superpolymer, i. e., a superpolymer in which the crystalline regions or micelles comprising molecular chains or bundles of molecular chains show a high degree of orientation along the fiber axis. Although in viscose, the crystallites or micelles are randomly dispersed and oriented in all directions or agglomerated into small gel-like masses, they tend to reorient themselves in the direction of the fiber axis during spinning. According to modern concepts, the physical strength of fibers of regenerated cellulose depends upon the ratio of crystalline portions to amorphous portions, homogeneity at all portions of the fibers, and the degree to which the crystalline portions are reoriented with respect to the fiber axis. It is known that stretching of freshly spun fibers comprising partially regenerated cellulose improves the crystallinity and orientation of the crystallites along the fiber axis, with improvement in the tenacity of the fibers. However, regenerated cellulose fibers obtained by conventional procedures are not homogeneous and do not show maximum uniform crystallite orientation consistent with good extensibility, even after being subjected to strong stretching while in the gel state in which they are initially obtained.

In accordance with conventional procedures, the fibers are manufactured by extruding viscose through a spinneret into an acid coagulating and regenerating bath, and continuously withdrawing the fibers formed by the action of the bath away from the spinneret face and to the exterior of the bath for eventual after-treatment and purification.

During the spinning operation and concomitantly with conversion of the freshly formed highly plastic or gel-like fibers to the form in which they are finally set up, the fibers undergo a series of changes due to various chemical processes such as dehydration, dexanthation of the cellulose xanthate, etc. which proceed more or less simultaneously. While the fibers are undergoing

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such changes, and the molecules are more or less in a state of flux, a number of factors arise which interfere with the freedom of the crystallites to satisfy their natural tendency to reorient themselves in the direction of the fiber axis, as for instance, the stresses and strains which tend to develop in the molecular structure during the spinning operation, the difficulty of controlling the rate at which dexanthation proceeds at all portions of the fiber cross-section, and at all points along the fiber length, with regeneration of the cellulose, and doubtless other factors, as well.

Under usual manufacturing conditions, the fibers are withdrawn from the bath in the form of a gel comprising partially regenerated cellulose, dependence being placed upon diffusion of the acid carried from the bath through the skin initially set up on the fibers in the bath about a still fluid core to effect complete regeneration of the cellulose at all portions of the fiber cross-section. The rate of diffusion of the adhering acid to the interior of the fiber, and the uniformity of such diffusion, controls the rate at which the xanthate groups are split off and the uniformity of the dexanthation at any given moment, which in turn controls the speed with which the fibers are finally set up, with formation of hydroxyl-to-hydroxyl bonds between the cellulose molecules. Apparently, the speed with which the hydroxyl-to-hydroxyl bonds are formed affects the degree to which the molecules are reoriented with respect to the fiber axis, since, obviously, when such bonds have been formed, the crystallites are less free to undergo molecular rotations and align themselves along the fiber axis.

Under usual spinning conditions, it is difficult, if not impossible, to control the rate at which dexanthation proceeds at all portions of the fiber cross-section and along all portions of the fiber length. At some portions of the freshly spun fibers, a greater number of xanthate groups appear to be split off, with more rapid complete regeneration of the cellulose, in a given time period, than occurs at other portions, which apparently is one factor leading to lack of homogeneity in the final fibers and their failure to show the maximum uniform crystallinity and crystallite orientation consistent with good extensibility, which theoretically should be effected by the stretching to which the fibers are subjected during their production.

One object of the present invention is to provide a method of manufacturing artificial fibers which exhibit a high degree of micellar orienta-

tion with respect to the fiber axis. Another object is to provide a method of manufacturing regenerated cellulose fibers from viscose characterized by maximum uniform tenacity consistent with sufficient extensibility to render the fibers suitable for commercial purposes. A further object is to provide a method of manufacturing regenerated cellulose fibers from viscose which permits a greater measure of control over the changes which take place in the freshly formed fibers, before they are finally set up, to yield more homogeneous and uniform fibers, and the delivery of the fibers in the more homogeneous condition to the after-treatment stages.

In accordance with the present invention, freshly formed artificial fibers are subjected, at any time prior to final setting up thereof, that is, while they are in the highly plastic condition in which they are obtained initially and before final setting of the molecular structure, in the case of regenerated cellulose fibers, while they are in the form of a gel comprising partially regenerated cellulose, to the radiations of sound waves having a frequency in the range of about 1000 to a million or more cycles per second, herein called high frequency sound waves.

