

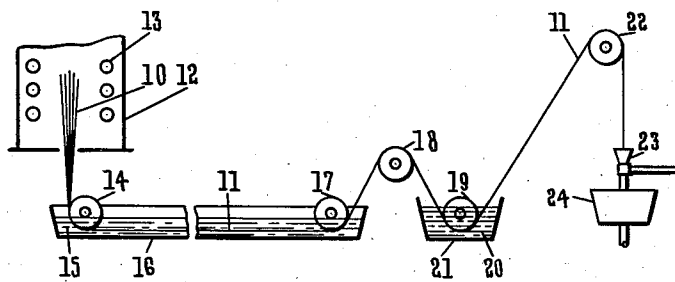
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MANUFACTURE OF ARTIFICIAL MATERIALS

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## UNITED STATES PATENT OFFICE

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## MANUFACTURE OF ARTIFICIAL MATERIALS

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This invention relates to the manufacture of artificial textile and other materials, by the evaporative process, and is particularly concerned with the manufacture of materials of this sort of enhanced tensile strength. This application is a continuation in part of application S. No. 403,140 filed October 28, 1929.

The process of the invention comprises a step resulting in the formation of the products by an evaporative process, hereinafter referred to as the "spinning" operation, followed by the step of stretching the formed products.

In the dry spinning process as commercially practised, the length of travel of the filaments, temperature, air flow and speed of spinning are always such that the filaments emerging from the spinning zone retain at the most a proportion of volatile solvent quite inadequate materially to facilitate stretching of the product. In fact, the spinning conditions are such that the maximum recovery of the solvent consistent with obtaining a product having the desired physical characteristics is effected.

According to the present invention, filaments, threads, ribbons and like materials are formed by extruding a solution of a film forming base in a volatile solvent into an evaporative atmosphere and are continuously passed, while they still retain substantial quantities of a stretch-assisting agent contained in said solution, into a bath of stretch-assisting agent and stretched. The term "volatile solvent" includes solvent mixtures, which need not include any true solvent for the base provided the mixture is a solvent therefor, and which need not be volatile as a whole at the temperature of the evaporative atmosphere provided sufficient evaporation takes place at that temperature to form the materials from the solution. The stretch-assisting agent is preferably a major constituent of the spinning solution and may with advantage constitute the whole of the volatile solvent thereof. The proportion of the stretch-assisting agent retained in the materials when they are passed into the bath is preferably at least 20% on their weight and may with advantage be more e. g. 25 or 30% up to 40 or 50%. The proportion may indeed be as great as is consistent with operating the process without breakage of the materials.

The process of the invention greatly facilitates the attainment of equilibrium between the solvent and the material to be stretched. This is of considerable importance in ensuring uniformity of the product and in enabling stretching to be

rapidly effected even when the degree of stretch is considerable, without loss of uniformity.

The invention is generally applicable in the manufacture of filaments, threads, yarns, fibres, ribbons, films and the like by the evaporative process, and in particular to the manufacture by this process of materials made of or containing derivatives of cellulose. The invention is described herein with particular reference to products the basic material of which is a derivative of cellulose.

The invention is not limited to the employment of any particular form of dry spinning apparatus; the apparatus may be of any form capable of being worked so as to deliver to the stretching apparatus products of the desired sort. The spinning apparatus may for example be of the type described in U. S. Patent No. 1,602,125 wherein spinning is effected in a downward direction in an enclosing casing, and the formed products are led continuously out of the casing. The drawings of U. S. Patent No. 1,602,125 show a casing providing a travel of about 6 feet from the extrusion dies to the opening in the casing through which the products issue therefrom, and with this form of apparatus in spinning solutions of cellulose acetate in acetone spinning speeds of from 100 to 200 or even 300 metres per minute are usual. In carrying out the spinning process of the present invention, the length of path may be considerably reduced without sacrifice of speed so as to retain in the products the desired proportion of volatile solvent.

Reducing the length of travel of the materials through the spinning zone is however merely one expedient which may be used in the spinning operation of the invention to limit evaporation and so bring about the retention in the products of the desired amount of the stretch-assisting agent, and the invention is not limited to any particular means for obtaining this result. The means may for example comprise limiting evaporation by reducing the flow of evaporative medium through the spinning zone. Reduction of the flow of evaporative medium below a certain value may result in the production of products of flat cross-section. Though in normal spinning these are to be avoided, in the present invention their production in the spinning operation is not objectionable, since the subsequent treatment is capable of removing such defects.

