

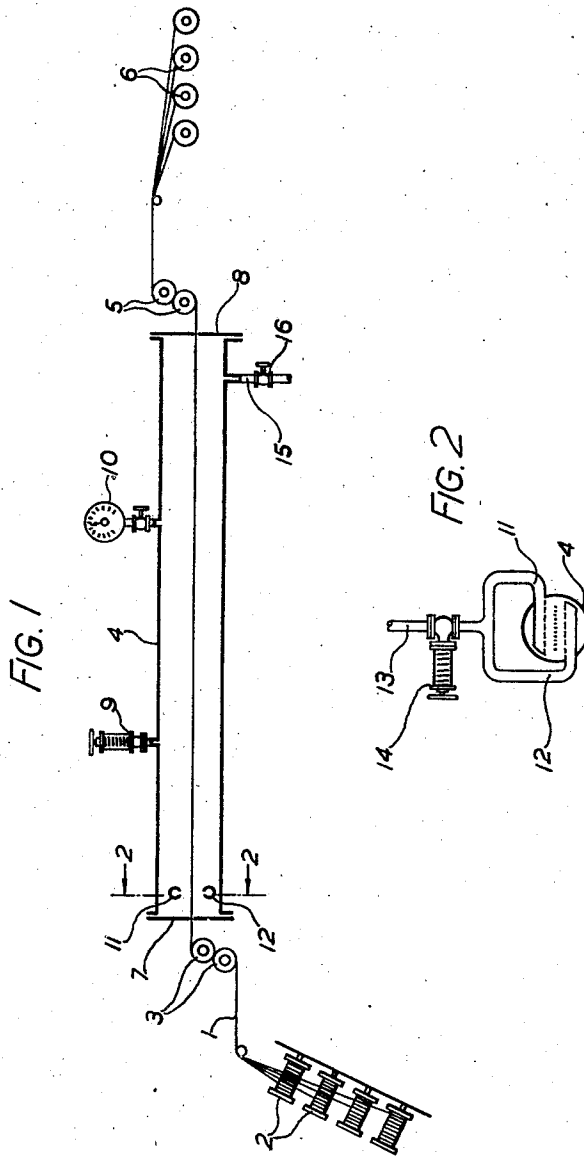
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MANUFACTURE OF CELLULOSE DERIVATIVE MATERIALS

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MANUFACTURE OF CELLULOSE DERIVATIVE MATERIALS

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This invention relates to new processes 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.

This application is a divisional application of our application Serial No. 4,510, filed Feb. 1, 1935.

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 saturated or wet steam under pressure at a temperature above 100° C. 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, 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 a degree of stretch for example, 200% and even 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 is employed under pressure and the materials may thus 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°, 125°, 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 quite low, e. g. only a few pounds above atmospheric pressure, the temperature being preferably so adjusted that at the pressure chosen the steam is only 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. Patent No. 2,077,078. 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 stretching 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.

Apparatus enabling the stretching to be carried out is described and claimed in our co-pending application No. 48,084. Broadly the apparatus 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. The steam chamber is constructed to resist the required pressure and the inlet and outlet orifices for the materials are sufficiently small to enable the pressure to be maintained.

A suitable apparatus for carrying out the process of the invention is illustrated 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 onto the threads 1 entering the steam box or pipe. The latter is further provided with a drain pipe 15 and valves 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 are sufficiently small to enable the pressure to be maintained. Their size may be varied 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. Patent No. 2,058,422. 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 dye-stuffs, 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 stretch-

ing 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.

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

1. Process for improving the strength of filaments, threads and similar textile materials and of foils, films and the like made of an organic substitution derivative of cellulose, which comprises stretching the materials in wet steam under pressure at a temperature above 100° C.

2. Process for the production of lustrous filaments, threads and similar textile materials and of foils, films and the like made of an organic substitution derivative of cellulose, which materials have an improved tenacity which comprises stretching the materials to a degree greater than the dry extensibility thereof in wet steam under pressure at a temperature between 100° C. and 110° C. during their travel from one point to another.

3. Process for improving the strength of filaments, threads and similar textile materials and of foils, films and the like made of cellulose acetate, which comprises stretching the materials to a degree greater than the dry extensibility thereof in wet steam under pressure at a temperature between 100° C. and 110° C. during their travel from one point to another.

4. Process for improving the strength of filaments, threads and similar textile materials and of foils, films and the like made of an organic substitution derivative of cellulose, which comprises stretching the materials to a degree greater than the dry extensibility thereof in wet steam under pressure at a temperature of 110° C. to 125° C. during their travel from one point to another.

5. Process for improving the strength of filaments, threads and similar textile materials and of foils, films and the like made of cellulose acetate, which comprises stretching the materials to a degree greater than the dry extensibility thereof

in wet steam under pressure at a temperature of 110° C. to 125° C. during their travel from one point to another.

6. Process for improving the strength of filaments, threads and similar textile materials and of foils, films and the like made of cellulose acetate, which comprises stretching the materials to a degree greater than the dry extensibility thereof in wet steam under pressure at a temperature above 125° C. during their travel from one point to another.

7. Process for improving the strength of filaments, threads and similar textile materials made of cellulose acetate, which comprises stretching the materials to at least 200% of their original length in wet steam under pressure at a temperature between 100° C. and 110° C. during their travel from one point to another.

8. Process for improving the strength of filaments, threads and similar textile materials made of an organic substitution derivative of cellulose, which comprises stretching the materials to at least 200% of their original length in wet steam under pressure at a temperature of 110 to 125° C. during their travel from one point to another.

9. Process for improving the strength of filaments, threads and similar textile materials made of cellulose acetate, which comprises stretching the materials to at least 200% of their original length in wet steam under pressure at a temperature of 110° C. to 125° C. during their travel from one point to another.

10. Process for improving the strength of filaments, threads and similar textile materials made of cellulose acetate, which comprises stretching the materials to at least 200% of their original length in wet steam under pressure at a temperature above 125° C. during their travel from one point to another.

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