By subjecting the freshly formed highly plastic or gel-like fibers to the high frequency sound wave radiations it is possible to exercise control over the various processes which take place prior to final setting up of the fibers, and to radically influence the molecular structure of the final fibers, with material improvement in their physical properties. Thus, under the influence of the extremely rapid longitudinal alternate waves of compression and rarefaction to which fibers comprising partially regenerated cellulose are subjected, in accordance with the invention, either in the spinning bath or soon after their withdrawal from the bath and while they are in the acid-moist condition, it is possible to control the rate of diffusion of the acid through the fibers so that it is uniform at all portions of the fiber, with consequent uniform control of the rate at which dexanthation and regeneration of the cellulose proceeds to completion, thus insuring that, before final setting up of the fibers, the crystallites are uniformly free to align themselves in the direction of the fiber axis. The high frequency sound wave radiations have the effect of accelerating the rate of diffusion of the bath to the interior of the fibers. However, although the diffusion rate is accelerated as compared to the rate at which it proceeds normally, it is uniform at all portions of the fiber, and under the conditions of the invention, by appropriate choice of the frequency and magnitude of the waves, the duration of exposure of the fibers to the radiations, and the stage in the manufacture at which the sound wave radiations are applied, the rate of dexanthation may be controlled to the extent that the fibers are stretched while the cellulose is only partially dexanthated under which conditions optimum orientation of the crystallites is realized. Preferably, in addition to the jet stretch to which the fibers are subjected incidental to withdrawal thereof from the spinning bath, the fibers are given a predetermined after-stretch, as measured, for example, by differential in godet speeds, and acceleration of dexanthation is so controlled that, during the after-stretching step, the cellulose is only partially dexanthated, the dexanthation being completed, however, soon after the fibers leave the after-stretching zone, and the after-stretched fibers being in a condition for immediate processing and purification, without requiring a period of storage to permit complete regeneration of the cellulose, or only a shortened storage period for that purpose. By uniformly controlling the rate of diffusion of the acid to all portions of the fiber cross-section, and the rapidity with which the fibers are finally set up, it is possible to control the degree of reorientation of the crystallites with respect to the fiber axis within the limits in which maximum uniform increase in the tenacity of the fibers is obtained without embrittlement of the fibers.

The sound waves may be applied either directly or indirectly to the fibers, threads, or yarns, at all stages in their manufacture, from the time of their formation to just prior to subjectiig them to the usual after-treating processes, or at any one or more of such stages. Further, it is within the scope of this invention to subject the fibers, while they are in the form of a gel comprising partially regenerated cellulose, simultaneously or successively to the radiations of sound waves having different frequencies within the range stated and of different magnitudes.

The fibers may be subjected to the sound wave radiations concurrently with their formation in the spinning bath, as they proceed through the bath, before, during, or after subjecting them to after-stretching, after collection in the form of a wound package or other thread mass and during storage of the mass to permit complete regeneration of the cellulose by the adhering spinning bath, as they proceed to a thread storage, thread-advancing reel, which may also be a stretching reel, or as they are advanced over such a reel. When the high frequency sound wave field is established in the path of a thread proceeding from one stage to another, as from the spinning bath to a stretching zone, or from one thread handling device to another, as between godets or between a thread-handling godet and a stretching reel, the waves may be applied in any direction, with respect to the direction of travel of the thread, as for instance in a direction parallel to the direction of travel, or transversely thereof.

The freshly spun fibers may be subjected to the high frequency sound wave radiations prior to final setting up thereof, set up, with or without after-stretching thereof, and, at a subsequent stage, again brought to unplasticized condition and after-stretched, if desired.

The sound waves may be generated by any suitable means, as by piezo electric sound generators, magneto-strictive sound generators, electromagnetic sound generators, etc.

The accompanying drawing will serve to illustrate various specific embodiments of the invention. In the drawing

Fig. 1 is an elevation, partly in section, of apparatus suitable for carrying out one modification of the invention;

Fig. 2 is an elevation, partly in section, of apparatus suitable for carrying out another modification;

Fig. 3 is an elevation, partly in section, of apparatus suitable for carrying out still another modification of the invention; and

Fig. 4 is a cross-section through a spinning tank of a vertical spinning bath, the tank being divided into a plurality of troughs each constituting an individual unit of the spinning machine, one trough being shown.

