Jan. 3, 1939. H. DREYFUS ET AL 2,142,717

MANUFACTURE OF CELLULOSE DERIVATIVE MATERIALS

Original Filed Feb. 1, 1935

FIG. 1

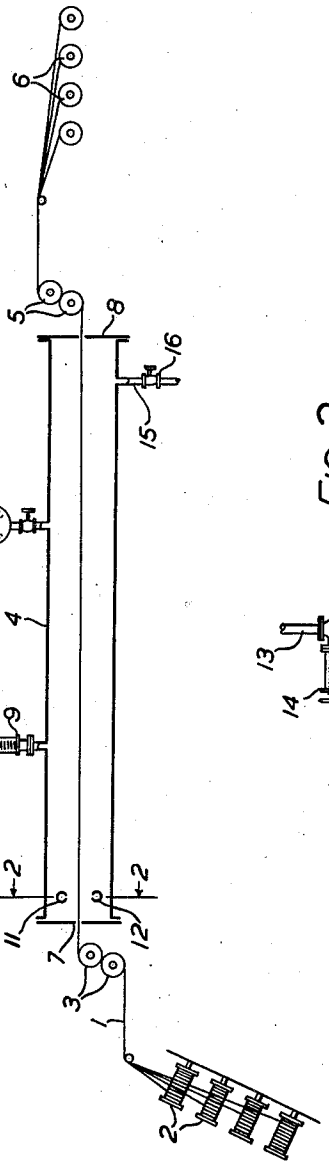
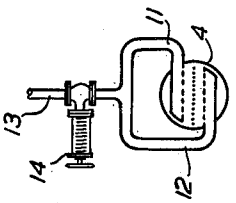


FIG. 2



HENRY DREYFUS
ROBERT W. MONCRIEFF
FRANK B. HILL
INVENTORS

ATTORNEYS

Patented Jan. 3, 1939

2,142,717

UNITED STATES PATENT OFFICE

2,142,717

MANUFACTURE OF CELLULOSE DERIVATIVE MATERIALS

Henry Dreyfus, London, and Robert Wighton Moncrieff and Frank Brentnall Hill, Spondon, near Derby, England, assignors to Celanese Corporation of America, a corporation of Delaware

Original application February 1, 1935, Serial No. 4,510. Divided and this application November 4, 1935, Serial No. 48,084. In Great Britain February 2, 1934

7 Claims. (Cl. 28—59)

This application is divided out from our U. S. application S. No. 4,510 filed February 1, 1935, and relates to new apparatus for the production of artificial filaments, threads, yarns, ribbons, foils, films, and other materials having a basis of cellulose acetate or other cellulose ester or ether and having improved properties, particularly improved tensile strength.

The production of materials of the above character, and particularly filaments and similar textile materials, having an increased tensile strength is of considerable importance from the commercial point of view. The processes which have been hitherto proposed with this object have involved subjecting the threads or other materials to a stretching operation, and in order to obtain substantial increases in tensile strength it has been considered necessary to subject the materials to the action of liquids which are solvents or swelling agents for the materials, for example dioxane, acetone, acetic acid, and diacetone alcohol, all applied for example in aqueous solution. It has also been suggested to use such liquids in the vapour state.

It has now been discovered that the tensile strength of materials having a basis of cellulose acetate or other cellulose ester or ether may be increased in a very simple manner by subjecting them to a stretching operation in the presence of steam. While dry steam may be employed for the purpose, it is highly advantageous to employ the steam in the saturated or wet condition. Besides being cheap and easy from the manipulative point of view, the new process has the advantage over the prior processes that it is considerably more flexible. Thus, in processes in which aqueous dioxane or aqueous acetone is employed as the agent facilitating the stretching of cellulose derivative threads, relatively minor changes in the conditions, particularly the temperature and concentration, have or may have a profound effect upon the properties of the stretched threads, and accordingly to obtain a uniform product it is essential to maintain the conditions constant within very fine limits. With the new process the range of tolerance is much greater.

By the new process high degrees of stretch may be obtained. For example when the steam is in the saturated or wet condition stretches of many hundreds per cent. may be obtained. Thus apart from increased tensile strength, the new process enables filaments or threads of fine denier and thin foils, films and the like to be produced.

The steam may be, and preferably is, employed under pressure so that the materials may be subjected to relatively high temperatures while maintaining the steam in the saturated or wet condition. Thus, for instance, the steam may be at a temperature of 110°, 120°, 130° or 135° C. or more and at the appropriate pressure for the steam to be saturated or wet, such as 10, 20, 30 pounds or more per square inch above atmospheric pressure. The best conditions for treatment are dependent upon the degree of stretch required and the rate of stretching and upon a number of factors to be referred to hereinafter. For relatively low degrees of stretch the pressure may be substantially atmospheric pressure or only a few pounds above atmospheric pressure, the temperature being preferably so adjusted that at the pressure chosen the steam is just saturated or slightly wet.

