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MANUFACTURE OF ARTIFICIAL MATERIALS

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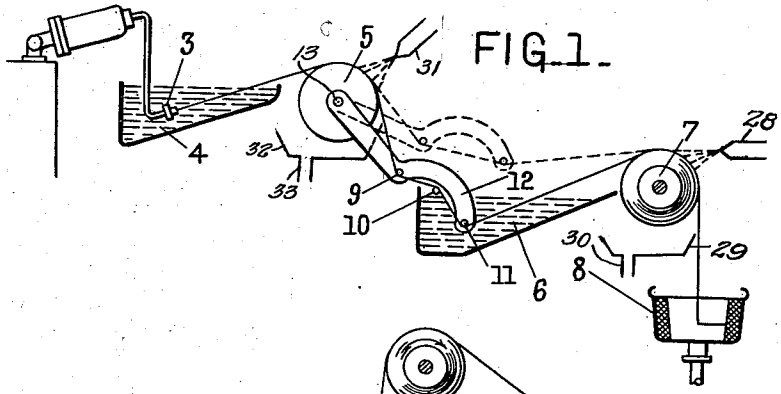


FIG. 1.

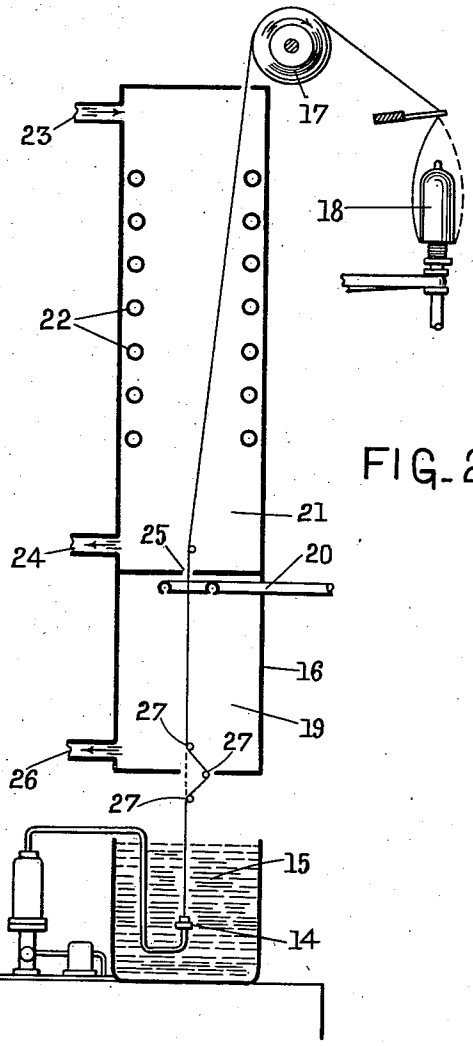


FIG. 2.

HENRY DREYFUS
WILLIAM I. TAYLOR
INVENTORS

J. Kelly & C. W. Seviour

ATTORNEYS

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MANUFACTURE OF ARTIFICIAL MATERIALS

Henry Dreyfus, London, and William Ivan Taylor, Spondon, near Derby, England, assignors to Celanese Corporation of America, a corporation of Delaware

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13 Claims. (Cl. 18—54)

This invention relates to the manufacture of artificial filaments, threads, ribbons or other products by methods involving the coagulation of spinning solutions by means of liquid coagulating media. The object of the invention is the production of filaments of fine or extremely fine denier. Another object of the invention is the production of filaments which are possessed of increased strength, whether such filaments are of normal, fine, or extremely fine denier, or otherwise. The invention is applicable to the production of any type of artificial filaments capable of being spun by the wet or coagulation method. Thus, the filaments may have a basis of cellulose acetate or other derivative of cellulose, or of reconstituted cellulose, such as is spun from viscose or cuprammonium solutions of cellulose.

According to the invention, a spinning solution is extruded in the form of filaments, ribbons, or other products which are partially coagulated by means of liquid media, a skin-like outer layer being thereby formed on the products, while the core remains soft or plastic. The products are then subjected to a softening treatment which makes the above-mentioned outer layer extensible, and while in this condition, stretch is applied to the products. Coagulation or setting of the products is then completed by subjecting them to the action of further setting medium, which may be applied as a liquid, or, in some cases, as an evaporative atmosphere.

Softening of the outer skin may be carried out by applying to the filaments or other products a liquid having the requisite solvent or softening action on the substance of the outer layer, or an atmosphere consisting of or containing solvent vapour may be used, alone or supplementary to a liquid softening medium.