An alternative or additional means of limiting the amount of evaporation effected consists in reducing the temperature of the spinning zone. The possibility of spinning at lower temperatures

relative to the boiling point of the solvent makes possible the use of less volatile solvents than those customarily employed, without necessitating undesirably high temperatures. If, on the other hand, the ordinary, more volatile solvents are used, such for example as acetone, the temperature of the spinning zone may be lower, and in consequence the spinning apparatus may be simplified. Again, the proportion of volatile solvent to cellulose acetate or other basic material, in the dope, may be increased beyond that which is commercially practicable where a fully set product is desired as a result of the spinning operation. Hence basic materials of very high viscosity characteristics may be used in relatively dilute solution, whereas the same materials in the concentrations normally used would give spinning solutions of undesirably high viscosity. The advantages attendant on the use of high viscosity materials are referred to later.

Reduction of the amount of solvent lost by the products in the spinning operation by increasing the speed with which they pass through the spinning zone is another possibility. In determining the spinning speed to be adopted, however, due regard must be had to the time factor of the subsequent treatment with the assisting agent which the products have to undergo.

As a solvent for the basic material single liquids or mixtures of two or more liquids may be used in the dope. Among suitable solvents for cellulose acetate mention may be made of acetone, methyl ethyl ketone, methylene ethylene ether and dioxane. Mixtures of latent solvents with non-solvents, which may or may not themselves be latent solvents or swelling agents, capable of forming with them solvent mixtures, may be used provided that spinning can be effected so that the liquid remaining in the materials is capable of facilitating stretching thereof.

The stretch-assisting agent applied in the bath is preferably of the same nature as that remaining in the products from the spinning operation. The two may however be different, provided they are miscible. The two stretch-assisting agents may be such that the mixture has greater stretch-assisting properties than either of the constituents alone. Where the two stretch-assisting agents are different, it is of advantage, with a view to their recovery, that they should be readily separable. Among the large number of stretch-assisting agents for cellulose acetate the following may be mentioned: acetone, diacetone alcohol, the esters, ethers and ether-esters of olefine glycols and of poly-olefine glycols; for example the di-methyl ether of ethylene glycol, and the mono methylene ether of diethylene glycol, methylene ethylene ether and dioxane, glycol mono-acetate, methyl glycol mono-acetate, ethyl lactate, diethyl tartrate and mono- and di-acetins. The composition of the stretch-assisting medium should not be such that under the conditions of the treatment it dissolves the material to any appreciable extent. Where strong solvents for the material are used therefore, the presence of a diluent, for example water, is advisable. Excellent results have been obtained when the spinning solution comprises a solution of cellulose acetate in dioxane, and the formed products are treated with an assisting agent comprising a 40-50% aqueous solution of the same solvent.

Preferably the products emerging from the spinning zone are stretched by means of a series of godets or rollers rotated within the bath con-

taining the stretch-assisting agent, means being provided for continuously replenishing the stretch-assisting agent in the bath.

One method of carrying out the invention is illustrated in the accompanying drawing.

In the drawing a bundle of filaments 10, formed within the spinning cell 12, of which only the lower portion is shown, and which is provided with heating pipes 13, is passed from the cell under a roller 14 partially immersed in a stretch-assisting liquid 15 contained in the bath 16, carried round a further roller 17, also immersed in the stretch-assisting bath, and rotating at a higher speed than the first roller, and from thence over a godet 18, under a roller 19 partially immersed in a washing bath, round a further godet 22 into a funnel guide 23 of a centrifugal spinning box 24 wherein the materials are collected.

As described below further rollers rotating at speeds intermediate between those of the rollers 14 and 17 may with advantage be interposed so as to effect the stretch in stages.

The filaments may be brought together prior to entering the stretching bath, so as to form a single filament of large denier. Preferably, however, the individual filaments are kept separate until after entering the stretching bath.

Stretching may be effected in two or more stages, and between stages the tension may be relaxed and the products allowed to shrink in the presence of the stretch-assisting agent. This intermediate shrinking step is important in that the tensile strength of the products appears to be enhanced more by the application of a given stretch in two or more stages with "rest-intervals" between than by the application of the same stretch in one stage.

The invention is not limited to imparting any particular degree of stretch to the materials. They may, for instance, be stretched to 200 or 300% of their original length or to 500% or more. The invention enables very fine filaments to be obtained and a very high tenacity e. g. 2 or 2.5 up to 3 or more grams per denier is obtainable in the products of the invention.

