

# UNITED STATES PATENT OFFICE

2,066,492

## PRODUCTION OR TREATMENT OF ARTIFICIAL FILAMENTS OR THE LIKE

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No Drawing. Application June 17, 1933, Serial No. 676,326. In Great Britain July 4, 1932

8 Claims. (Cl. 8--20)

This invention relates to the manufacture or treatment of artificial filaments, yarns, threads, ribbons and the like, and is more especially of value in the manufacture or treatment of such materials which are made of or contain cellulose acetate or other cellulose esters or ethers.

In the manufacture of artificial filaments, threads, yarns and the like, it is customary to draw out the filaments somewhat during their manufacture. With relatively small applied stretch the micelles do not become orientated to any appreciable extent, and in consequence the X-ray photograph shows that the filaments are amorphous in character. When, however, the draw down is very considerable the X-ray analysis of the filaments reveals that the micelles have become more or less parallelized and consequently the filament exhibits an organized structure. Similarly, when already formed filaments are stretched, particularly when stretched to a considerable extent, and when using suitable softening agents, the micelles again become orientated and X-ray analysis reveals a structure in the filament. Where the cellulose molecules become orientated in this manner the tensile strength of the resulting filaments is increased as compared with the tensile strength of filaments containing unorientated micelles or cellulose molecules. While it is of great advantage to achieve this increased tensile strength, as is clear from the many processes which have been proposed for applying stretch either during or subsequent to the manufacture of the filaments, nevertheless the increased tensile strength is not entirely unattended with disadvantages. It appears that when the tensile strength is increased substantially there is a simultaneous diminution in the elasticity of the filaments. While this diminution of elasticity may not be a serious disadvantage from the point of view of the subsequent textile operations, such as twisting, knitting and weaving, nevertheless it does increase the liability of the filaments or yarns when in fabric form to show creases. The present invention aims to produce filaments, yarns and other products in which increased tensile strength consequent upon orientation is still attained in the filaments or other products and yet the liability to creasing is reduced or eliminated.

According to the present invention filaments, threads, yarns, ribbons and like products which have been stretched either during or subsequent to their production and therefore exhibit an X-ray structure are treated superficially so as to destroy or decrease the parallelization of the

cellulose micelles in the outer layers of the product. The de-orientation of the outer layers of the filaments, yarns or other products may be produced according to the present invention by treating the orientated filaments and other products with a solvent or other softening agent in such a manner that only the outer layers of the material are acted upon or softened or rendered plastic by the solvent or other softening agent. The restriction of the action of the softening agent to the outer layers only of the filaments or other products may be achieved in any suitable way. Thus, for example, the agent may be and preferably is in all cases applied in only small quantities so that it does not penetrate to the core of the filaments. Alternatively or in addition, the time factor may be utilized to prevent the softening agent from penetrating to the core and destroying the orientation of the cellulose at this point. Thus, for example, the filaments, yarns or other products may be run through a bath of strong solvent and the solvent quickly removed from the materials. Evaporation may be resorted to or immersion of the materials in a bath which removes the solvent from the softened filaments and which is a precipitant for the cellulose or cellulose derivative constituting the filaments or other products.

As indicated, it is advisable, however, in accordance with the present invention to apply only a restricted amount of softening agent in order to inhibit the possibility of the agent penetrating to the core of the materials. Thus a solvent may be applied in the vapour state, though even here it is advisable to treat the materials only for a short time. Alternatively, a solvent may be applied in medium or relatively low concentration in a diluent so that the filaments or other products, after treatment with the solvent, carry only a restricted amount of such agent. In order to hasten the action of the solvent after application in the diluted state in the diluent, it is advisable to concentrate the restricted amount of solvent on the materials so that it has a very high softening or solvent action upon the surface. For this purpose the diluent employed may be more volatile than the solvent and may be evaporated off so that the restricted amount of solvent upon the filaments becomes highly concentrated and therefore powerful in its action. Further, in order to confine the solvent or other softening agent to the outer layers, it is advisable to employ a relatively volatile softening agent so that it can be quickly removed from the materials, or to remove either a volatile or

a non-volatile agent by other means, such as solution in a suitable diluent. The heat required to remove solvent quickly by evaporation may itself, of course, be the means of increasing and localizing the solvent action upon the filaments. Thus, for instance, filaments carrying a restricted amount of solvent, whether or not diluted with a diluent, may be carried over heated drums or other heated surfaces or through a bath of heated liquid so as to bring the solvent action into play quickly and then to remove the solvent. When a heated liquid is employed, such liquid has preferably no action on the materials and may either be a non-solvent for the softening agent, or may have solvent properties therefor, so that the solvent action and removal of the solvent are effected in the same bath.