The process may be directed to the production of fine or extremely fine denier filaments or products of low cross-section, or to the production of filaments or other products of increased strength, whatever their denier or cross-section. For the purpose of obtaining strong filaments or other products, the applied stretch may be wholly or partially limited to that part of the filaments or products of which the outer skin has been softened. This limitation of the incidence of the stretch may be applied whether the filaments or other products are coarse, normal, or fine, but particularly valuable results are obtained when the limitation is resorted to in spinning operations which are directed to the production of fine or extremely fine denier, since the reduction in denier can at the same time be accompanied by an increase of strength.

Filaments or other products produced according to the invention may be given a further stretching after completion of the coagulation or setting, so as to reduce their denier or cross-section and increase their strength.

The method of carrying the invention into effect will now be given in greater detail, but it is to be understood that the following description is by way of example only, and is in no way limitative.

According to one application of the invention to the processes for the production by wet spinning methods of artificial filaments from viscose or cuprammonium spinning solutions, extrusion of the solution takes place into a suitable coagulating bath from which the filaments are withdrawn when the setting has resulted in the formation of an outer skin on the surface of the filaments, the interior of the filaments remaining soft or plastic. The length of path of the filaments through the bath and/or the concentration of the bath are chosen to give the desired amount of setting.

After leaving the bath, the filaments are subjected to the action of a solvent or softening liquid such as zinc chloride, Schweitzer's reagent, or a thiocyanate, e. g. ammonium, sodium, or calcium thiocyanate, which may be applied in any convenient manner. For example, the filaments may be led into and through a bath of the liquid or along a trough which is fed with a stream of the liquid, preferably in a direction opposite to the travel of the filaments. The concentration of the softening liquid and/or the duration of its action on the filaments are such that the outer skin formed in the setting bath is softened. The liquid may alternatively be sprayed onto the filaments, or applied by devices such as rollers, rods, or wicks moistened with the liquid. Care should be taken to avoid the filaments sticking together while they are in a softened state.

The filaments emerging from the coagulating bath may, if desired, be washed before the softening liquid is applied, particularly if a relatively strongly acid setting bath be employed for producing the partial coagulation.

Stretch may be applied to the filaments by passing them, after the outer skin has been softened, round a roller having a peripheral speed greater than the speed of extrusion, the softening of the outer skin enabling or assisting the filaments to withstand the stress of stretching and to receive any desired reduction in denier.

Any desired extension of the filaments may be effected by suitable adjustment of the stretch in relation to the rate of extrusion of the filaments. In order to facilitate the application of stretch to the filaments, the stretching roller may be of conical form, the filaments being taken round a smaller diameter of the roller when starting up and afterwards traversed on to a larger diameter when stretching is to commence.

In order to complete the setting of the filaments by setting the soft or plastic core remaining during the stretching operation, a further coagulating bath, which may be either acid or alkaline, and of any desired length and concentration, is employed, the filaments on emerging from this bath being collected on bobbins, in centrifugal boxes, or otherwise.

By employing such rates of extrusion, sizes of the spinning orifices, and other spinning conditions as would give low denier in normal wet-spinning porcesses, the application of the steps of the present invention enables a reduction of this already fine denier to be obtained.

In order to produce filaments or products of still further increased strength, means may be provided to confine the stretch wholly or partly to the softened part.

Thus, after leaving the first setting bath, and before or while receiving the softening liquid, the filaments may encounter means such as a roller, guide, or tension device which offers a resistance to their passage, the amount of the resistance determining whether the stretch is in part allowed to extend to the unsoftened filaments or wholly confined to the softened portion. Thus, the filaments may be caused to pass through a gate-tension device, which is preferably adjustable to enable its resistance to be regulated. A form of tension device particularly suitable for the purpose comprises "gates" of polished steel bars, adjustment being provided to cause the bars to intermesh. The bars are arranged to open apart to allow the filaments to be passed through on starting up, and, when stretch is to be imparted are adjusted by the intermeshing of the bars. Other arrangements of two or more bars may be employed to offer the required resistance by their engagement with the filaments. Thus, guides used for leading the filaments from the first setting bath and/or into the softening bath or trough may be used for the purpose.

Alternatively, a roller may be arranged to receve the filaments, this roller being allowed or caused to rotate at a peripheral speed either equal to or greater than the speed of extrusion according to whether the stretch is to be wholly or partly restricted to the softened part of the filaments.

The means for confining the stretch may be combined with the means for applying the solvent or softener. Thus, when one or more wicks are used to apply the solvent or softener, the passage of the filaments over the wick or wicks may be caused to restrict the stretch. Resistance may be imparted gradually, and, if desired, simultaneously with the traversing of the filaments on to the larger diameter of the conical stretching roller.