Referring to Figure 1, viscose is extruded through spinneret 2, positioned in vessel 3 con-

taining the spinning bath 4, the spinneret being arranged to spin generally vertically upwardly to the godets 5 and 6, which are diven (by means not shown) at different peripheral speeds for imparting a predetermined stretch to the fibers. A sound generator 7 is supported above the level of the bath, in a bracket 8 secured to the side of the spinning machine, and associated with the generator is a transmission tube 9. The tube is provided with a slot or groove 10 through which the thread travels to the godets, the tube thus serving not only to transmit the high frequency vibrations to the threads or fibers, but also as a guide for controlling the thread as it proceeds to the lowermost godet, and for removing excess coagulating liquid from the thread. The thread is passed around godet 5, over a guide 5a, and thence over godet 6. The fibers, threads or yarns leaving the spinning bath are thus subjected to the high frequency sound wave radiations as they enter the stretching zone.

In Figure 2, a different arrangement is illustrated, the sound generator 7 supported in bracket 8 being mounted on the side of the spinning machine so that the thread is subjected to the sound wave radiations as it proceeds from the stretching zone. In Figure 2 the spinneret 2 is positioned for horizontal spinning.

Figure 3 is illustrative of an apparatus set-up for subjecting the fibers to high frequency sound wave radiations simultaneously with stretching thereof. As shown, the fibers are passed between godets 11 and 12 disposed with their axes in a horizontal plane above the bath (not shown) the godets being driven at different peripheral speeds to impart the desired stretch to the fibers. Positioned between the godets is a trough 13 which is supplied with liquid, such as hot water or hot dilute acid, through pipe 14, the trough being provided, adjacent one end, with a slotted partition 15 forming an overflow chamber which discharges through pipe 16. Supported above the trough in a bracket 17 is a sound generator 18 having an associated transmission tube 19 which extends into the liquid in trough 13. The fibers, threads or yarns travelling through the liquid are indirectly subjected to the high frequency sound wave vibrations transmitted to the liquid by tube 19.

It will be readily understood that godet 12 may be replaced by a thread-advancing reel and that the reel may be driven at a peripheral speed relative to the speed of godet 11 such that a predetermined amount of stretch is imparted to the thread as it passes through trough 13. Alternatively the thread may be passed over a pair of thread handling and advancing godets of different sizes, the upper and longer godet being driven, and the lower and smaller godet being an idler, or a pair of godets of the same size and driven at the same speed may be used, the thread being passed from the upper godet through a trough 13 as described to the thread advancing reel and stretched as it passes through the vibrating plasticizing liquid in trough 13.

With reference to Fig. 4 which is illustrative of an arrangement for subjecting the fibers to the effects of high frequency sound wave radiations concurrently with their formation, there is shown one unit of a spinning machine comprising a spinning tank or other similar container for the spinning bath which, it will be understood, is divided into a plurality of compartments or troughs separated by partitions and each accommodating a single spinneret and provided

with suitable means for introducing fresh spinning bath into each trough and removing the spent liquid, a sound generator being associated with each trough. As shown in Fig. 4 a spinneret 20 is positioned in the trough 21 containing the bath 22, viscose or other fiber-forming solution being extruded in generally vertical direction upwardly through the spinneret which is joined to a rounder 23 in turn connected with a spinning pump (not shown). Mounted in a bracket 24 supported on the face of the spinning machine is a sound generator 25 having a transmission tube 26 extending into the bath to the spinneret and through which the high frequency sound wave vibrations are applied to the spinning bath, adjacent the spinneret, and in a direction generally parallel to the direction of spinning. The spinning solution emerging through the spinneret orifices is subjected to the sound wave radiations, and the fibers formed by the action of the bath are exposed to the effects of the sound waves as they are drawn through the bath and through guide 27 to a thread handling device (not shown).

Alternatively, the spinneret may be arranged to spin in a horizontal direction, and in the case of either vertical or horizontal spinning, the sound wave radiations may be applied in any direction relative to the direction of spinning.

The invention is not limited to the manufacture of fibers of regenerated cellulose from viscose, or to the manufacture of fibers from fiber-forming solutions by wet spinning methods, but may be practiced in the manufacture of fibers of different chemical constitution from solutions of various fiber-forming materials. Thus, fibers of regenerated proteins may be subjected to high frequency sound wave radiations while they are in the plastic condition in which they are initially obtained, to assist in the agglomeration of polypeptide chains into parallel bundles, as occurs in the manufacture of fibers from solutions of casein, for example.

