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METHOD OF PREPARING ARTIFICIAL THREADS

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11 Claims. (Cl. 18—54)

This invention relates to the manufacture of threads and films made from cellulose esters and ethers.

In British Patent 352,445 there has been described and claimed a process for the purpose of giving to threads made from cellulose esters and ethers a structure analogous to that of natural fibers, that is to say, an axially regular crystalline structure recognizable as such by means of X-ray analysis, which carries with it a great improvement of the properties of the thread, particularly of the tensile strength.

These results have been obtained by putting threads in contact with a neutral organic swelling medium and thereby producing a highly swollen condition of the thread and high degree of plasticity, and subjecting these threads to a stretching of at least 200% of their original length, the state of super-plasticity of the thread being such that a stretching of the thread of 1000% or even more can be easily obtained.

The swelling medium referred to in British Patent 352,445 as being particularly possessive of the necessary qualities to produce the state of super-plasticity and highly swollen condition is a mixture of approximately 50% dioxane and approximately 50% water, and it is employed at room temperature.

I have found that results analogous to those obtained in British Patent 352,445 can be obtained with acetone-water baths.

I have found that by using an acetone water bath at room temperature it is impossible to effect on a finished thread, made either by dry or wet spinning process, a stretching which would give to the thread an axial crystalline structure.

If the concentration of acetone is too small, it is not possible to effect any stretching but, in proportion as the concentration of acetone is increased, stretching can be effected. However, at no time, even at a concentration greater than 40% by weight of acetone, does one approach the state of super-swelling and plasticity which characterizes the dioxane-water mixtures of concentration described in the aforementioned British patent. The threads obtained do not possess the properties of threads stretched out in the dioxane-water mixture particularly the axially regular crystalline structure.

On the other hand, I have discovered that I can produce cellulose acetate threads possessing axially regular crystalline structure, recognizable as such by X-ray analysis, by stretching the threads in an aqueous solution of acetone which can be comparatively dilute, provided that it is maintained at a relatively high temperature, the thread being introduced into this solution not in the form of a finished thread but in the form of a thread swollen with acetone at the moment when it emerges from the spinneret.

Specifically, I have found that by wet spinning a cellulose acetate solution into a coagulation bath of an aqueous acetone solution under the conditions hereinafter more fully explained I can produce an artificial thread of high tensile strength and having an axially regular crystalline structure recognizable as such by X-ray analysis.

The temperature of the aqueous acetone bath is higher than room temperature and it should be near and even above that of the boiling point of acetone. Satisfactory results have been secured when the bath was at a temperature of from 50° C. to 65° C. The concentration of acetone of the media used should be from 20% to 30% by weight. I have found that by working in this manner it is not necessary in order to obtain glossy threads to add to the super-swelling bath substances which retard the action of precipitation. This is possibly due to the fact that I work with a stretching greater than 1000%.

I have also found that it is highly advantageous in carrying the invention into effect to carry out the stretching at a very high speed, that is to say, that the finished thread should be wound at a speed of at least 100 meters a minute, and this speed can reach 250 or even 300 meters a minute. These winding speeds are given only by way of example and are not limitative. In general, I have found that the more the speed of thread-making is raised, the more the concentration of the acetone in the coagulation bath can be reduced.

The process described can be easily carried out utilizing spinning solutions of cellulose acetate in some suitable solvent, preferably acetone, whose concentration can vary according to the original viscosity of the cellulose ester. This concentration may, for example, be between 20% and 35%.

The spinnerets used have holes the diameters of which are always greater than 0.12 millimeter and preferably lie between 0.15 and 0.20 millimeter.

In order to diminish the pressure of the spinning solution at the spinneret and to facilitate the diffusion of the acetone at the moment of making the thread, it is useful to maintain the spinning solution before making the thread at

a raised temperature preferably of the order of the coagulation bath. To simplify matters, the coagulation bath itself can be utilized as a heating fluid for the spinneret head. In this case the temperature of the spinning solution will be the same as that of the coagulation bath.

The thread produced according to the present invention possesses axially regular crystalline structure and a strength of more than 3 grams to the denier. A strength of 4 grams can be reached and probably even more. The elongation at the breaking point is about 6% to 7%. After or during the making of the thread it can be twisted, lubricated, dressed, oiled, or otherwise treated to facilitate its final employment in various textile operations. It may be submitted to a saponification treatment to give it an affinity for direct cotton dyes, and it may also be submitted to a swelling treatment with or without tension in order to increase its elongation at breaking point.