It will be understood that where the stretch is only partly confined to the softened part of the filaments, part of the stretch may operate to reduce the denier of the filaments during extrusion in a manner corresponding to the stretching employed in normal "stretch-spinning" processes, the remainder of the stretch resulting in increased strength being given to the filaments.

By applying restricted stretch as described above, stronger filaments are produced, and by applying this stretch together with conditions which would normally give low denier filaments, fine or extremely fine filaments can be produced which at the same time exhibit this increased strength.

Similar methods to the above may be adopted for the treatment of artificial filaments or other products of nitrocellulose or of cellulose acetate or other organic derivatives of cellulose in their production by the wet method. The setting bath, of any suitable nature, operates to form an outer skin on the filaments or products, leaving the interior soft or plastic, and the products after leaving the setting bath have the outer skin softened to enable them to receive stretch which is applied by a roller of conical or other form. The softening may be carried out by means of troughs or boths contaning liquids such as alcohol/ether, amyl acetate, acetone, alone or mixed with alcohol or water, acetic acid, formic acid, lactic acid, acetyl acetone, dimethyl-ethylene glycol, monomethylethylene glycol, monoethylethylene glycol, dioxane, diacetone alcohol, methyl or ethyl lactate and mixtures thereof. The liquids may be diluted, if necessary, to regulate their solvent or softening power on the cellulose ester or ether.

Instead, however, of employing solvents or softeners in liquid form, they may be used as vapours, the filaments or other products whose outer skin is to be softened being led through a chamber having an atmosphere consisting of or containing the vapour.

After the filaments have been softened and stretched, the completion of the setting may be effected either by the dry or evaporative method or means of a coagulating or setting liquid.

Other organic derivatives of cellulose which may be used for the purpose of the present invention are, for example cellulose formate, propionate, and butyrate, or methyl, ethyl, or benzyl cellulose.

The accompanying drawing shows diagrammatically two methods of carrying the invention into effect, Figure 1 illustrating a process in which the partially coagulated filaments are treated with a liquid softening agent and are subsequently set by means of a liquid coagulating agent and Figure 2 a process in which the partially coagulated filaments are treated with a vaporous softening agent and are subsequently set by the dry or evaporative method..

Referring to Fig. 1, the spinning solution, which may for example be an ordinary viscose spinning solution or a 20–26% solution of cellulose acetate in acetone, is extruded by means of a nozzle **3** into a coagulating bath **4**, the composition of the bath and length of travel of the filaments therein being so adjusted as to obtain only a partial coagulation of the filaments with the formation of an outer skin. Using a viscose spinning solution the bath may have the following composition by weight:—

	Per cent
Sulphuric acid	7.5
Sodium sulphate	14.5
Glucose	12.0
Water	66.0

while with the cellulose acetate solution, the bath may be a 10–12% aqueous solution of diacetone alcohol or of methyl or ethyl lactate. In both cases the length of travel of the filaments in the bath may be about 4–6 inches. The filaments, which consist of an outer skin and a soft or

plastic core, are passed round a roller 5 rotating at a peripheral speed of 40–50 metres per minute. This speed may be equal to or higher than the speed of extrusion of the solution from the nozzle 3. While traveling round the feed roller the filaments are sprayed with a suitable softening liquid, for instance a concentrated solution of zinc chloride or of sodium or calcium thiocyanate in the case of viscose or a concentrated aqueous solution of acetone (e. g. 70% acetone) or alcoholic acetone (e. g. 50%) in the case of cellulose acetate, and the filaments are thereafter carried into the second coagulating bath 6, which may be of the same composition as that of the first bath 4, the length of immersion being about 6–10 inches. For this purpose, a spray 31 may be used, a tray 32 with a drainage tube 33 being provided to carry away excess liquid. The filaments are carried thence to a conical feed roller 7 and finally into a centrifugal spinning box 8, where they may be washed. In order to stretch the partially coagulated softened filaments, the feed roller 7 is rotated at a higher peripheral speed than the feed roller 5, for example at a speed of 60–100 metres per minute or even higher. To confine the stretch thus applied to the softened part of the filaments, the latter are passed round smooth glass bars 9, 10, 11 between the feed rollers 5 and 7, the bars 9 and 11 being carried by arms 12 mounted on the shaft 13 of the roller 5, while the bar 10 is fixed in relation to the bath 6. On starting up, the filaments from the roller 5 are passed under the bars 9 and 11, which are raised for that purpose into the position shown in dotted lines, and are passed round the small diameter of the roller 7. The members 12 are then lowered so that the bars 9 and 11 form intermeshing "gates" with the fixed bar 10, the filaments being then traversed slowly to the large diameter of the roller 7, so that the stretch is applied gradually.