The invention is also of advantage in the manufacture of fibers from solutions of cellulose esters, such as cellulose acetate, by either the dry or wet spinning methods. Cellulose acetate fibers obtained by spinning the solution of the ester into an evaporative atmosphere, such as heated air or other fluid medium, and while still in the plastic condition in which they are obtained initially, or such fibers obtained by spinning the solution into a setting bath which may comprise, for instance, an aqueous salt solution, hydrocarbons, or oils, etc., may be subjected to high frequency sound wave radiations, for the obtention of fibers having enhanced tenacity by virtue of improved micellar orientation.

Modifications and variations may be made in the specific procedures described herein, without departing from the spirit and scope of the invention as defined by the appended claims.

I claim:

1. The method comprising extruding a solution of an organic polymeric fiber-forming material capable of being formed into fibers which exhibit crystallinity into a setting medium to form initially highly plastic fibers, and prior to converting the fibers from their initially plastic condition to a final, stretched unplasticized condition, subjecting the fibers to high frequency sound wave radiations having a frequency of at least 1000 cycles per second.

2. The method comprising extruding a solution of an organic polymeric fiber-forming material capable of being formed into fibers which

exhibit crystallinity into a setting medium to form initially highly plastic fibers, subjecting the plastic fibers, before final setting thereof, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second and thereafter stretching the fibers while they are in the plastic condition.

3. The method comprising extruding a solution of an organic polymeric fiber-forming material capable of being formed into fibers which exhibit crystallinity into a setting medium to form initially highly plastic fibers, stretching the plastic fibers, and thereafter subjecting the plastic fibers, prior to final setting thereof, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second.

4. The method comprising extruding a solution of an organic polymeric fiber-forming material capable of being formed into fibers which exhibit crystallinity into a setting medium to form initially highly plastic fibers, and thereafter simultaneously stretching the fibers and subjecting them to high frequency sound wave radiations having a frequency of at least 1000 cycles per second, while they are in the highly plastic condition and before final setting thereof.

5. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, and prior to converting them to a final, stretched, unplasticized condition subjecting the partially regenerated cellulose fibers to high frequency sound wave radiations having a frequency of at least 1000 cycles per second.

6. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, subjecting the partially regenerated cellulose fibers to high frequency sound wave radiations having a frequency of at least 1000 cycles per second, and thereafter stretching the fibers while they are in plastic condition.

7. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, stretching the fibers, and thereafter subjecting the fibers while they are still in highly plastic condition and before final setting thereof to high frequency sound wave radiations having a frequency of at least 1000 cycles per second.

8. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, and simultaneously stretching the fibers and subjecting them to high frequency sound wave radiations having a frequency of at least 1000 cycles per second while they are in highly plastic condition and before final setting thereof.

9. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, withdrawing the fibers from the bath, stretching the fibers in plastic condition, storing the stretched fibers under conditions to effect complete regeneration of the cellulose, and subjecting the fibers during such storage, and before completion of the cellulose regeneration, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second.

10. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, advancing the fibers from the point of their formation to a collecting stage, and subjecting the fibers while they are in highly plastic condition and before complete regeneration of the cellulose, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second applied in a direction transverse to the direction of travel of the fibers to the collecting stage.

11. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, advancing the fibers from the point of their formation to a collecting stage, and subjecting the fibers while they are in the highly plastic condition and before complete regeneration of the cellulose, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second applied in a direction parallel to the direction of travel of the fibers to the collecting stage.

12. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, advancing the fibers from the point of their formation to a collecting stage, and subjecting the fibers while they are in the plastic condition and before complete regeneration of the cellulose, to high frequency sound wave radiations having a frequency of at least 1000 cycles per second applied in a direction transverse to the direction of travel of the fibers to the collecting stage, while simultaneously stretching the fibers.

13. The method comprising extruding viscose into a spinning bath to form initially highly plastic fibers comprising partially regenerated cellulose, advancing the fibers from the point of their formation to a collecting stage, and subjecting the fibers while they are in the plastic condition and before complete regeneration of the cellulose to high frequency sound wave radiations having a frequency of at least 1000 cycles per second applied in a direction parallel to the direction of travel of the fibers to the collecting stage, while simultaneously stretching the fibers.

14. A method as defined in claim 1 in which the setting medium is a liquid and the radiations are applied to the fibers as they proceed through the liquid setting medium.

15. A method as defined in claim 5 in which the radiations are applied to the fibers as they proceed through the spinning bath.

16. A method as defined in claim 10 in which the radiations are applied to the fibers as they proceed through the spinning bath.

17. A method as defined in claim 11 in which the radiations are applied to the fibers as they proceed through the spinning bath.

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