The steam may be brought into the saturated or wet condition by any suitable means. If superheated steam be used as the source of supply, then cooling, for example by radiation, may be resorted to to secure the required conditions, or water may be injected into the steam so as to produce both a cooling and the presence of liquid water. The steam may be generated in situ in the stretching chamber.

Textile materials to be treated in accordance with the invention may be threads or other materials composed of continuous filaments, or may be spun yarns made of short or more or less long lengths of filament, and may be obtained by cutting up or by any other suitable process, for example by the processes of U. S. application S. No. 726,264 filed May 18, 1934. The materials may initially have any desired lustre from a high lustre to medium lustre or approximately the lustre of natural silk or even a dull lustre or no lustre at all. Thus, the materials may have a low lustre due to the presence therein of titanium dioxide or other pigments. Preferably the materials on entering the steam are substantially free from volatile solvents.

The operation may be carried out on the materials in any suitable form; for example threads may be stretched in hank form, for instance between rollers positioned in a vessel containing steam. Preferably, however, stretching of the materials is effected during the course of their travel from one point to another. The treatment may be applied to a single thread, e. g. during winding from a cake or cheese or bobbin on to a bobbin, or it may be applied to a number of threads simultaneously. Thus, threads may be stretched while running from one creel of bobbins to another creel. When simultaneously stretch-

ing a number of threads, it is advantageous to arrange the threads in parallel alignment in the form of a sheet or, if desired, in the form of two parallel sheets.

The stretching operation may be carried out so that the desired degree of stretch is obtained in a single stage of stretching or, if desired, intermediate rollers or other stretching devices may be provided so as to carry out the stretch in more than one stage.

Broadly the apparatus according to the present invention comprises a steam chamber, means for creating an atmosphere of steam therein and means for stretching the materials while in the chamber. Preferably the steam is introduced from outside the chamber. For the treatment of running threads or other materials, the new apparatus comprises a steam chamber provided with means for introducing steam and one or more inlet and outlet orifices for the materials, associated with means for stretching the materials while in the chamber. Preferably the means for introducing the steam into the chamber are such as to direct steam jets on to the materials immediately upon entry into the steam chamber. The steam chamber may be provided with a pressure-relief valve, a water drain-off for condensed steam, and a pressure gauge. For carrying out the operation in its preferred form, namely under pressure, the steam chamber will be constructed to resist the required pressure and the inlet and outlet orifices for the materials will be as fine as possible.

A suitable apparatus in accordance with the invention is illustrated by way of example in the accompanying drawing in which

Fig. 1 illustrates the general lay-out of the apparatus, the steam chamber being shown in longitudinal section; and

Fig. 2 illustrates a cross-section of the steam chamber on the line 2—2 of Fig. 1.

A number of threads 1 drawn from a creel of bobbins 2 is carried through suitable nip rollers 3 positioned in front of a steam box or pipe 4. The threads are drawn through the steam box or pipe by means of nip rollers 5 positioned at the far end of the steam box or pipe, and finally are taken up on a creel of bobbins 6. To the ends of the steam box or pipe 4 are secured closure plates 7 and 8 bored with the appropriate holes for the inlet and outlet of the threads. The steam box or pipe 4 is provided with a pressure-relief valve 9, a pressure gauge 10 and perforated steam pipes 11 and 12 supplied with steam from a pipe 13 through a reducing valve 14. The perforations in the pipes 11, 12 are arranged so as to direct the steam on to the threads 1 entering the steam box or pipe. The latter is further provided with a drain pipe 15 and valve 16.

The length of thread subjected to the action of the steam may be quite short and good stretching may be obtained with a length of only a few inches. However, it is preferable to extend the length of the steam chamber to some feet, for example 3–6 feet or more, the precise length depending upon the temperature and the amount and rate of stretching.

As previously indicated, for any particular degree of stretch and rate of stretching, a range of temperatures and therefore steam pressures is available. The best temperature at which to work depends upon a large number of factors, including the twist on the initial thread which for quick stretching should be as small as possible, the nature of the cellulose derivative under treatment, materials made of high viscosity cellulose derivatives requiring in general rather higher temperatures and therefore pressures than materials made of low viscosity cellulose derivatives, and the size of the inlet and outlet orifices, and also the position of the steam jets in the steam chamber in relation to the incoming materials. As previously indicated, the inlet and outlet holes should be made as small as possible to reduce escape of steam. Their size will naturally vary with the denier of the threads undergoing stretching. Generally, subject to variation by the above factors, the higher degrees of stretch or higher rates of stretching require higher temperatures and therefore higher pressures of the steam. The temperature and therefore the pressure of the steam is preferably adjusted to enable as low a stretching tension as possible to be used. Under some conditions the individual filaments of the threads tend to fuse together to form a monofil, and the process of the present invention may be utilized to make a product of this character where it is desired. On the other hand, where, as is desirable in most cases, the individual filamentary character of the threads is to be preserved, simple adjustments in the pressure, rate of stretching, amount of stretching, size of inlet and outlet orifices, and the various other factors readily enable this to be done.