The following example illustrates the invention:—

#### Example

A 25% solution of cellulose acetate in dioxane is extruded into an evaporating atmosphere maintained at an average temperature of 100° C. and drawn through said atmosphere at such a rate that the filaments formed as they emerge from the spinning zone contain 25-35% on their weight of dioxane. The filaments pass from the spinning zone, through a short air path in which very little further evaporation occurs, under a roller partially immersed in a bath containing 40-45% aqueous dioxane, and in succession round two further rollers also partially immersed in the bath, after which they leave the bath, pass through a short washing bath, and are collected. The second roller in the stretching bath rotates at 1.5 times the speed of the first and the third roller at twice the speed of the second.

The filaments may be brought together to form a single thread before entering the stretching bath but are preferably kept apart, in which case they may be twisted simultaneously with winding, by means, for instance, of a centrifugal spinning box. When this form of collecting device is used it is unnecessary to dry the filaments before collecting them since the most convenient way of assisting their passage through the funnel guide

of the spinning box is by means of a slow stream of water or other liquid.

The most important application of the invention is in connection with materials comprising cellulose acetate. Other organic derivatives of cellulose may however be employed, for example other organic esters, including cellulose formate, propionate and butyrate, cellulose ethers, including methyl, ethyl and benzyl cellulose, and mixed ether-esters of cellulose, for instance oxy-ethyl cellulose acetate. The basis of the material may moreover be an inorganic ester of cellulose, for example one of the various cellulose nitrates, of composition intermediate between the trinitrate and the dinitrate. The invention is not limited to the use of materials comprising derivatives of cellulose having any particular degree of etherification or esterification. In the case of cellulose acetate, however, I prefer to use esters having an acetyl content calculated as acetic acid of between 50 and 60%.

As a basis for the materials of the invention derivatives of cellulose having a relatively high viscosity are to be preferred, particularly since the use of such materials allows of the production of products of still greater tensile strength than those obtainable using a basic material of lower viscosity. Thus, where the basic material comprises cellulose acetate, although cellulose acetates of viscosity of 10 or even less are not excluded, it is preferable to employ an acetate of viscosity at least 30, and still more advantageous when the viscosity is between 50 and 100 or even between 100 and 200, measured by comparing the viscosity of a 6% solution of the cellulose acetate in acetone at 25° C. with that of glycerine at the same temperature, taken as a standard of 100.

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

1. Process for the manufacture of artificial filaments, threads, ribbons and like materials, which comprises, as a continuous operation, forming the materials by extruding a solution of a filament-forming base in a volatile solvent into an evaporative atmosphere, passing them, while they still retain 25–50% on their weight of a volatile stretch-assisting agent contained in said solution, into a bath containing the same stretch-

assisting agent and stretching them during their passage through said bath.

2. Process for the manufacture of artificial filaments, threads, ribbons and like materials, which comprises, as a continuous operation, forming the materials by extruding a solution of an organic derivative of cellulose in a volatile solvent into an evaporative atmosphere, passing them, while they still retain 25–50% on their weight of a volatile stretch-assisting agent contained in said solution, into a bath containing the same stretch-assisting agent and stretching them during their passage through said bath.

3. Process for the manufacture of artificial filaments, threads, ribbons and like materials, which comprises, as a continuous operation, forming the materials by extruding a solution of cellulose acetate in a volatile solvent into an evaporative atmosphere, passing them, while they still retain 25–50% on their weight of a volatile stretch-assisting agent contained in said solution, into a bath containing the same stretch-assisting agent and stretching them during their passage through said bath.

4. Process for the manufacture of artificial filaments, threads, ribbons and like materials, which comprises, as a continuous operation, forming the materials by extruding a solution of cellulose acetate in a volatile solvent comprising acetone into an evaporative atmosphere, passing them while they still retain 25–50% on their weight of acetone into a bath containing acetone and stretching them during their passage through said bath.

5. Process for the manufacture of artificial filaments, threads, ribbons and like materials, which comprises, as a continuous operation, forming the materials by extruding a solution of cellulose acetate in a volatile solvent comprising dioxane into an evaporative atmosphere, passing them while they still retain 25–50% on their weight of dioxane into a bath containing dioxane and stretching them during their passage through said bath.

6. Process according to claim 1, wherein the materials are stretched in the bath to at least 200% of their original length.

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