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MANUFACTURE OF ARTIFICIAL PRODUCTS

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8 Claims. (Cl. 18—54)

This invention relates to the manufacture of filaments, yarns, threads, ribbons, films and the like, by the dry-spinning of solutions of cellulose acetate or other cellulose esters or ethers.

In my U. S. specification No. 1,566,384, I have described the advantages which accrue, in the manufacture by dry spinning methods of artificial threads or filaments from solutions of cellulose esters or ethers, by incorporating in the spinning solutions a solvent of higher boiling point than the main solvent of the spinning solutions, and particularly solvents having boiling points between 100 and 150 to 170° C. The said specification instances, for use in conjunction with acetone solutions of cellulose acetate, the following higher boiling solvents:—acetone-alcohol, acetyl-acetone, cyclo-pentanone, diacetone-alcohol and cyclo-butanone.

The incorporation of the said higher boiling solvents retards the rate of the evaporation of the volatile solvent and the rate of the solidification of the solution, and therefore facilitates the drawing out of the filaments. In addition, products of very round cross-section may thereby be produced. Similar results are achieved by incorporating in the spinning solutions higher boiling non-solvents or precipitants, as described in my U. S. specification No. 1,616,787.

Again, in my U. S. application S. No. 328,305 filed December 24th, 1928, I have described the advantages which are obtained by incorporating in the spinning solutions higher boiling non-solvents together with still higher boiling solvents, U. S. application S. No. 522,530 filed March 13th, 1931, being concerned with specific proportions of the higher boiling non-solvents and solvents.

My U. S. application S. No. 535,284 filed May 5th, 1931, further describes processes in which spinning solutions of cellulose acetate or other cellulose ester or ether, in a mixture consisting of a volatile solvent and a higher boiling solvent with or without a non-solvent, are dry spun into an atmosphere charged with vapours of a non-solvent.

I have now found that improved results may be obtained in all the above processes by employing, as a higher boiling solvent, a solvent which dissolves the cellulose acetate or other cellulose ester or ether under consideration to form a solution of relatively high viscosity as compared with a solution of the cellulose derivative in the volatile solvent alone, for example cellulose acetate in acetone. Thus, for instance, I may incorporate in the spinning solutions solvents which form solutions of cellulose acetate having a viscosity at least four times the viscosity of the corresponding solution of cellulose acetate in acetone. Preferably the higher boiling solvent yields a solution of the cellulose derivative having at least 6 times, or even 10 or 20 times, the viscosity of the corresponding solution in acetone.

Very useful results indeed are obtained by employing as the higher boiling solvent mono- or diacetin or glycol monoacetate. Methyl glycol acetate also gives good results, as does methyl glycol itself, but in general they are not so good as the partial esters of polyhydric alcohols already mentioned. Dioxane, benzyl formate, butyl lactate and diethylene glycol monoacetate may also be employed. The proportions in which such higher boiling solvents may be incorporated in the spinning solutions may be similar to those already indicated in the patents mentioned above, and furthermore, as described in U. S. applications S. Nos. 328,305 filed December 24th 1928, 522,530 filed March 13th 1931, and 535,284 filed May 5th, 1931, a non-solvent may also be incorporated in the spinning solution and/or be present in vaporous form in the evaporative atmosphere.

If desired, after the spinning operation is complete the higher boiling solvents or any proportion thereof which remains in the products may be wholly or partially removed, as for example by washing. Further, the presence of said high boiling solvents in the products may be used to facilitate a stretching operation applied after the products are fully set.

By means of the invention, not only is the draw down of the filaments or other products improved, so that fine denier filaments of low cross-section are easily obtained, but in addition the cross-section itself is very good and uniform, the cross-section of filaments, for example, being unusually round. Draw down may be still further improved by suitable heating of the spinning solution. Again, by providing a large input of heat in the region of the nozzles hollow or voluminous filaments may be produced (compare U. S. application S. No. 375,151 filed July 1st, 1929, and U. S. application S. No. 379,853 filed July 20th, 1929).

While the invention has been described above more particularly with reference to cellulose acetate, it may be applied to dry-spinning of any suitable cellulose derivatives, as for example, cellulose formate, cellulose propionate, cellulose butyrate, cellulose nitroacetate, cellulose nitrate, or other esters or mixed esters of cellulose, cellulose ethers or mixed ethers, as for example ethyl, propyl, butyl or benzyl cellulose, and cellulose ether-esters, e. g. ethyl cellulose acetate and oxyethyl cellulose acetate.