The process of the invention may be applied to the treatment of filaments, threads, yarns or other materials of the cellulosic type, as for instance viscose, cuprammonium and nitro-cellulose artificial silks, but is more valuable in relation to materials made of or containing cellulose derivatives, as for instance cellulose acetate, and particularly acetone-soluble cellulose acetate, cellulose formate, cellulose propionate or butyrate or other esters or mixed esters, and methyl, ethyl, butyl and benzyl celluloses, or other cellulose ethers or mixed ethers and also mixed ether-esters, for instance, ethyl cellulose acetate and oxyethyl cellulose acetate, since the number and variety of solvents available for the treatment of this type of product is very considerable. In the treatment of materials of the cellulosic type thiocyanates, caustic soda, zinc chloride and like agents may be used to produce a softening action, while in the treatment of materials of the cellulose derivative type thiocyanates, zinc chloride, lower aliphatic acids, for example formic acid, acetic acid, lactic acid and the like, ketones, for instance acetone and methyl-ethyl ketone, esters, for instance methyl acetate, ethyl acetate, ethyl lactate, diethyl tartrate and the like, the ethers, including cyclic ethers, esters and ether-esters of olefine or polyolefine glycols or glycerine, e. g. glycol monoacetate, methyl or ethyl glycol monoacetate, dioxane, methylene ethylene dioxide and the like, chlorinated hydrocarbons, as for instance chloroform, tetrachlorethane, trichlorethylene, dichlorethylene and the like, phenols, certain alcohols, e. g. ethyl alcohol, etc. may be employed. Moreover, liquids which at normal temperatures have no softening action or substantially no softening action but which at higher temperatures have such action may be employed at temperatures above atmospheric temperature. Examples of such liquids are glycols, glycerine and Turkey-red oil.

In the case of evaporating off a relatively volatile solvent so as to restrict its action upon the filaments, it is desirable to take special precautions to prevent a bundle of filaments or collection of yarns from adhering together while in the sticky state, and thereby forming a monofil type of product, if such type of product is not desired. Thus, the filaments may be kept apart during the concentration of the solvent, for instance by passing them over a curved guide or by applying the heat while the filaments are passing through a hot liquid, for instance a high boiling hydrocarbon, such as tetrahydro or decahydro naphthalene or the higher boiling petroleum hydrocarbons.

The invention includes, however, treatment of the filaments and the like in such a way that the

superficial softening also produces adherence of the filaments so as to obtain a monofil product or a product in which any desired numbers of the filaments are superficially coalesced.

The treatment in accordance with the present invention may be so conducted that the final filaments are lustrous in appearance or have a medium or quite dull lustre. If the treatment is carried out with a solvent in solution in a less volatile diluent or non-solvent, then a dull lustre may result from the present treatment, while if the solvent and diluent are about equally volatile or the diluent is more volatile than the solvent, then the lustre may be maintained or even increased.

The treatment may be carried out so that a slight stretch is applied to the filaments during the solvent treatment. It is found that this slight stretch will not materially prevent the de-orientation of the molecules in the outer layer of the filaments or other products.

As previously indicated, the stretching to produce the initial orientation in the filaments or other products may be carried out during the actual production of the materials or in an after-treatment of the already formed filaments or like materials. In the latter case any of the solvents previously mentioned may be employed to assist the stretching which is preferably, by suitable adjustment of the concentration of solvent agent, carried to more than 100%, and especially over 300%, of the original length of the materials. In producing the requisite stretch during spinning; means are preferably adopted, such as the coagulating baths of U. S. applications S. Nos. 402,785 filed October 26, 1929, 418,414 filed January 3, 1930, 437,423 filed March 20, 1930, 469,622 filed July 21, 1930, 589,428 filed January 28, 1932, 601,043 filed March 24, 1932, 607,688 filed April 26, 1932, and British Patents Nos. 177,868 and 179,234, to maintain the plasticity of the filaments during stretching.

Examples of processes which may be employed in the stretching of formed filaments and the like are those described in U. S. Patent No. 1,709,470, U. S. applications S. Nos. 378,684 filed July 16, 1929, and 573,424 filed November 6, 1931.