A similar apparatus may be employed for applying the softening liquid from a bath. In this case the filaments may be passed round the roller 5, where they may, if desired or requisite, be sprayed with a washing liquid, and thence round the bars 9, 10, 11 into the bath 6 which contains the softening liquid. The filaments emerging from this bath may be carried as before round the conical roller 7, rotating at a higher peripheral speed than the roller 5, into a further coagulating bath (not shown), round a third feed roller and finally into the centrifugal spinning box. The third feed roller may be rotated at the same speed as the roller 7, or, if still further stretching is desired, at a higher speed. If desired or requisite, a suitable washing treatment may be applied between the softening and the final setting, for example by spraying the filaments while they are on the roller 7 as by means of the spray 28, a tray 29 with a drainage tube 30 being provided to carry off excess liquid. In this bath method of applying the softening liquid. the stretch may be rigidly confined to the softened part of the filaments, i. e. that part between the bar 11 and the roller 7.

Referring now to Fig. 2, the spinning solution, which may again be a 20–26% solution of cellulose acetate in acetone, is extruded through a spinning nozzle 14 into a coagulating bath 15, designed to coagulate the filaments partially, leaving a soft or plastic core. A 10–12% aqueous solution of diacetone alcohol or of methyl or ethyl lactate with an immersion length of 4–6 inches is again very suitable. The filaments leaving the coagulating bath are passed into a cell 16 round a conical feed roller 17 to a cap-spinning device 18. The cell 16 has a lower compartment 19 which is fed by means of a pipe 20 terminating in a perforated ring with vapours of acetone or other volatile solvent or softening agent. The vapours may consist wholly of the solvent or softening agent or the latter may, if desired, be diluted with air or other suitable gas or vapour. The upper compartment 21 of the cell 16 is furnished with heating pipes 22 serving to heat the air or any other suitable evaporative medium entering at 23 and leaving at 24. The orifice 25 through which the filaments pass from the compartment 19 to the compartment 21 is preferably so dimensioned that a proportion of the air or other evaporative medium passes into the compartment 19 and out at 26. Alternatively the outlets 24 and 26 may be supplied with suitable suction apparatus to achieve this result. It is found advantageous to have a small proportion of the air or other medium passing through the compartment 19. On starting up, the filaments are passed round the small diameter of the conical roller 17 and are slowly traversed to the large diameter, which serves to impart thereto a speed higher, and preferably considerably higher, than the speed of extrusion of the spinning solution through the nozzle 14, for example 2–5 times or more. If desired, the stretch thus imparted may be confined to the softened portion of the filaments inside the compartment 19. To effect this, the filaments may be passed round suitable smooth bars 27, positioned so as to divert the filaments through a considerable angle and thereby prevent the stretch from operating, or allow only a certain proportion of the stretch to operate, upon the filaments before the softening treatment.

What we claim and desire to secure by Letters Patent is:—

1. Process for the production of artificial filaments, threads, ribbons or other products, comprising partially coagulating a spinning solution so as to form products having a skin-like outer layer, applying a softening treatment and stretching and completing the setting of the products.

2. Process for the production of artificial filaments, threads, ribbons or other products, comprising partially coagulating a spinning solution containing an organic derivative of cellulose so as to form products having a skin-like outer layer, applying a softening treatment and stretching and completing the setting of the products.

3. Process according to claim 2, wherein the softening is effected by application of a solvent or softening agent in liquid form.

4. Process according to claim 2, wherein the products comprise a substitution derivative of cellulose and the softening is effected by means of a solvent or softening agent for said derivative of cellulose products applied in vapour form.

5. Process according to claim 2, wherein the solvent of the spinning solution is a volatile liquid and the setting of the products is completed by the dry or evaporative method.

6. Process according to claim 2, wherein the stretch is confined to the softened portion of the filaments or other products.

7. Process according to claim 2, wherein stretch is also applied to the filaments or other products during the initial partial coagulation.

8. Process according to claim 2, wherein a further stretching is applied, subsequent to that applied to the softened products.

9. Process according to claim 1, characterized in that the spinning solution consists of a solution of cellulose adapted to yield products of the cellulosic type.

10. Process according to claim 1, characterized in that the spinning solution consists of viscose.

11. Process according to claim 2, characterized in that the spinning solutions contain cellulose esters.

12. Process according to claim 2, characterized in that the spinning solutions contain cellulose acetate.

13. Process for the production of artificial filaments, threads, ribbons or other products, comprising coagulating an acyl cellulose spinning solution so as to form products having a skin-like outer layer, applying a softening treatment and stretching and completing the setting of the products.

HENRY DREYFUS.
WILLIAM IVAN TAYLOR.