In order to more fully explain the invention, there are hereafter set forth several specific embodiments thereof. It is to be understood that the specific examples are illustrative of the invention and that the invention is not restricted thereto.

Example 1

A spinning solution containing 27% cellulose acetate dissolved in acetone was spun into an aqueous coagulation bath containing 23% to 25% of acetone by weight and maintained at a temperature of 63° C. The spinneret holes were 0.18 millimeter, and the speed of winding was 114 meters a minute.

The thread obtained gave a titre of 60 deniers for 80 filaments and possessed a tensile strength of 3.5 to 3.8 grams per denier with an elongation at breaking point of 6% to 7.5%.

Example 2

A spinning solution composed of 35% cellulose acetate dissolved in acetone was spun into an aqueous coagulation bath containing 28% to 29% acetone by weight maintained at a temperature of 50° C. The winding speed was 100 meters a minute and the spinneret used had holes of 0.18 millimeter. The thread obtained gave a titre of 100 deniers for 150 filaments and had a tensile strength of 3.1 grams per denier and an elongation of 6.2%.

Since it is obvious that various changes and modifications may be made in the above description without departing from the nature or spirit thereof, this invention is not restricted thereto except as set forth in the appended claims.

I claim:

1. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a cellulose acetate spinning solution through spinnerets having holes, the diameters of which are greater than .12 millimeter and not over .20 millimeter, in a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of the order of the boiling point of acetone, and winding the thread at a speed of at least 100 meters per minute.

2. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a cellulose acetate solution through spinnerets having holes from .15 to .20 millimeter in diameter into a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of from 50° C. to 65° C., and winding the thread at a speed of at least 100 meters per minute.

3. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes, the diameters of which are greater than .12 millimeter and not over .20 millimeter, in a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of the order of the boiling point of acetone, and winding the thread at a speed of at least 100 meters per minute.

4. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes, the diameters of which are greater than .12 millimeter and not over .20 millimeter, into a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of from 50° C. to 65° C., and winding the thread at a speed of at least 100 meters per minute.

5. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes from .15 to 20 millimeters in diameter in a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of the order of the boiling point of acetone, and winding the thread at a speed of at least 100 meters per minute.

6. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes from .15 to .20 millimeter in diameter into a coagulating bath consisting of water and 20% to 30% by weight of acetone and maintained at a temperature of from 50° C. to 65° C., and winding the thread at a speed of at least 100 meters per minute.

7. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes 0.18 millimeter in diameter in a coagulating bath consisting of water and 23% to 25% by weight of acetone and maintained at a temperature of 63° C., and winding the thread at a speed of approximately 114 meters per minute.

8. A process of producing artificial thread having relatively high tensile strength and possessing axially regular crystalline structure visible under X-rays which comprises spinning a solution of cellulose acetate in acetone through spinnerets having holes 0.18 millimeter in diameter in a coagulating bath consisting of water and 28% to 29% by weight of acetone and maintained at a temperature of 50° C., and winding the thread at a speed of approximately 100 meters per minute.

9. In a process of producing artificial thread having high tensile strength and possessing

axially regular crystalline structure visible under X-rays, the steps which comprise spinning a solution of cellulose acetate through a spinneret into a coagulating bath consisting of water and 20% to 30% by weight of acetone maintained at a temperature of the order of the boiling point of the acetone, and winding the thread at a speed to stretch the previously produced thread at least 1000%.

10. In a process of producing artificial thread having high tensile strength and possessing axially regular crystalline structure visible under X-rays, the steps which comprise spinning a solution of cellulose acetate through a spinneret into a coagulating bath consisting of water and 20% to 30% by weight of acetone maintained at a temperature of the order of the boiling point of the acetone, and winding the thread at a speed of at least 100 meters per minute.

11. In a process of producing artificial thread having high tensile strength and possessing axially regular crystalline structure visible under X-rays, the steps which comprise spinning a solution of cellulose acetate through a spinneret into a coagulating bath consisting of water and 20% to 30% by weight of acetone maintained at a temperature of from 50° C. to 65° C., and winding the thread at a speed of at least 100 meters per minute.

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CERTIFICATE OF CORRECTION.

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It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, second column, line 35, claim 5, for ".15 to 20 millimeters" read .15 to .20 millimeter; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 14th day of March, A.D. 1939.

Henry Van Arsdale.

(Seal) Acting Commissioner of Patents.