Treatment with the softening agents according to the present invention may be applied to materials in the form of hanks or other suitable packages, but preferably it is carried out during the travel of the materials from one point to another. Moreover, the treatment may be effected as an operation distinct from the stretching operation, or continuously therewith. Thus a number of filaments or threads in the form of a warp, which have been stretched, e. g. as described in U. S. application S. No. 573,424, may be dried and then treated according to the present invention continuously with the stretching operation.

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

#### Example 1

A cellulose acetate yarn which has been drawn down to fine denier during a wet spinning process is immersed in hank form in a 70% aqueous solution of methylated spirit until it is well wetted out and is then dried at 65° C.

#### Example 2

A dry spun cellulose acetate yarn which has been stretched to 400% of its original length is immersed for about 2 minutes at room tempera-

ture in a solution consisting of 80 parts of tetrachlorethane and 20 parts of toluene by volume. It is then removed, washed out in toluene and dried.

*Example 3*

A similar yarn to that treated in Example 2 is unwound from a bobbin or other suitable device and passed through a bath containing 10% of glycol mono-acetate at such a speed that it is immersed in the bath for a period of about 30 seconds. On issuing from the bath it is washed with water in any suitable manner and then dried and rewound.

*Example 4*

A stretched cellulose acetate yarn is passed from a bobbin or other suitable device over a heated roller running in di-ethylene glycol maintained at 105° C. at a speed of about 20 metres per minute, then over a wick, pad or other wiping device to a suitable apparatus where it is rewound.

*Example 5*

A cellulose acetate fabric containing stretched yarns is treated with Turkey red oil at 80° C. for about 15 minutes and is then removed, scoured and dried.

The materials obtained according to the above examples have a reduced liability to creasing. The term "swelling agent" as employed hereinafter in the claims is to be construed as including not only agents which exert only a swelling action on the materials but also true solvents.

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

1. Process for the treatment of stretched artificial filaments, threads, yarns, ribbons and the like containing organic derivatives of cellulose and fabrics containing the same to improve their resistance to creasing, which comprises treating them with a medium comprising a swelling agent under such conditions that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

2. Process for the treatment of stretched artificial filaments, threads, yarns, ribbons and the like containing cellulose acetate and fabrics containing the same to improve their resistance to creasing, which comprises treating them with a medium comprising a swelling agent under such conditions that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

3. Process for the treatment of artificial filaments, threads, yarns, ribbons and the like which contain cellulose acetate and which have been stretched to at least 300% of their original length, and fabrics containing the same to improve their resistance to creasing, which com-

prises treating them with a medium, containing a swelling agent having a low softening action on the products, under such conditions that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

4. Process for the treatment of stretched artificial filaments, threads, yarns, ribbons and the like containing organic derivatives of cellulose, and fabrics containing the same to improve their resistance to creasing, which comprises treating them with a restricted quantity of a medium, containing a swelling agent having a low softening action on the products, so that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

5. Process for the treatment of artificial filaments, threads, yarns, ribbons and the like which contain cellulose acetate and which have been stretched to at least 300% of their original length, and fabrics containing the same to improve their resistance to creasing, which comprises treating them for a short period of time with a medium, containing a swelling agent having a low softening action on the products, so that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

6. Process for the treatment of artificial filaments, threads, yarns, ribbons and the like which contain cellulose acetate and which have been stretched to at least 300% of their original length, and fabrics containing the same to improve their resistance to creasing, which comprises treating them with a medium comprising a swelling agent of relatively low volatility so that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

7. Process according to claim 6 wherein the swelling agent of relatively low volatility employed is glycol mono-acetate.

8. Process for the treatment of artificial filaments, threads, yarns, ribbons and the like which contain cellulose acetate and which have been stretched to at least 300% of their original length, and fabrics containing the same to improve their resistance to creasing, which comprises treating them with a medium which comprises a swelling agent of relatively low volatility and which has a low softening action on the products so that only the micelles of the outer layers are acted upon and disarranged by said agent and the artificial filaments, threads, yarns, ribbons and the like are prevented from adhering to one another.

PERCY FREDERICK COMBE SOWTER.

## CERTIFICATE OF CORRECTION.

Patent No. 2,066,492.

January 5, 1937.

PERCY FREDERICK COMBE SOWTER.

It is hereby certified that error appears in the above numbered patent requiring correction as follows: In the grant and heading to the printed specification, title of invention, for the words "or the like" read and the like; page 2, second column, line 39, for the serial number "607,688" read 607,668; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of March, A. D. 1937.

(Seal)

Henry Van Arsdale  
Acting Commissioner of Patents.