The following examples are given by way of illustration, but it is to be clearly understood that they do not limit the invention in any way:—

Example 1

A 25% solution of cellulose acetate in a mixture of 65 parts of acetone and 10 parts of mono-

acetin is spun through jets of about .08 mm. diameter into an evaporative medium maintained at a temperature of about 70°–80° C. Products having improved properties, e. g. good cross-section and extensibility, are obtained.

Example 2

A 25% solution of cellulose acetate in a mixture of 66 parts of acetone, 5 parts of xylene and 4 parts of glycol mono-acetate is spun under similar conditions to those of Example 1, and products of improved properties are obtained.

Example 3

A cellulose acetate solution such as that specified in Example 2 is spun into an evaporative atmosphere maintained at about 70°–80° C. and containing water vapour corresponding to saturation at a temperature of about 45°–55° C.

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

1. In a process for the manufacture of artificial filaments, threads, ribbons and like materials, the steps of forming a solution which comprises an organic derivative of cellulose of normal viscosity characteristics, and which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent, and extruding said solution through a shaping device into an evaporative atmosphere.

2. In a process for the manufacture of lustrous artificial filaments, threads, ribbons and like artificial materials, the steps of forming a solution which comprises an organic derivative of cellulose of normal viscosity characteristics, and which contains, in addition to a relatively volatile solvent, a non-solvent for the organic derivative of cellulose in an amount equal to 6 to 12% based on the weight of the solvent medium, and a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent, and extruding said solution through a shaping device into an evaporative atmosphere.

3. In a process for the manufacture of lustrous artificial filaments, threads, ribbons and like artificial materials, the steps of forming a solution which comprises an organic derivative of cellulose of normal viscosity characteristics, and which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent, and extruding said solution through a shaping device into an evaporative atmosphere which contains the vapors of a non-solvent for the organic derivative of cellulose other than that derived from the spinning solution.

4. In a process for the manufacture of lustrous artificial filaments, threads, ribbons and like artificial materials, the steps of forming a solution which comprises an organic derivative of cellulose of normal viscosity characteristics, and which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent, together with 6 to 12%, based on the weight of the solvent medium, of a non-solvent for the organic derivative of cellulose having a boiling point intermediate between that of the volatile solvent and that of the higher boiling solvent, and extruding said solution through a shaping device into an evaporative atmosphere.

5. In a process for the manufacture of artificial filaments, threads, ribbons and like artificial materials, the steps of forming a solution which comprises cellulose acetate of normal viscosity characteristics, and which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the cellulose acetate of a partial ester of a polyhydric alcohol capable of forming a solution of the cellulose acetate having a viscosity at least six times that of the corresponding solution of the cellulose acetate in the volatile solvent, and extruding said solution through a shaping device into an evaporative atmosphere.

6. A spinning solution for use in the manufacture of artificial filaments, threads, ribbons and like artificial materials which comprises an organic derivative of cellulose of normal viscosity characteristics dissolved in an organic solvent medium which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent.

7. A spinning solution for use in the manufacture of lustrous artificial filaments, threads, ribbons and like artificial materials which comprises an organic derivative of cellulose of normal viscosity characteristics dissolved in an organic solvent medium which contains, in addition to a relatively volatile solvent, a non-solvent for the organic derivative of cellulose in an amount equal to 6 to 12% based on the weight of the solvent medium and a proportion of at least 16% on the weight of the organic derivative of cellulose of a higher boiling solvent capable of forming a solution of the organic derivative of cellulose having a viscosity at least six times that of the corresponding solution of the organic derivative of cellulose in the volatile solvent.

8. A spinning solution for use in the manufacture of artificial filaments, threads, ribbons and like artificial materials which comprises cellulose acetate of normal viscosity characteristics dissolved in an organic solvent medium which contains, in addition to a relatively volatile solvent, a proportion of at least 16% on the weight of the cellulose acetate of a partial ester of a polyhydric alcohol capable of forming a solution of the cellulose acetate having a viscosity at least six times that of the corresponding solution of the cellulose acetate in the volatile solvent.

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