The stretched materials may be subjected to any desired after-treatment processes. Thus, the filaments, threads, foils, films and the like may be treated with shrinking agents to improve their extensibility. Suitable processes for this purpose are described in U. S. application S. No. 611,240 filed May 13, 1932. In particular reference is made to the latent solvents referred to in that specification, for example a mixture of methylene chloride and benzene.

Again, stretched cellulose ester materials may be subjected to processes of saponification. The saponification may be such as to lead to a relatively small loss in weight, for example sufficient to give the materials an affinity for cotton dyestuffs, or may be complete or substantially complete, i. e. so as to eliminate all or substantially all of the ester content. The saponification may be effected under such conditions of tension that shrinkage takes place during or immediately after the saponification, this being advantageous from the point of view of the extensibility of the products.

Such further treatments may be carried out as operations separate from the stretching or may be carried out continuously with stretching. Thus, for example, the invention includes a continuous operation which involves first stretching and then shrinking, for example a stretching to several hundreds per cent. of the original length of the material followed by a shrinking by 7–12% of the length of the stretched material; a continuous operation involving stretching followed by saponification; and a continuous operation involving stretching, shrinking, and finally saponification.

While the invention has been described above more particularly with reference to cellulose acetate, it may also be applied to materials containing other cellulose esters or ethers, for example cellulose formate, cellulose propionate, cellulose butyrate, methyl, ethyl, or benzyl cellulose, or mixtures of esters and/or ethers or of mixed esters, ethers, or ether-esters. Furthermore, ribbons, foils, films, sheets and the like may be stretched by the processes described and

in the case of foils, films and the like the stretching may be longitudinal and/or lateral or a lateral tension may be applied sufficient to prevent lateral shrinkage during the application of a longitudinal stretch.

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

1. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned on both sides of said inlet orifice for introducing steam in said chamber, inlet and outlet orifices in said chamber for passage of the materials, a positively driven forwarding device adapted to forward the materials to said chamber and a second positively driven forwarding device adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

2. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned on both sides of said inlet orifice for introducing steam in said chamber, inlet and outlet orifices in said chamber for passage of the materials, a positively driven forwarding device located outside said chamber adapted to forward the materials to said chamber and a second positively driven forwarding device located outside said chamber adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

3. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned so that it is above and below the materials being passed therethrough for introducing steam therein on both sides of the materials, said means being adapted to direct steam on to the materials immediately after their entry into said chamber, inlet and outlet orifices in said chamber for passage of the materials, a positively driven forwarding device adapted to forward the materials to said chamber and a second positively driven forwarding device adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

4. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned so that it is above and below the materials being passed therethrough for introducing steam therein on both sides of the materials, said means being adapted to direct steam on to the materials immediately after their entry into said chamber, inlet and outlet orifices in said chamber for passage of the materials, a positively driven forwarding device located outside said chamber adapted to forward the materials to said chamber and a second positively driven forwarding device located outside said chamber adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

5. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned so that it is above and below the materials being passed therethrough for introducing steam therein on both sides of the materials, inlet and outlet orifices in said chamber for passage of the materials, said orifices being of such size as to prevent substantial escape of steam, a positively driven forwarding device located outside said chamber adapted to forward the materials to said chamber and a second positively driven forwarding device located outside said chamber adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

6. Apparatus for stretching running filaments, threads, foils and similar materials which comprises a substantially closed pressure chamber for steam under pressure, means associated with said chamber and positioned so that it is above and below the materials being passed therethrough for introducing steam therein on both sides of the materials, said means being adapted to direct steam on to the materials immediately after their entry into said chamber, inlet and outlet orifices in said chamber for passage of the materials, said orifices being of such size as to prevent substantial escape of steam, a positively driven forwarding device located outside said chamber adapted to forward the materials to said chamber and a second positively driven forwarding device located outside said chamber adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

7. Apparatus for stretching running filaments, threads, foils and similar materials, which comprises a substantially closed pressure chamber for steam under pressure having detachable end plates provided with orifices for the passage of the materials into and out of the chamber, means associated with said chamber and positioned so that it is above and below the materials being passed therethrough for introducing steam therein on both sides of the materials, a positively driven forwarding device located outside said chamber adapted to forward the materials to said chamber and a second positively driven forwarding device located outside said chamber adapted to withdraw the materials from said chamber at a speed greater than that at which they are forwarded by the first forwarding device so that the materials are stretched between the forwarding devices during their passage through said chamber.

HENRY DREYFUS.
ROBERT WIGHTON MONCRIEFF.
FRANK BRENTNALL HILL.